243 SHERIDAN STREET

NEW CASSEL, NEW YORK

SECTION 11, BLOCK 44, LOT 74

Phase II Environmental Site Investigation Work Plan

EPA Petroleum No. BF-9649919
AKRF Project Number: 200225

Prepared for:

United States Environmental Protection, Region 2
290 Broadway, 25th Floor
New York, NY 10007

On Behalf Of:

The Town of North Hempstead
210 Plandome Road
Manhasset, NY 11030

Prepared by:

AKRF, Inc.
440 Park Avenue South, 7th Floor
New York, NY 10016

JUNE 2023

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1.0 INTRODUCTION

This Phase II Environmental Site Investigation (ESI) Work Plan (Work Plan) has been prepared by AKRF, Inc. (AKRF) on behalf of the Town of North Hempstead (TONH) for the property located at 243 Sheridan Street in the Hamlet of New Cassel, NY (the "Site"). The Site location is shown on Figure 1. This Work Plan provides a plan to sample and analyze environmental media for the ESI to be conducted under United States Environmental Protection Agency (USEPA) Petroleum Grant No. BF-9649919. A Health and Safety Plan (HASP) outlining the safety procedures to be employed to protect on-site personnel and the public during implementation of the Phase II ESI is included as Appendix A. A Quality Assurance Project Plan (QAPP) that describes the protocols and procedures that will be followed during implementation of the Phase II ESI is included as Appendix B. The HASP and QAPP constitute the planning documents for the proposed work. This Work Plan is being submitted to the USEPA for approval prior to commencing environmental testing at the Site.

2.0 BACKGROUND

2.1 Site Description

The Site is located at 243 Sheridan Street in the Hamlet of New Cassel, NY, and is defined on the Nassau County Tax Map as Section 11, Block 44, Lot 74. The approximately 6,000-square foot (sf) Site contains a one-story, single-family residence with a cellar, a detached garage, and exterior paved and landscaped areas. The Site is bounded to the north, south, and west by single-family residences, and to the east by Sheridan Street, followed by single-family residences. The area surrounding the Site was primarily residential and commercial, with some educational, industrial, and recreational uses. The Site location is shown on Figure 1, and a map detailing the physical layout of the Site including adjacent land use is provided as Figure 2.

2.2 Previous Environmental Investigations

AKRF completed a Phase I Environmental Site Assessment (ESA) of the Site in conformance with American Society for Testing and Materials (ASTM) Standard E1527-13, *Standard Practice for Environmental Site Assessments: Phase I Environmental Site Assessment Practice* in September 2022. At the time of the Phase I ESA, the Site contained a vacant, one-story, single-family residence with a cellar, a detached garage, and exterior paved and landscaped areas. Historically, the Site was developed with a one-story residence with a cellar and a detached garage by 1929; an addition was constructed in the southwestern portion of the residence prior to 1941. The footprints of the Site buildings remained generally consistent with the footprints observed during AKRF's Phase I ESA reconnaissance since at least 1941. The Phase I ESA identified the following Recognized Environmental Conditions (RECs):

- Suspected fill and vent piping potentially associated with a current or former petroleum storage tank were observed at the Site; the piping protruded from the foundation wall along the asphalt-paved driveway, adjacent to the northwestern corner of the residence.
- The Site was connected to the Nassau County sewer system by 1982; however, many of the properties in the surrounding area contained cesspools prior to the 1980s. No records indicating the presence or subsequent closure of a cesspool at the Site were identified.
- A spill incident (Spill No. 0011983) was reported at 746 Broadway, located north-adjacent to the Site. According to available information, petroleum-contaminated soil was identified following the removal of a 550-gallon UST; however, no detail regarding the nature of the release or the actions taken to address the conditions were identified.

In addition to the RECs, the Phase I ESA also identified Business Environmental Risks (BERs), including: a mound of potential fill material from an unknown source in the southwestern portion of the Site; the potential presence of asbestos-containing material (ACM), lead-based paint (LBP), lead-containing paint (LCP), polychlorinated biphenyl (PCB)-containing material, and/or mercury-containing material in the Site buildings, fill material, buried structures, and/or buried demolition debris; and the potential for water damage and mold within the Site buildings.

No known sampling assessments have been conducted at the Site.

3.0 FIELD PROGRAM

The purpose of this Work Plan is to develop a USEPA-approved plan to sample and analyze environmental media. To accomplish this goal, the Phase II ESI field program will focus on collecting soil data, and, depending on field observations, groundwater data. A summary of the tasks is provided in the following subsections. Detailed procedures for each of the sampling tasks are provided in the QAPP provided as Appendix B.

It should be noted that suspect ACM and/or LBP/LCP may be present in fill material, buried structures, and/or buried demolition debris, which will be addressed prior to redevelopment of the Site. As these hazardous contaminants are not being addressed under Petroleum Grant No. BF-9649919, they are not included as part of this Work Plan.

The purpose of this Work Plan is to develop a plan to sample and analyze environmental media at the Site, and to provide a document to the USEPA for review and approval of the plan. A summary of the tasks is provided in the following subsections. Detailed procedures for each of the sampling tasks are provided in the QAPP provided as Appendix B.

3.1 Geophysical Survey and Utility Mark-outs

A geophysical survey will be conducted across the accessible portions of the Site to clear the proposed soil boring locations for subsurface utilities and to locate other potential buried structures, including bulk storage tanks, fuel oil supply lines, and septic systems. The geophysical survey will include both electromagnetic (EM) and ground penetrating radar (GPR) methods. Any anomalies identified will be marked in the field using spray paint and recorded in the field book.

Utility mark-outs are required by law; therefore, the drilling contractor will call Dig Safely New York at least three days prior to implementation of the work outlined in this Work Plan.

3.2 Soil Boring Advancement

Up to six soil borings (SB-01 through SB-06) will be advanced at the approximate locations shown on Figure 2. Four soil borings (SB-01 through SB-04) will be advanced in exterior portions of the Site to approximately 12 feet below surface grade, the anticipated excavation depth for a new residential building, using a track-mounted Geoprobe® Direct Push Probe (DPP) rig. If the residence is deemed safe for entry, two interior soil borings (SB-05 and SB-06) would be advanced to approximately 5 feet below cellar grade in the vicinity of the suspected petroleum storage tank (if present) or in areas where evidence of a current/former petroleum storage tank are identified using a remote-access Geoprobe® DPP rig. If the residence is inaccessible, the two interior soil borings may be relocated to exterior portions of the Site.

If field evidence of contamination other than fill material [e.g., elevated photoionization detector (PID) readings, staining, product, and/or odors] is identified in any of the soil borings, the soil boring will be advanced until the vertical extent of contamination can be delineated or until equipment refusal is encountered, and additional samples may be collected.

Table I – Soil Boring/Sample Summary

Soil Boring ID	On-Site Location	Rationale
SB-01	Southeastern portion of the Site	To assess soil quality in the southeastern portion of the Site, and to assess subsurface conditions adjacent to the suspected cesspool (if located during the geophysical survey)

Table I – Soil Boring/Sample Summary

Soil Boring ID	On-Site Location	Rationale
SB-02	Southwestern portion of the Site	To assess soil quality in the vicinity of the mound of potential fill material, and to assess subsurface conditions adjacent to the suspected cesspool (if located during the geophysical survey)
SB-03	Northwestern portion of the Site	To assess soil quality in the vicinity of the garage, and to assess subsurface conditions adjacent to the suspected cesspool (if located during the geophysical survey)
SB-04	Northern portion of the Site	To assess soil quality in the vicinity of the suspected petroleum storage tank fill and vent pipes, and to assess subsurface conditions adjacent to the suspected cesspool (if located during the geophysical survey)
SB-05	Within the building cellar	To assess soil quality in the vicinity of the suspected petroleum storage tank or where evidence of a current/former petroleum storage tank are identified
SB-06	Within the building cellar	To assess soil quality in the vicinity of the suspected petroleum storage tank or where evidence of a current/former petroleum storage tank are identified

3.3 Soil Sample Collection and Analysis

Continuous soil cores will be collected from surface grade to the terminal depth in each soil boring using 3- or 5-foot long, 2-inch diameter, stainless steel macrocore piston rod samplers fitted with internal, dedicated acetate liners. The soil cores will be inspected for evidence of contamination (e.g., staining, product, and/or odors), screened for the presence of volatile organics with a PID equipped with a 10.6 electron volt (eV) lamp, and logged using the modified Burmister soil classification system (QAPP Section 25.1). The PID will be calibrated daily in accordance with the manufacturer's recommendations. Any soil sample collected via hand tools would be collected with a decontaminated stainless steel hand auger. All sampling equipment (e.g., drilling rods/casing, macrocore samplers, and probe rods) will be either dedicated or decontaminated between sampling locations (QAPP Section 25.5).

At least one soil sample from each soil boring will be analyzed for the full list of compounds included on the Target Compound List (TCL) and in New York State Department of Environmental Protection (NYSDEC) Part 375, Table 375-6.8(a) for the following parameters:

- Volatile organic compounds (VOCs) according to USEPA Method 8260;
- Semivolatile organic compounds (SVOCs) according to USEPA Method 8270;
- Metals according to USEPA Method 6000/7000 series;
- Polychlorinated biphenyls (PCBs) according to USEPA Method 8082;
- Pesticides according to USEPA Method 8081; and
- Herbicides according to USEPA Method 8151.

The sampling interval for the soil samples submitted for laboratory analysis will be selected based on field evidence of contamination, including any staining, odors, elevated PID readings, and/or

observation of historic fill. If no evidence of contamination is identified, one sample will be collected from the 2-foot interval immediately below the surface cover (i.e., grass, asphalt, concrete, etc.). If field evidence of contamination other than fill material (e.g., elevated PID readings, staining, product, and/or odors) is identified in any of the soil borings, the soil boring will be advanced until the vertical extent of contamination can be delineated or until equipment refusal is encountered, and additional samples may be collected.

All samples will be analyzed by a New York State Department of Health (NYSDOH)-certified laboratory with Category B deliverables. Sample containers will be labeled and placed in an ice-filled cooler and shipped to the laboratory via courier with appropriate chain of custody (COC) documentation (QAPP Sections 15.1, 15.2, and 15.3).

One blind duplicate, field (aqueous rinsate) blank, trip blank, and matrix spike/matrix spike duplicate will be collected for quality control/quality assurance (QA/QC) purposes for every 20 field samples collected. The QA/QC samples, besides trip blanks, will be analyzed for the same testing parameters as the environmental samples (as listed above). Trip blanks will be analyzed for VOCs only. The data will be reviewed by a third-party validator, and a Data Usability Summary Report (DUSR) will be prepared to document the usability and validity of the data. The soil boring locations will be surveyed using the Global Positioning System (GPS).

3.4 Temporary Groundwater Well Installation, and Groundwater Sample Collection and Analysis

Based on United States Geological Survey (USGS) mapping, the depth to groundwater beneath the Site is estimated to be approximately 45 to 55 feet below surface grade. If contamination is identified in any of the soil borings within 10 feet of the groundwater interface, a temporary groundwater well will be installed in the open borehole. A maximum of three wells will be installed. The temporary well(s) will be constructed with 2-inch diameter polyvinyl chloride (PVC) casing with 10-foot long, 0.020-inch slotted well screen straddling the water table. A sand pack will be installed around the well annulus from the bottom of the well to approximately 5 feet above the screened interval. Following installation, each well will be developed to remove any accumulated fines and establish a hydraulic connection with the surrounding aquifer (QAPP Section 25.2). If three or more wells are installed, the wells will be surveyed by a New York State licensed surveyor to determine their accurate location and elevation (QAPP Section 25.4).

After development, one groundwater sample will be collected from each well. The expected targeted purge rate will be approximately 100 milliliter per minute (ml/min) and geochemical water parameters will be monitored during purging (QAPP Section 25.3).

The groundwater sample(s) will be analyzed for the full list of TCL/Part 375 compounds for the following parameters:

- VOCs according to USEPA Method 8260;
- SVOCs according to USEPA Method 8270;
- Metals according to USEPA Method 6000/7000 series;
- PCBs according to USEPA Method 8082;
- Pesticides according to USEPA Method 8081; and
- Herbicides according to USEPA Method 8151.

All samples will be analyzed by a NYSDOH-certified laboratory with Category B deliverables. Sample containers will be labeled and placed in an ice-filled cooler and shipped to the laboratory via courier with appropriate COC documentation (QAPP Sections 15.1, 15.2, and 15.3).

One blind duplicate, field (aqueous rinsate) blank, trip blank, and matrix spike/matrix spike duplicate will be collected for QA/QC purposes for every 20 field samples collected. The QA/QC samples, besides trip blanks, will be analyzed for the same testing parameters as the environmental samples (as listed above). Trip blanks will be analyzed for VOCs only. The data will be reviewed by a third-party validator, and a DUSR will be prepared to document the usability and validity of the data. The well locations will (also) be surveyed using the GPS.

3.5 Management of Investigation-Derived Waste (IDW)

All soil IDW will be used to backfill the corresponding borehole that generated them to within 24 inches of surface grade or will be disposed of or treated according to applicable local, state, and federal regulations. The purge water and decontamination fluids will be discharged to the ground in accordance with NYSDEC DER-10 (QAPP Section 25.6) unless evidence of contamination is noted during sampling or purging. Soil and groundwater IDW exhibiting evidence of gross contamination will be containerized in Department of Transportation (DOT)-approved 55-gallon drums. The drums will be sealed at the end of each work day and labeled with the date, the soil boring/well number(s), the type of waste (i.e., drill cuttings, decontamination fluids, purge water, etc.) and the name and phone number of the AKRF project manager. Both the drum lid and the drum will be labeled with permanent and weatherproof marker, and a drum log will be kept in the field book. All drums will be labeled "pending analysis" until laboratory data is available, at which point the drums will be re-labeled with either a “non-hazardous” or a “hazardous” label prior to off-site disposal. Handling of IDW and backfilling of boreholes will be conducted in accordance with Section 3.3(e) of DER-10 (QAPP, Section 25.6).

3.6 Phase II Environmental Site Investigation Report

A Phase II ESI Report will be prepared summarizing the finding from the investigation. The report will include a description of the investigation and sampling methods, and will present the field and laboratory analytical results with recommendations for future remediation and/or development. Field data sheets, photographic documentation, a figure with sampling locations, and laboratory analytical reports will be included as attachments.

4.0 QUALIFICATIONS OF PROPOSED STAFF

The following project team will manage and conduct the Phase II ESI at the Site. Full resumes for each proposed staff are provided in Appendix C.

4.1 Project Director

The project director will be responsible for the general oversight of all aspects of the project, including scheduling, budgeting, data management, and decision-making regarding the field program. The project director will communicate regularly with all members of the AKRF project team and USEPA, to ensure a smooth flow of information between involved parties. Deborah Shapiro, QEP, will serve as the project director for the project. Ms. Shapiro's resume is included in Appendix C.

4.2 Project Manager

The project manager will be responsible for directing and coordinating all elements of the Work Plan. They will prepare reports and participate in meetings with the contractors, client, and USEPA. Timothy McClintock will serve as the project manager for this project. Mr. McClintock's resume is included in Appendix C.

4.3 Field Team Leader

The field team leader will be responsible for supervising the daily sampling and health and safety activities in the field and will ensure adherence to the Work Plan and HASP. They will report to the AKRF project manager on a regular basis regarding daily progress and deviations from the Work Plan. The field team leader will be a qualified, responsible person, able to act professionally and promptly during soil disturbing activities. Michael Bates will be the field team leader for this project. Mr. Bates' resume is included in Appendix C.

4.4 Project Quality Assurance/Quality Control Officer

The QA/QC Officer will be responsible for adherence to the QAPP. They will review the procedures with all personnel prior to commencing any fieldwork and will conduct periodic site visits to assess implementation of the procedures. The QA/QC officer will also be responsible for reviewing analytical data reports and associated QA/QC sampling data. Michelle Lapin, P.E., will serve as the QA/QC officer for this project. Ms. Lapin's resume is included in Appendix C.

4.5 Laboratory Quality Assurance/Quality Control Officer

The laboratory QA/QC officer will be responsible for quality control procedures and checks in the laboratory and ensuring adherence to laboratory protocols. They will track the movement of samples from the time they are checked in at the laboratory to the time that analytical results are issued. They will conduct a final check on the analytical calculations and sign off on the laboratory reports. Carl Armbruster is the QA manager for the Eurofins Environment Testing of America of Edison, New Jersey, where the Phase II ESI samples will be analyzed. Mr. Armbruster's resumes is included in Appendix C, and a copy of Eurofins Environment Testing of America's Quality Assurance Manual is provided as Appendix D.

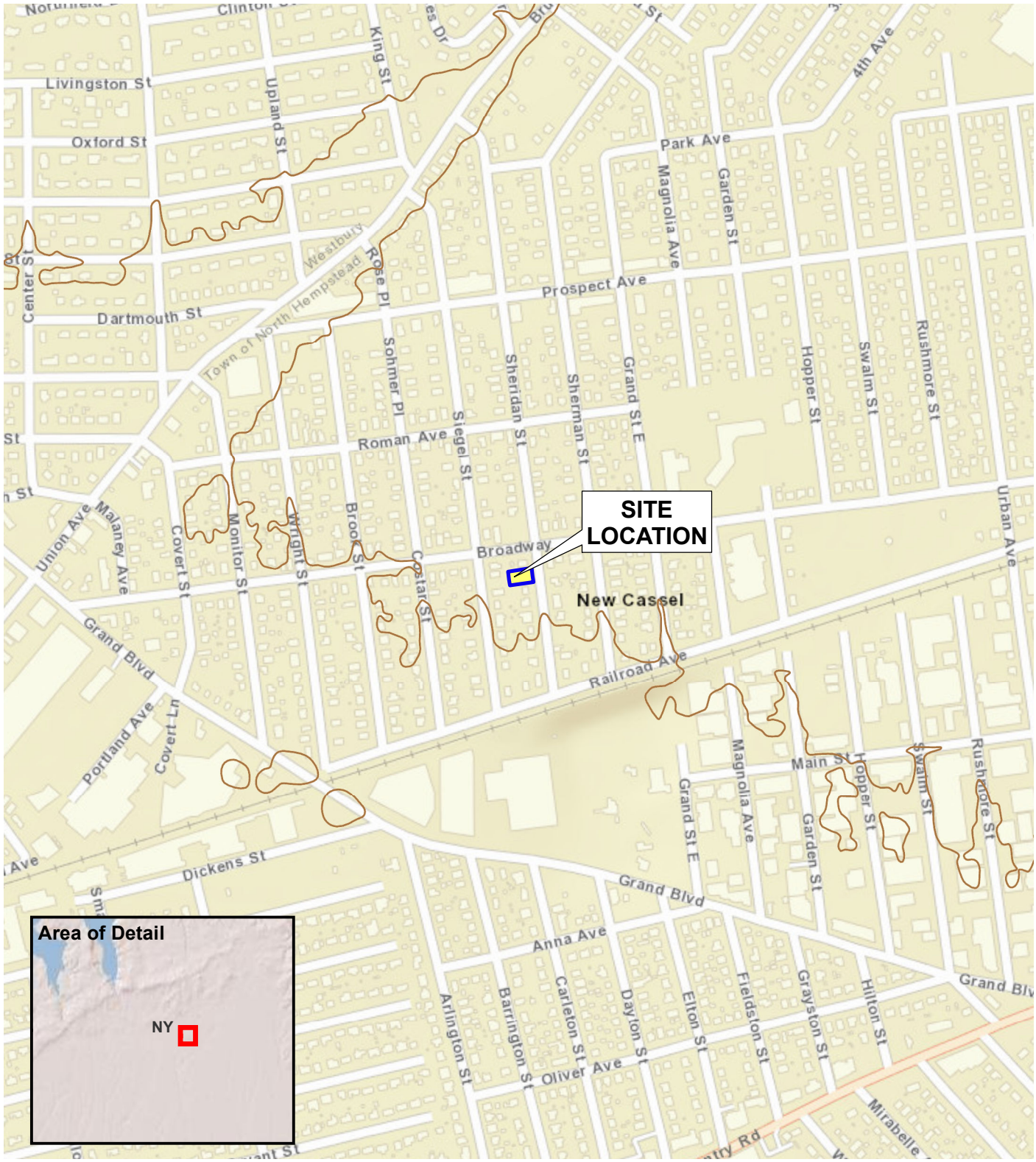
4.6 Third-Party Data Validator

All laboratory analytical data will be reviewed by a third-party validator and a Data Usability Summary Report (DUSR) will be prepared to document the usability and validity of the data. Lori Beyer is President of L.A.B. Validation and will be responsible for reviewing the data and preparing the DUSR. Ms. Beyer's resume and certifications are included in Appendix C.

5.0 PROJECT SCHEDULE

After USEPA approval of this Work Plan, the field work portion of the Phase II ESI will be scheduled. Depending upon subcontractor availability, weather, and accessibility of the Site to complete the work, it is anticipated that the field work will be completed within approximately two weeks of USEPA Work Plan approval. The field work is anticipated to take two to three days to complete. The geophysical survey will be completed on the first day; and soil boring advancement, soil sampling, groundwater well installation, groundwater sampling, and well surveying will be conducted over two days. As the samples will be analyzed on a standard turnaround time, sample results will be received approximately two weeks after laboratory receipt of the samples. Not including USEPA's review time, the Phase II ESI Report will be submitted approximately three to four weeks after completion of the field work.

FIGURES



Service Layer Credits: ESRI World Street Map, 2021.

0 600 1,200

SCALE IN FEET

AKRF
440 Park Avenue South, New York, NY 10016

243 Sheridan Street
New Cassel, New York

SITE LOCATION

DATE
11/22/2022

PROJECT NO.
200225

FIGURE
1

LEGEND

- PROJECT SITE BOUNDARY
- 74 — LOT BOUNDARY AND TAX LOT NUMBER
- **44** — BLOCK NUMBER
- ● — PROPOSED SOIL BORING LOCATION

Map Source:
http://www.nassaucountyny.gov/mynassauproperty/main.jsp.

Aerial Source:
ESRI World Imagery 2021.

0 30 60
SCALE IN FEET

AKRF
440 Park Avenue South, New York, NY 10016

243 Sheridan Street
New Cassel, New York

SITE AND PROPOSED SAMPLE LOCATION PLAN

DATE
11/22/2022

PROJECT NO.
200225

FIGURE
2

APPENDIX A
HEALTH AND SAFETY PLAN

243 SHERIDAN STREET

NEW CASSEL, NEW YORK

SECTION 11, BLOCK 44, LOT 74

Health and Safety Plan

EPA Petroleum No. BF-9649919
AKRF Project Number: 200225

Prepared for:

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JUNE 2023

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FIGURES

ATTACHMENTS

1.0 INTRODUCTION

This Health and Safety Plan (HASP) has been prepared for the property located at 243 Sheridan Street in the Hamlet of New Cassel, NY (the "Site"). The approximately 6,000-square foot (sf) Site is defined on the Nassau County Tax Map as Section 11, Block 44, Lot 74, and contains a one-story, single-family residence with a cellar, a detached garage, and exterior paved and landscaped areas. The Site is bounded to the north, south, and west by single-family residences, and to the east by Sheridan Street, followed by single-family residences. A map showing the location of the Site is provided as Figure 1.

This environmental HASP has been developed for implementation during the Phase II Environmental Site Investigation (ESI) activities conducted by all personnel on-site, both AKRF employees and others. The Phase II ESI will include a geophysical survey, soil boring advancement and soil sampling, and temporary groundwater well installation and groundwater sampling, as needed. This HASP does not discuss other routine health and safety issues common to general construction/excavation, including but not limited to slips, trips, falls, shoring, and other physical hazards.

All AKRF employees are directed that all work must be performed in accordance with the Company's Generic HASP and all Occupational Safety and Health Administration (OSHA) applicable regulations for the work activities required for the project. All project personnel are furthermore directed that they are not permitted to enter Permit Required Confined Spaces (as defined by OSHA). For issues unrelated to contaminated materials, all non-AKRF employees are to be bound by all applicable OSHA regulations as well as any more stringent requirements specified by their employer in their corporate HASP or otherwise. AKRF is not responsible for providing oversight for issues unrelated to contaminated materials for non-employees. This oversight shall be the responsibility of the employer of that worker or other official designated by that employer.

2.0 HEALTH AND SAFETY GUIDELINES AND PROCEDURES

2.1 Hazard Evaluation

2.1.1 Hazards of Concern

Check all that apply		
(**X**) Organic Chemicals	(**X**) Inorganic Chemicals	( ) Radiological
( ) Biological	(**X**) Explosive/Flammable	( ) Oxygen Deficient Atm
(**X**) Heat Stress	(**X**) Cold Stress	( ) Carbon Monoxide
Comments: No personnel are permitted to enter permit confined spaces.		

2.1.2 Physical Characteristics

Check all that apply		
(**X**) Liquid	(**X**) Solid	( ) Sludge
(**X**) Vapors	( ) Unknown	( ) Other
Comments:		

2.1.3 Hazardous Materials

Check all that apply					
Chemicals	**Solids**	**Sludges**	**Solvents**	**Oils**	**Other**
( ) Acids	( ) Ash	( ) Paints	(**X**) Halogens	( ) Transformer	( ) Lab
( ) Caustics	( ) Asbestos	( ) Metals	(**X**) Petroleum	( ) Other DF	( ) Pharm
( ) Pesticides	( ) Tailings	( ) POTW	( ) Other	(**X**) Motor or Hydraulic Oil	( ) Hospital
(**X**) Petroleum	(**X**) Other: Fill material	( ) Other		(**X**) Gasoline	( ) Rad
( ) Inks				(**X**) Fuel Oil	( ) MGP
(**X**) PCBs					( ) Mold
(**X**) Metals					( ) Cyanide
(**X**) Other: VOCs & PAHs					

2.1.4 Chemicals of Concern

Chemicals	REL/PEL/STEL (ppm)	Health Hazards
Arsenic	REL= 0.002 mg/m^3 PEL= 0.01 mg/m^3	Ulceration of nasal septum, dermatitis, gastrointestinal disturbances, peripheral neuropathy, respiratory irritation, hyperpigmentation of skin; potential occupational carcinogen.
Barium	REL: TWA 10 mg/m^3 (total) TWA 5 mg/m^3 (resp) PEL†: TWA 15 mg/m^3 (total) TWA 5 mg/m^3 (resp)	Irritation eyes, nose, upper respiratory system; benign pneumoconiosis (baritosis)
Benzene	REL = 0.1 ppm PEL = 1 ppm STEL = 5 ppm	Irritation eyes, skin, nose, respiratory system; dizziness; headache, nausea, staggered gait; anorexia, lassitude, dermatitis; bone marrow depression, potential occupational carcinogen.
Cadmium	TWA 0.005 mg/m^3	Pulmonary edema, dyspnea (breathing difficulty), cough, chest tightness, substernal (occurring beneath the sternum) pain; headache; chills, muscle aches; nausea, vomiting, diarrhea; anosmia (loss of the sense of smell), emphysema, proteinuria, mild anemia; [potential occupational carcinogen]
Chromium	REL= 0.5 mg/m^3 PEL= 1 mg/m^3	Irritation eyes, skin; lung fibrosis (histologic).
Ethylbenzene	REL = 100 ppm PEL = 100 ppm	Irritation eyes, skin, mucous membrane; headache; dermatitis; narcosis, coma.
Toluene	REL = 100 ppm PEL = 200 ppm STEL = 300 ppm	Irritation eyes, nose; lassitude, confusion, euphoria, dizziness, headache; dilated pupils, lacrimation (discharge of tears); anxiety, muscle fatigue, insomnia; paresthesia (skin tingling or numbness); dermatitis; liver, kidney damage.
Xylenes	REL = 100 ppm PEL = 100 ppm	Irritation eyes, skin, nose, throat; dizziness, excitement, drowsiness, poor coordination, staggering gait; corneal vacuolization; anorexia, nausea, vomiting, abdominal pain; dermatitis.
PCBs	PCB-1242: REL = 1 mg/m^3 PEL = 0.001 mg/m^3 PCB-1254: REL = 0.5 mg/m^3 PEL = 0.001 mg/m^3	Rash; anemia, liver, stomach, thyroid damage; reduced ability to fight disease; impaired reproduction.
Lead	REL=0.1 mg/m^3 PEL=0.05 mg/m^3	Weakness, lassitude, insomnia; facial pallor, pale eye, anorexia, low-weight, malnutrition, constipation, abdominal pain, colic; anemia; gingival lead line; tremors, paralysis wrists and ankles; encephalopathy; kidney disease; irritation eyes; hypotension.
Polycyclic Aromatic Hydrocarbons (PAHs)	PEL = 5 mg/m^3	Harmful effects to skin, bodily fluids, and ability to fight disease, reproductive problems; [potential occupational carcinogen]
Selenium	REL=0.2 mg/m^3 PEL=0.2 mg/m^3	Irritation eyes, skin, nose, throat; visual disturbance; headache; chills, fever; dyspnea (breathing difficulty), bronchitis; metallic taste, garlic breath, gastrointestinal disturbance; dermatitis; eye, skin burns; In Animals: anemia; liver necrosis, cirrhosis; kidney, spleen damage.
Silver	REL=0.01 mg/m^3 PEL=0.01 mg/m^3	Blue-gray eyes, nasal septum, throat, skin; irritation, ulceration skin; gastrointestinal disturbance.

Chemicals	REL/PEL/STEL (ppm)	Health Hazards
Mercury	PEL= 0.1 mg/m^3	irritation eyes, skin; cough, chest pain, dyspnea (breathing difficulty), bronchitis, pneumonitis; tremor, insomnia, irritability, indecision, headache, lassitude (weakness, exhaustion).
Comments: REL = NIOSH Recommended Exposure Limit PEL = OSHA Permissible Exposure Limit STEL = OSHA Short Term Exposure Limit		

The above listed chemicals of concern are reasonably anticipated based on the limited information obtained from the September 2022 Phase I Environmental Site Assessment (ESA) of the Site. The potential health effects from these chemicals of concern are included in Attachment A.

2.2 Designated Personnel

AKRF will appoint its field team leader as the Site Safety Officer (SSO). This individual will be responsible for the implementation of the HASP. The SSO will be one of our experienced field geologists, scientists, or engineers with an environmental-related B.S. or B.A. degree and experience in implementation of air monitoring and hazardous materials sampling programs. Health and safety training required for the SSO and all field personnel are outlined in Section 2.3 of this HASP.

2.3 Training

All personnel who enter the work area while intrusive activities are being performed will have completed the 40-hour Hazardous Waste Operations and Emergency Response (HAZWOPER) training course that meets OSHA requirements of 29 Code of Federal Regulation (CFR) Part 1910, OSHA Standards. In addition, all personnel will have up-to-date 8-hour refresher training as well as additional in-house health and safety training. The training will allow personnel to recognize and understand the potential hazards to health and safety. All field personnel must attend a training program, whose purpose is to:

- Make them aware of the potential hazards they may encounter;
- Provide the knowledge and skills necessary for them to perform the work with minimal risk to health and safety;
- Make them aware of the purpose and limitations of safety equipment; and
- Ensure that they can safely avoid or escape from emergencies.

Each member of the field crew will be instructed in these objectives before they go onto the site. A site safety meeting will be conducted at the start of the project. Additional meetings shall be conducted, as necessary, for new personnel working at the site.

2.4 Medical Surveillance Program

All AKRF and subcontractor personnel performing field work involving subsurface disturbance at the Site are required to have passed a complete medical surveillance examination in accordance with 29 CFR 1910.120 (f). A physician's medical release for work will be confirmed by the SSO before an employee can begin Site activities. The medical release shall consider the type of work to be performed and the required personal protective equipment (PPE). The medical examination will, at a minimum, be provided annually and upon termination of hazardous waste Site work.

West Nile Virus/St. Louis Encephalitis Prevention information is included as Attachment B.

2.5 Site Work Zones

During any activities involving subsurface disturbance, the work area must be divided into various zones to prevent the spread of contamination, ensure that proper protective equipment is donned, and provide an area for decontamination. Control measures will include caution tape and/or traffic cones to create adequate buffers to protect visitors to the Site and the general public. Field adjustment to the work zones may be necessary based on actual work locations, field evidence of contamination [odors, staining, and/or photoionization detector (PID) readings] or Site observations, or other health and safety concerns.

The Exclusion Zone is defined as the area where exposure to impacted media could be encountered. The Contamination Reduction Zone (CRZ) is the area where decontamination procedures take place and is located next to the Exclusion Zone. The Support Zone is the area where support facilities such as vehicles, fire extinguishers, and first aid supplies are located. The emergency staging area (part of the Support Zone) is the area where all workers on-site would assemble in the event of an emergency. A summary of these areas is provided below. These zones may be changed by the SSO, depending on that day's activities. All field personnel will be informed of the location of these zones before work begins.

Task	Exclusion Zone	CRZ	Support Zone
Soil Boring Advancement	10 feet from drill rig	25 feet from drill rig	As deemed appropriate by SSO
Comments: Control measures such as "caution tape" and/or traffic cones will be placed around the perimeter of the work area when work is being done in a public area.			

2.6 Air Monitoring

The purpose of the air monitoring program is to identify any exposure of the field personnel to potential environmental hazards in the soil and soil vapor. Results of the air monitoring will be used to determine the appropriate response action, if needed.

2.6.1 Volatile Organic Compounds (VOCs)

A PID will be used to perform air monitoring during soil disturbance activities to determine airborne levels of total VOCs. The PID will be calibrated at the start of the work day with 100 parts per million (ppm) isobutylene standard gas.

2.6.2 Work Zone Air Monitoring

Real time air monitoring will be performed with the PID. Measurements will be taken prior to commencement of work and continuously during the work, as outlined in the following table. Measurements will be made as close to the workers as practicable and at the breathing height of the workers. The SSO shall set up the equipment and confirm that it is working properly. His/her designee may oversee the air measurements during the day. The initial measurement for the day will be performed before the start of work and will establish the background level for that day. The final measurement for the day will be performed after the end of work. The action levels and required responses are listed in the following table.

Instrument	Action Level	Response Action
PID	Less than 5 ppm in breathing zone	Level D or D-Modified
	Between 5 ppm and 10 ppm	Level C
	More than 10 ppm	Stop work. Resume work when readings are less than 20 ppm.

2.7 Personal Protection Equipment (PPE)

The PPE required for various kinds of site investigation tasks are based on 29 CFR 1910.120, HAZWOPER, Appendix B, "General Description and Discussion of the Levels of Protection and Protective Gear."

AKRF field personnel and other Site personnel shall wear, at a minimum, Level D personal protective equipment. The protection will be based on the air monitoring described in Section 2.6.

LEVEL OF PROTECTION & PPE		Sampling
Level D (**X**) Steel Toe Shoes (**X**) Hard Hat (within 25 feet of drill rig) (**X**) Work Gloves	(**X**) Safety Glasses ( ) Face Shield (**X**) Ear Plugs (within 25 feet of drill rig) (**X**) Nitrile Gloves (**X**) Tyvek for drill rig operator if NAPL present	Yes
Level C ***(in addition to Level D)*** (**X**) Half-Face or Full Face Respirator ( ) Full-Face PAPR	( ) Particulate Cartridge ( ) Organic Cartridge (**X**) Dual Organic/ Particulate Cartridge	If PID > 5 ppm (breathing zone)
Comments: Cartridges to be changed out at least once per shift unless warranted beforehand (e.g., more difficult to breathe or any odors detected).		

2.8 General Work Practices

To protect the health and safety of the field personnel, field personnel will adhere to the guidelines listed below during activities involving subsurface disturbance:

- Eating, drinking, chewing gum or tobacco, and smoking are prohibited, except in designated areas on the site. These areas will be designated by the SSO.
- Workers must wash their hands thoroughly on leaving the work area and before eating, drinking, or any other such activity.
- The workers should shower as soon as possible after leaving the Site. Contact with contaminated or suspected surfaces should be avoided.
- The buddy system should always be used; each buddy should watch for signs of fatigue, exposure, and heat/cold stress.

3.0 EMERGENCY PROCEDURES AND EMERGENCY RESPONSE PLAN

The field crew will be equipped with emergency equipment, such as a first aid kit and disposable eye washes. In the case of a medical emergency, the SSO will determine the nature of the emergency and they will have someone call for an ambulance, if needed. If the nature of the injury is not serious (i.e., the person can be moved without expert emergency medical personnel) they should be taken to a hospital by on-site personnel. Attachment C includes forms to be completed in the event that a problem arises or an incident occurs. Appendix D includes the hand signals to be used in the event of an emergency. In addition to the general work practices listed above, COVID-19 considerations will be employed at all times while implementing the Phase II ESI at the Site. The COVID-19 considerations are included as Attachment E.

Directions to the hospital are provided below, and a hospital route map is attached as Figure 2.

3.1 HOSPITAL DIRECTIONS

Hospital Name:	Nassau University Medical Center Emergency Room
Phone Number:	(516) 296-2100
Address/Location:	2201 Hempstead Turnpike, East Meadow, NY 11554
Directions:	1. Turn RIGHT out of the Site driveway, heading north on Sheridan Street. 2. Turn RIGHT onto Brush Hollow Road. 3. Turn RIGHT onto the ramp to Wantagh Pkwy South/Jones Beach. 4. Take exit W3 West on the RIGHT for NY-24W towards Hempstead. 5. Turn RIGHT onto NY-24 West/Hempstead Bethpage Turnpike/Hempstead Turnpike. 6. Turn RIGHT at Perimeter E, then turn immediately LEFT. The emergency room entrance will be on the RIGHT.

3.2 EMERGENCY CONTACTS

Company	Individual Name	Title	Contact Number
AKRF	Deborah Shapiro, QEP	Project Director	(646) 388-9544 (office) (917) 957-8991 (cell)
	Timothy McClintock	Project Manager	(914) 922-2374 (office) (914) 439-1629 (cell)
	Michael Bates	SSO	(914) 355-0693 (cell)
TONH	Neal Stone, AICP, MCIP	Client Representative	(516) 869-7809 (office)
Emergency Services	-	-	911
NYSDEC Spill Hotline	-	-	800-457-7362

4.0 APPROVAL & ACKNOWLEDGMENTS OF HASP

APPROVAL

Signed: _____ Date: _____
AKRF Project Manager

Signed: _____ Date: _____
AKRF Health and Safety Officer

Below is an affidavit that must be signed by all workers who enter the site. A copy of the HASP must be on-site at all times and will be kept by the SSO.

AFFIDAVIT

I,_____(name), of_____(company name), have read the Health and Safety Plan (HASP) for the property located at 243 Sheridan Street in the Hamlet of New Cassel, NY (the "Site"). I agree to conduct all on-site work in accordance with the requirements set forth in this HASP and understand that failure to comply with this HASP could lead to my removal from the site.

Signed:	_____	Company:	_____	Date:	_____
Signed:	_____	Company:	_____	Date:	_____
Signed:	_____	Company:	_____	Date:	_____
Signed:	_____	Company:	_____	Date:	_____
Signed:	_____	Company:	_____	Date:	_____
Signed:	_____	Company:	_____	Date:	_____
Signed:	_____	Company:	_____	Date:	_____
Signed:	_____	Company:	_____	Date:	_____
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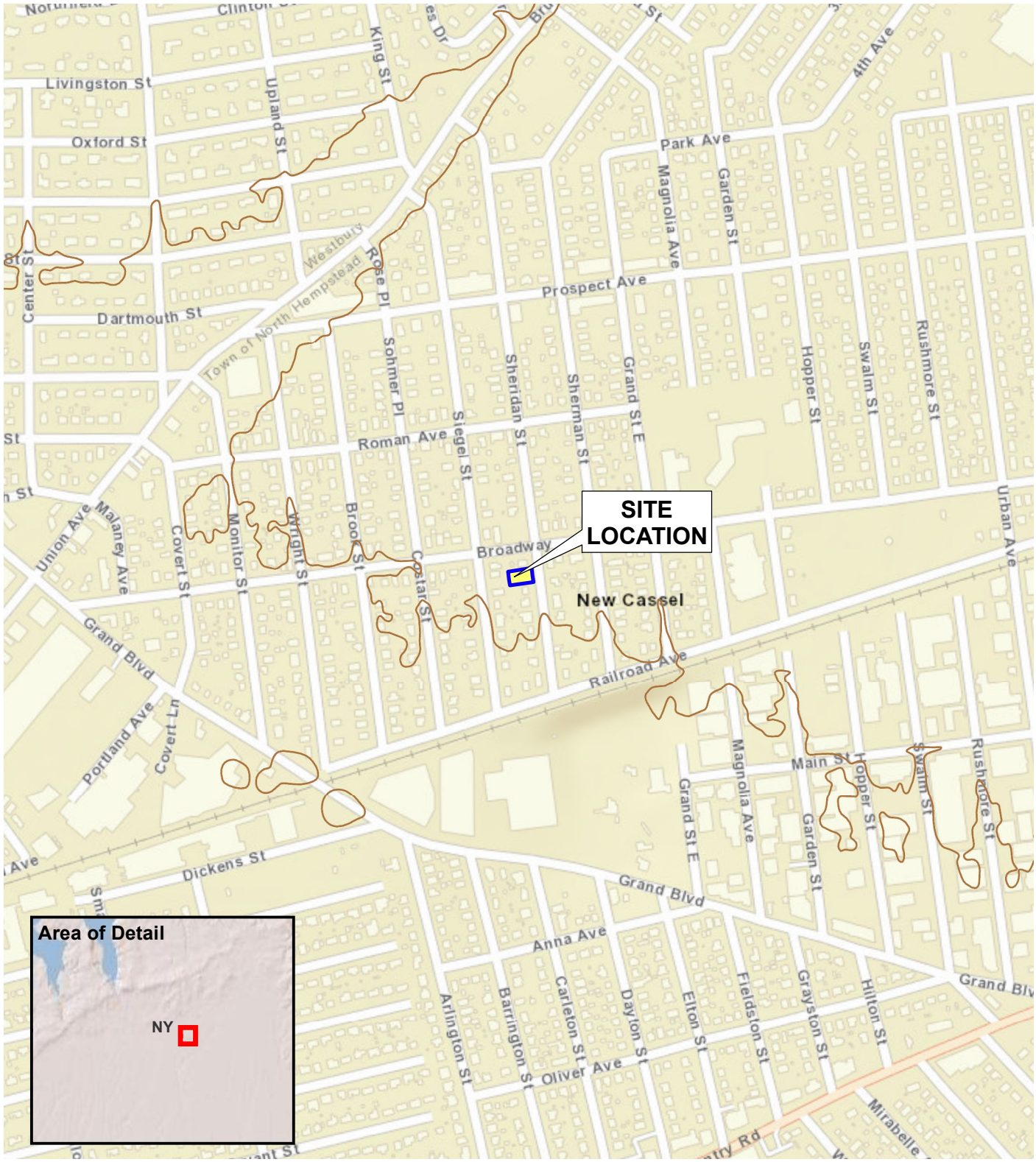
Signed:	_____	Company:	_____	Date:	_____
Signed:	_____	Company:	_____	Date:	_____
Signed:	_____	Company:	_____	Date:	_____
Signed:	_____	Company:	_____	Date:	_____
Signed:	_____	Company:	_____	Date:	_____
Signed:	_____	Company:	_____	Date:	_____
Signed:	_____	Company:	_____	Date:	_____

FIGURE 1

SITE LOCATION MAP



Service Layer Credits: ESRI World Street Map, 2021.

0 600 1,200

SCALE IN FEET

AKRF
440 Park Avenue South, New York, NY 10016

243 Sheridan Street
New Cassel, New York

SITE LOCATION

DATE
11/22/2022

PROJECT NO.
200225

FIGURE
1

FIGURE 2

HOSPITAL ROUTE AND LOCATION

Service Layer Credits: ESRI World Street Map 2020.

0 2,000 4,000

SCALE IN FEET

AKRF
440 Park Avenue South, New York, NY 10016

243 Sheridan Street
New Cassel, New York

HOSPITAL ROUTE AND LOCATION

DATE
11/22/2022

PROJECT NO.
200225

FIGURE
2

ATTACHMENT A

POTENTIAL HEALTH EFFECTS FROM ON-SITE CONTAMINANTS

SAFETY DATA SHEET

Version 6.1
Revision Date 01/15/2020
Print Date 05/29/2020

SECTION 1: Identification of the substance/mixture and of the company/undertaking

1.1 Product identifiers

Product name : Arsenic

Product Number : 202657
Brand : Aldrich
Index-No. : 033-001-00-X
CAS-No. : 7440-38-2

1.2 Relevant identified uses of the substance or mixture and uses advised against

Identified uses : Laboratory chemicals, Synthesis of substances

1.3 Details of the supplier of the safety data sheet

Company : Sigma-Aldrich Inc.
3050 Spruce Street
ST. LOUIS MO 63103
UNITED STATES

Telephone : +1 314 771-5765
Fax : +1 800 325-5052

1.4 Emergency telephone number

Emergency Phone # : +1-703-527-3887

SECTION 2: Hazards identification

2.1 Classification of the substance or mixture

GHS Classification in accordance with 29 CFR 1910 (OSHA HCS)

Acute toxicity, Oral (Category 4), H302
Acute toxicity, Inhalation (Category 3), H331
Carcinogenicity (Category 1B), H350
Short-term (acute) aquatic hazard (Category 1), H400
Long-term (chronic) aquatic hazard (Category 1), H410

For the full text of the H-Statements mentioned in this Section, see Section 16.

2.2 GHS Label elements, including precautionary statements

Pictogram

Signal word Danger

Hazard statement(s)

H302	Harmful if swallowed.
H331	Toxic if inhaled.
H350	May cause cancer.
H410	Very toxic to aquatic life with long lasting effects.

Precautionary statement(s)

P201	Obtain special instructions before use.
P202	Do not handle until all safety precautions have been read and understood.
P261	Avoid breathing dust/ fume/ gas/ mist/ vapours/ spray.
P264	Wash skin thoroughly after handling.
P270	Do not eat, drink or smoke when using this product.
P271	Use only outdoors or in a well-ventilated area.
P273	Avoid release to the environment.
P280	Wear protective gloves/ protective clothing/ eye protection/ face protection.
P301 + P312 + P330	IF SWALLOWED: Call a POISON CENTER/doctor if you feel unwell. Rinse mouth.
P304 + P340 + P311	IF INHALED: Remove person to fresh air and keep comfortable for breathing. Call a POISON CENTER/doctor.
P308 + P313	IF exposed or concerned: Get medical advice/ attention.
P391	Collect spillage.
P403 + P233	Store in a well-ventilated place. Keep container tightly closed.
P405	Store locked up.
P501	Dispose of contents/ container to an approved waste disposal plant.

2.3 Hazards not otherwise classified (HNOC) or not covered by GHS - none

SECTION 3: Composition/information on ingredients

3.1 Substances

Formula	:	As
Molecular weight	:	74.92 g/mol
CAS-No.	:	7440-38-2
EC-No.	:	231-148-6
Index-No.	:	033-001-00-X

Component	Classification	Concentration
Arsenic		
	Acute Tox. 4; Acute Tox. 3; Carc. 1B; Aquatic Acute 1; Aquatic Chronic 1; H302, H331, H350, H400, H410 M-Factor - Aquatic Acute: 10	<= 100 %

For the full text of the H-Statements mentioned in this Section, see Section 16.

SECTION 4: First aid measures

4.1 Description of first aid measures

General advice
Consult a physician. Show this safety data sheet to the doctor in attendance.Move out of dangerous area.

If inhaled
If breathed in, move person into fresh air. If not breathing, give artificial respiration. Consult a physician.

In case of skin contact
Wash off with soap and plenty of water. Take victim immediately to hospital. Consult a physician.

In case of eye contact
Flush eyes with water as a precaution.

If swallowed
Never give anything by mouth to an unconscious person. Rinse mouth with water. Consult a physician.

4.2 Most important symptoms and effects, both acute and delayed
The most important known symptoms and effects are described in the labelling (see section 2.2) and/or in section 11

4.3 Indication of any immediate medical attention and special treatment needed
No data available

SECTION 5: Firefighting measures

5.1 Extinguishing media

Suitable extinguishing media
Use water spray, alcohol-resistant foam, dry chemical or carbon dioxide.

5.2 Special hazards arising from the substance or mixture
Arsenic oxides

5.3 Advice for firefighters
Wear self-contained breathing apparatus for firefighting if necessary.

5.4 Further information
No data available

SECTION 6: Accidental release measures

6.1 Personal precautions, protective equipment and emergency procedures
Wear respiratory protection. Avoid dust formation. Avoid breathing vapours, mist or gas. Ensure adequate ventilation. Evacuate personnel to safe areas. Avoid breathing dust. For personal protection see section 8.

6.2 Environmental precautions
Prevent further leakage or spillage if safe to do so. Do not let product enter drains. Discharge into the environment must be avoided.

6.3 Methods and materials for containment and cleaning up

Pick up and arrange disposal without creating dust. Sweep up and shovel. Keep in suitable, closed containers for disposal.

6.4 Reference to other sections

For disposal see section 13.

SECTION 7: Handling and storage

7.1 Precautions for safe handling

Avoid contact with skin and eyes. Avoid formation of dust and aerosols.Further processing of solid materials may result in the formation of combustible dusts. The potential for combustible dust formation should be taken into consideration before additional processing occurs.
Provide appropriate exhaust ventilation at places where dust is formed.
For precautions see section 2.2.

7.2 Conditions for safe storage, including any incompatibilities

Keep container tightly closed in a dry and well-ventilated place.

Keep in a dry place.
Storage class (TRGS 510): 6.1B: Non-combustible, acute toxic Cat. 1 and 2 / very toxic hazardous materials

7.3 Specific end use(s)

Apart from the uses mentioned in section 1.2 no other specific uses are stipulated

SECTION 8: Exposure controls/personal protection

8.1 Control parameters

Components with workplace control parameters

Component	CAS-No.	Value	Control parameters	Basis
Arsenic	7440-38-2	TWA	0.01 mg/m3	USA. ACGIH Threshold Limit Values (TLV)
	Remarks	Lung cancer Substances for which there is a Biological Exposure Index or Indices (see BEI® section) Confirmed human carcinogen		
		C	0.0020 mg/m3	USA. NIOSH Recommended Exposure Limits
		Potential Occupational Carcinogen See Appendix A 15 minute ceiling value		

Biological occupational exposure limits

Component	CAS-No.	Parameters	Value	Biological specimen	Basis
Arsenic	7440-38-2	inorganic arsenic plus methylated metabolites	35µg As/l	Urine	ACGIH - Biological Exposure Indices (BEI)
	Remarks	End of the workweek (After four or five consecutive working			

		days with exposure)

8.2 Exposure controls

Appropriate engineering controls
Avoid contact with skin, eyes and clothing. Wash hands before breaks and immediately after handling the product.

Personal protective equipment

Eye/face protection
Face shield and safety glasses Use equipment for eye protection tested and approved under appropriate government standards such as NIOSH (US) or EN 166(EU).

Skin protection
Handle with gloves. Gloves must be inspected prior to use. Use proper glove removal technique (without touching glove's outer surface) to avoid skin contact with this product. Dispose of contaminated gloves after use in accordance with applicable laws and good laboratory practices. Wash and dry hands.

Full contact
Material: Nitrile rubber
Minimum layer thickness: 0.11 mm
Break through time: 480 min
Material tested:Dermatril® (KCL 740 / Aldrich Z677272, Size M)

Splash contact
Material: Nitrile rubber
Minimum layer thickness: 0.11 mm
Break through time: 480 min
Material tested:Dermatril® (KCL 740 / Aldrich Z677272, Size M)

data source: KCL GmbH, D-36124 Eichenzell, phone +49 (0)6659 87300, e-mail sales@kcl.de, test method: EN374
If used in solution, or mixed with other substances, and under conditions which differ from EN 374, contact the supplier of the CE approved gloves. This recommendation is advisory only and must be evaluated by an industrial hygienist and safety officer familiar with the specific situation of anticipated use by our customers. It should not be construed as offering an approval for any specific use scenario.

Body Protection
Complete suit protecting against chemicals, The type of protective equipment must be selected according to the concentration and amount of the dangerous substance at the specific workplace.

Respiratory protection
Where risk assessment shows air-purifying respirators are appropriate use a full-face particle respirator type N100 (US) or type P3 (EN 143) respirator cartridges as a backup to engineering controls. If the respirator is the sole means of protection, use a full-face supplied air respirator. Use respirators and components tested and approved under appropriate government standards such as NIOSH (US) or CEN (EU).

Control of environmental exposure
Prevent further leakage or spillage if safe to do so. Do not let product enter drains. Discharge into the environment must be avoided.

SECTION 9: Physical and chemical properties

9.1 Information on basic physical and chemical properties

a)	Appearance	Form: powder Colour: light grey, black
b)	Odour	No data available
c)	Odour Threshold	No data available
d)	pH	No data available
e)	Melting point/freezing point	Melting point/range: 817 °C (1503 °F) - lit.
f)	Initial boiling point and boiling range	613 °C 1135 °F - lit.
g)	Flash point	()Not applicable
h)	Evaporation rate	No data available
i)	Flammability (solid, gas)	No data available
j)	Upper/lower flammability or explosive limits	No data available
k)	Vapour pressure	No data available
l)	Vapour density	No data available
m)	Relative density	5.727 g/mL at 25 °C (77 °F)
n)	Water solubility	No data available
o)	Partition coefficient: n-octanol/water	No data available
p)	Auto-ignition temperature	No data available
q)	Decomposition temperature	No data available
r)	Viscosity	No data available
s)	Explosive properties	No data available
t)	Oxidizing properties	No data available

9.2 Other safety information

No data available

SECTION 10: Stability and reactivity

10.1 Reactivity

No data available

10.2 Chemical stability

Stable under recommended storage conditions.

10.3 Possibility of hazardous reactions
No data available

10.4 Conditions to avoid
Heat Exposure to air may affect product quality.

10.5 Incompatible materials
Strong oxidizing agents

10.6 Hazardous decomposition products
Hazardous decomposition products formed under fire conditions. - Arsenic oxides
Other decomposition products - No data available
In the event of fire: see section 5

SECTION 11: Toxicological information

11.1 Information on toxicological effects

Acute toxicity
LD50 Oral - Rat - 763 mg/kg
Remarks: Behavioral:Ataxia. Diarrhoea
LD50 Oral - Mouse - 145 mg/kg
Remarks: Behavioral:Ataxia. Diarrhoea
Inhalation: No data available
Dermal: No data available
No data available

Skin corrosion/irritation
No data available

Serious eye damage/eye irritation
No data available

Respiratory or skin sensitisation
No data available

Germ cell mutagenicity

Carcinogenicity
No data available

IARC:	1 - Group 1: Carcinogenic to humans (Arsenic)
NTP:	No component of this product present at levels greater than or equal to 0.1% is identified as a known or anticipated carcinogen by NTP.
OSHA:	No component of this product present at levels greater than or equal to 0.1% is on OSHA's list of regulated carcinogens.

Reproductive toxicity

Specific target organ toxicity - single exposure
No data available

Specific target organ toxicity - repeated exposure
No data available

Aspiration hazard
No data available

Additional Information
RTECS: CG0525000

To the best of our knowledge, the chemical, physical, and toxicological properties have not been thoroughly investigated.

Stomach - Irregularities - Based on Human Evidence
Stomach - Irregularities - Based on Human Evidence

SECTION 12: Ecological information

12.1 Toxicity

Toxicity to fish	LC50 - Pimephales promelas (fathead minnow) - 9.9 mg/l - 96.0 h
Toxicity to daphnia and other aquatic invertebrates	EC50 - Daphnia magna (Water flea) - 3.8 mg/l - 48 h

12.2 Persistence and degradability
No data available

12.3 Bioaccumulative potential
No data available

12.4 Mobility in soil
No data available

12.5 Results of PBT and vPvB assessment
PBT/vPvB assessment not available as chemical safety assessment not required/not conducted

12.6 Other adverse effects
An environmental hazard cannot be excluded in the event of unprofessional handling or disposal.
Very toxic to aquatic life with long lasting effects.

SECTION 13: Disposal considerations

13.1 Waste treatment methods

Product
Offer surplus and non-recyclable solutions to a licensed disposal company. Contact a licensed professional waste disposal service to dispose of this material. Dissolve or mix the material with a combustible solvent and burn in a chemical incinerator equipped with an afterburner and scrubber.

Contaminated packaging
Dispose of as unused product.

SECTION 14: Transport information

DOT (US)
UN number: 1558 Class: 6.1 Packing group: II
Proper shipping name: Arsenic
Reportable Quantity (RQ): 1 lbs
Reportable Quantity (RQ): 1 lbs

Poison Inhalation Hazard: No

IMDG
UN number: 1558 Class: 6.1 Packing group: II EMS-No: F-A, S-A
Proper shipping name: ARSENIC
Marine pollutant : yes

IATA
UN number: 1558 Class: 6.1 Packing group: II
Proper shipping name: Arsenic

SECTION 15: Regulatory information

SARA 302 Components
No chemicals in this material are subject to the reporting requirements of SARA Title III, Section 302.

SARA 313 Components
The following components are subject to reporting levels established by SARA Title III, Section 313:

	CAS-No.	Revision Date
Arsenic	7440-38-2	2015-11-23

SARA 311/312 Hazards
Acute Health Hazard, Chronic Health Hazard

Reportable Quantity : D004 lbs

Massachusetts Right To Know Components
No components are subject to the Massachusetts Right to Know Act.

Pennsylvania Right To Know Components

	CAS-No.	Revision Date
Arsenic	7440-38-2	2015-11-23

SECTION 16: Other information

Further information

The above information is believed to be correct but does not purport to be all inclusive and shall be used only as a guide. The information in this document is based on the present state of our knowledge and is applicable to the product with regard to appropriate safety precautions. It does not represent any guarantee of the properties of the product. Sigma-Aldrich Corporation and its Affiliates shall not be held liable for any damage resulting from handling or from contact with the above product. See www.sigma-aldrich.com and/or the reverse side of invoice or packing slip for additional terms and conditions of sale.

The branding on the header and/or footer of this document may temporarily not visually match the product purchased as we transition our branding. However, all of the information in the document regarding the product remains unchanged and matches the product ordered. For further information please contact mlsbranding@sial.com.

Version: 6.1 Revision Date: 01/15/2020 Print Date: 05/29/2020

SAFETY DATA SHEET

Version 6.3
Revision Date 01/21/2020
Print Date 06/02/2020

SECTION 1: Identification of the substance/mixture and of the company/undertaking

1.1 Product identifiers

Product name : Barium

Product Number : 735787
Brand : Aldrich
CAS-No. : 7440-39-3

1.2 Relevant identified uses of the substance or mixture and uses advised against

Identified uses : Laboratory chemicals, Synthesis of substances

1.3 Details of the supplier of the safety data sheet

Company : Sigma-Aldrich Inc.
3050 Spruce Street
ST. LOUIS MO 63103
UNITED STATES

Telephone : +1 314 771-5765
Fax : +1 800 325-5052

1.4 Emergency telephone number

Emergency Phone # : +1-703-527-3887

SECTION 2: Hazards identification

2.1 Classification of the substance or mixture

GHS Classification in accordance with 29 CFR 1910 (OSHA HCS)

Substances and mixtures, which in contact with water, emit flammable gases (Category 2), H261

For the full text of the H-Statements mentioned in this Section, see Section 16.

2.2 GHS Label elements, including precautionary statements

Pictogram

Signal word Danger

Hazard statement(s)
H261 In contact with water releases flammable gases.

Precautionary statement(s)
P223 Do not allow contact with water.
P231 + P232 Handle under inert gas. Protect from moisture.

P280	Wear protective gloves/ eye protection/ face protection.
P335 + P334	Brush off loose particles from skin. Immerse in cool water/ wrap in wet bandages.
P370 + P378	In case of fire: Use dry sand, dry chemical or alcohol-resistant foam to extinguish.
P402 + P404	Store in a dry place. Store in a closed container.
P501	Dispose of contents/ container to an approved waste disposal plant.

2.3 Hazards not otherwise classified (HNOC) or not covered by GHS - none

SECTION 3: Composition/information on ingredients

3.1 Substances

Formula	:	Ba
Molecular weight	:	137.33 g/mol
CAS-No.	:	7440-39-3
EC-No.	:	231-149-1

Component	Classification	Concentration
Barium		
	Water-react. 2; H261	<= 100 %

For the full text of the H-Statements mentioned in this Section, see Section 16.

SECTION 4: First aid measures

4.1 Description of first aid measures

General advice
Consult a physician. Show this safety data sheet to the doctor in attendance.Move out of dangerous area.

If inhaled
If breathed in, move person into fresh air. If not breathing, give artificial respiration. Consult a physician.

In case of skin contact
Wash off with soap and plenty of water. Consult a physician.

In case of eye contact
Flush eyes with water as a precaution.

If swallowed
Never give anything by mouth to an unconscious person. Rinse mouth with water. Consult a physician.

4.2 Most important symptoms and effects, both acute and delayed
The most important known symptoms and effects are described in the labelling (see section 2.2) and/or in section 11

4.3 Indication of any immediate medical attention and special treatment needed
No data available

SECTION 5: Firefighting measures

5.1 Extinguishing media

Suitable extinguishing media
Dry powder

5.2 Special hazards arising from the substance or mixture
Barium oxide

5.3 Advice for firefighters
Wear self-contained breathing apparatus for firefighting if necessary.

5.4 Further information
No data available

SECTION 6: Accidental release measures

6.1 Personal precautions, protective equipment and emergency procedures
Avoid dust formation. Avoid breathing vapours, mist or gas. Ensure adequate ventilation.
Evacuate personnel to safe areas.
For personal protection see section 8.

6.2 Environmental precautions
Prevent further leakage or spillage if safe to do so. Do not let product enter drains.

6.3 Methods and materials for containment and cleaning up
Sweep up and shovel. Contain spillage, and then collect with an electrically protected vacuum cleaner or by wet-brushing and place in container for disposal according to local regulations (see section 13). Do not flush with water. Keep in suitable, closed containers for disposal.

6.4 Reference to other sections
For disposal see section 13.

SECTION 7: Handling and storage

7.1 Precautions for safe handling
Further processing of solid materials may result in the formation of combustible dusts. The potential for combustible dust formation should be taken into consideration before additional processing occurs.
Provide appropriate exhaust ventilation at places where dust is formed.Keep away from sources of ignition - No smoking.
For precautions see section 2.2.

7.2 Conditions for safe storage, including any incompatibilities
Keep container tightly closed in a dry and well-ventilated place.
Never allow product to get in contact with water during storage.

Handle and store under inert gas.
Storage class (TRGS 510): 4.3: Hazardous materials, which set free flammable gases upon contact with water

7.3 Specific end use(s)
Apart from the uses mentioned in section 1.2 no other specific uses are stipulated

SECTION 8: Exposure controls/personal protection

8.1 Control parameters

Components with workplace control parameters

Component	CAS-No.	Value	Control parameters	Basis
Barium	7440-39-3	TWA	0.5 mg/m3	USA. ACGIH Threshold Limit Values (TLV)
	Remarks	Eye irritation Muscular stimulation Skin irritation Gastrointestinal irritation Not classifiable as a human carcinogen		

8.2 Exposure controls

Appropriate engineering controls
Handle in accordance with good industrial hygiene and safety practice. Wash hands before breaks and at the end of workday.

Personal protective equipment

Eye/face protection
Safety glasses with side-shields conforming to EN166 Use equipment for eye protection tested and approved under appropriate government standards such as NIOSH (US) or EN 166(EU).

Skin protection
Handle with gloves. Gloves must be inspected prior to use. Use proper glove removal technique (without touching glove's outer surface) to avoid skin contact with this product. Dispose of contaminated gloves after use in accordance with applicable laws and good laboratory practices. Wash and dry hands.

Full contact
Material: Nitrile rubber
Minimum layer thickness: 0.11 mm
Break through time: 480 min
Material tested:Dermatril® (KCL 740 / Aldrich Z677272, Size M)

Splash contact
Material: Nitrile rubber
Minimum layer thickness: 0.11 mm
Break through time: 480 min
Material tested:Dermatril® (KCL 740 / Aldrich Z677272, Size M)

data source: KCL GmbH, D-36124 Eichenzell, phone +49 (0)6659 87300, e-mail sales@kcl.de, test method: EN374
If used in solution, or mixed with other substances, and under conditions which differ from EN 374, contact the supplier of the CE approved gloves. This recommendation is advisory only and must be evaluated by an industrial hygienist and safety officer familiar with the specific situation of anticipated use by our customers. It should not be construed as offering an approval for any specific use scenario.

Body Protection
Flame retardant protective clothing, The type of protective equipment must be selected according to the concentration and amount of the dangerous substance at the specific workplace.

Respiratory protection
Where risk assessment shows air-purifying respirators are appropriate use a full-face particle respirator type N100 (US) or type P3 (EN 143) respirator cartridges as a backup to engineering controls. If the respirator is the sole means of protection, use a full-face supplied air respirator. Use respirators and components tested and approved under appropriate government standards such as NIOSH (US) or CEN (EU).

Control of environmental exposure
Prevent further leakage or spillage if safe to do so. Do not let product enter drains.

SECTION 9: Physical and chemical properties

9.1 Information on basic physical and chemical properties

a)	Appearance	Form: solid Colour: grey
b)	Odour	No data available
c)	Odour Threshold	No data available
d)	pH	No data available
e)	Melting point/freezing point	Melting point/range: 725 °C (1337 °F)
f)	Initial boiling point and boiling range	1,640 °C 2,984 °F at 1013 hPa
g)	Flash point	()Not applicable
h)	Evaporation rate	No data available
i)	Flammability (solid, gas)	No data available
j)	Upper/lower flammability or explosive limits	No data available
k)	Vapour pressure	No data available
l)	Vapour density	No data available
m)	Relative density	3.600 g/cm3 at 25 °C (77 °F)
n)	Water solubility	No data available
o)	Partition coefficient: n-octanol/water	No data available
p)	Auto-ignition temperature	No data available
q)	Decomposition temperature	No data available
r)	Viscosity	No data available
s)	Explosive properties	No data available

t) Oxidizing properties No data available

9.2 Other safety information
No data available

SECTION 10: Stability and reactivity

10.1 Reactivity
No data available

10.2 Chemical stability
Stable under recommended storage conditions.

10.3 Possibility of hazardous reactions
Reacts violently with water.

10.4 Conditions to avoid
Exposure to moisture

10.5 Incompatible materials
Oxidizing agents, Water, acids, Oxygen, Chlorinated solvents, Carbon dioxide (CO2), Halogens, Halogenated hydrocarbon, Alcohols, Sulphur compounds, Hydrogen sulfide gas

10.6 Hazardous decomposition products
Hazardous decomposition products formed under fire conditions. - Barium oxide
Other decomposition products - No data available
In the event of fire: see section 5

SECTION 11: Toxicological information

11.1 Information on toxicological effects

Acute toxicity
No data available
Inhalation: No data available
Dermal: No data available
No data available

Skin corrosion/irritation
No data available

Serious eye damage/eye irritation
No data available

Respiratory or skin sensitisation
No data available

Germ cell mutagenicity
No data available

Carcinogenicity
This product is or contains a component that is not classifiable as to its carcinogenicity based on its IARC, ACGIH, NTP, or EPA classification.

IARC: No component of this product present at levels greater than or equal to 0.1% is identified as probable, possible or confirmed human carcinogen by IARC.

NTP: No component of this product present at levels greater than or equal to 0.1% is

identified as a known or anticipated carcinogen by NTP.

OSHA: No component of this product present at levels greater than or equal to 0.1% is on OSHA's list of regulated carcinogens.

Reproductive toxicity
No data available
No data available

Specific target organ toxicity - single exposure
No data available

Specific target organ toxicity - repeated exposure
No data available

Aspiration hazard
No data available

Additional Information
RTECS: Not available

Stomach/intestinal disorders, Nausea, Vomiting, Drowsiness, Dizziness, Gastrointestinal disturbance, Weakness, Tremors, Seizures.
To the best of our knowledge, the chemical, physical, and toxicological properties have not been thoroughly investigated.

SECTION 12: Ecological information

12.1 Toxicity

Toxicity to fish	mortality NOEC - Cyprinodon variegatus (sheepshead minnow) - 500 mg/l - 96 h
	LC50 - Cyprinodon variegatus (sheepshead minnow) - > 500 mg/l - 96 h

12.2 Persistence and degradability
No data available

12.3 Bioaccumulative potential
No data available

12.4 Mobility in soil
No data available

12.5 Results of PBT and vPvB assessment
PBT/vPvB assessment not available as chemical safety assessment not required/not conducted

12.6 Other adverse effects
No data available

SECTION 13: Disposal considerations

13.1 Waste treatment methods

Product
Burn in a chemical incinerator equipped with an afterburner and scrubber but exert extra care in igniting as this material is highly flammable. Offer surplus and non-recyclable solutions to a licensed disposal company. Contact a licensed professional waste disposal service to dispose of this material.

Contaminated packaging
Dispose of as unused product.

SECTION 14: Transport information

DOT (US)
UN number: 1400 Class: 4.3 Packing group: II
Proper shipping name: Barium
Reportable Quantity (RQ): 1000 lbs
Poison Inhalation Hazard: No

IMDG
UN number: 1400 Class: 4.3 Packing group: II EMS-No: F-G, S-O
Proper shipping name: BARIUM

IATA
UN number: 1400 Class: 4.3 Packing group: II
Proper shipping name: Barium

SECTION 15: Regulatory information

SARA 302 Components
No chemicals in this material are subject to the reporting requirements of SARA Title III, Section 302.

SARA 313 Components
The following components are subject to reporting levels established by SARA Title III, Section 313:

	CAS-No.	Revision Date
Barium	7440-39-3	2007-07-01

SARA 311/312 Hazards
Reactivity Hazard

Reportable Quantity : D005 lbs

Massachusetts Right To Know Components

	CAS-No.	Revision Date
Barium	7440-39-3	2007-07-01

Pennsylvania Right To Know Components

California Prop. 65 Components
This product does not contain any chemicals known to State of California to cause cancer, birth defects, or any other reproductive harm.

SECTION 16: Other information

Further information

The above information is believed to be correct but does not purport to be all inclusive and shall be used only as a guide. The information in this document is based on the present state of our knowledge and is applicable to the product with regard to appropriate safety precautions. It does not represent any guarantee of the properties of the product. Sigma-Aldrich Corporation and its Affiliates shall not be held liable for any damage resulting from handling or from contact with the above product. See www.sigma-aldrich.com and/or the reverse side of invoice or packing slip for additional terms and conditions of sale.

The branding on the header and/or footer of this document may temporarily not visually match the product purchased as we transition our branding. However, all of the information in the document regarding the product remains unchanged and matches the product ordered. For further information please contact mlsbranding@sial.com.

Version: 6.3 Revision Date: 01/21/2020 Print Date: 06/02/2020

SAFETY DATA SHEET

Version 6.1
Revision Date 10/05/2019
Print Date 05/29/2020

SECTION 1: Identification of the substance/mixture and of the company/undertaking

1.1 Product identifiers

Product name : Benzene

Product Number : 270709
Brand : SIGALD
Index-No. : 601-020-00-8
CAS-No. : 71-43-2

1.2 Relevant identified uses of the substance or mixture and uses advised against

Identified uses : Laboratory chemicals, Synthesis of substances

1.3 Details of the supplier of the safety data sheet

Company : Sigma-Aldrich Inc.
3050 Spruce Street
ST. LOUIS MO 63103
UNITED STATES

Telephone : +1 314 771-5765
Fax : +1 800 325-5052

1.4 Emergency telephone number

Emergency Phone # : +1-703-527-3887

SECTION 2: Hazards identification

2.1 Classification of the substance or mixture

GHS Classification in accordance with 29 CFR 1910 (OSHA HCS)

Flammable liquids (Category 2), H225
Skin irritation (Category 2), H315
Eye irritation (Category 2A), H319
Germ cell mutagenicity (Category 1B), H340
Carcinogenicity (Category 1A), H350
Specific target organ toxicity - repeated exposure (Category 1), Blood, H372
Aspiration hazard (Category 1), H304
Short-term (acute) aquatic hazard (Category 2), H401
Long-term (chronic) aquatic hazard (Category 3), H412

For the full text of the H-Statements mentioned in this Section, see Section 16.

2.2 GHS Label elements, including precautionary statements

Pictogram	
Signal word	Danger

Hazard statement(s)

H225	Highly flammable liquid and vapour.
H304	May be fatal if swallowed and enters airways.
H315	Causes skin irritation.
H319	Causes serious eye irritation.
H340	May cause genetic defects.
H350	May cause cancer.
H372	Causes damage to organs (Blood) through prolonged or repeated exposure.
H401	Toxic to aquatic life.
H412	Harmful to aquatic life with long lasting effects.

Precautionary statement(s)

P201	Obtain special instructions before use.
P202	Do not handle until all safety precautions have been read and understood.
P210	Keep away from heat/sparks/open flames/hot surfaces. No smoking.
P233	Keep container tightly closed.
P240	Ground/bond container and receiving equipment.
P241	Use explosion-proof electrical/ ventilating/ lighting equipment.
P242	Use only non-sparking tools.
P243	Take precautionary measures against static discharge.
P260	Do not breathe dust/ fume/ gas/ mist/ vapours/ spray.
P264	Wash skin thoroughly after handling.
P270	Do not eat, drink or smoke when using this product.
P273	Avoid release to the environment.
P280	Wear protective gloves/ protective clothing/ eye protection/ face protection.
P301 + P310	IF SWALLOWED: Immediately call a POISON CENTER/doctor.
P303 + P361 + P353	IF ON SKIN (or hair): Take off immediately all contaminated clothing. Rinse skin with water/shower.
P305 + P351 + P338	IF IN EYES: Rinse cautiously with water for several minutes. Remove contact lenses, if present and easy to do. Continue rinsing.
P308 + P313	IF exposed or concerned: Get medical advice/ attention.
P331	Do NOT induce vomiting.
P332 + P313	If skin irritation occurs: Get medical advice/ attention.
P337 + P313	If eye irritation persists: Get medical advice/ attention.
P362	Take off contaminated clothing and wash before reuse.
P370 + P378	In case of fire: Use dry sand, dry chemical or alcohol-resistant foam to extinguish.
P403 + P235	Store in a well-ventilated place. Keep cool.
P405	Store locked up.
P501	Dispose of contents/ container to an approved waste disposal plant.

2.3 Hazards not otherwise classified (HNOC) or not covered by GHS - none

SECTION 3: Composition/information on ingredients

3.1 Substances

Formula	:	C_6H_6
Molecular weight	:	78.11 g/mol
CAS-No.	:	71-43-2
EC-No.	:	200-753-7
Index-No.	:	601-020-00-8

Component	Classification	Concentration
Benzene		
	Flam. Liq. 2; Skin Irrit. 2; Eye Irrit. 2A; Muta. 1B; Carc. 1A; STOT RE 1; Asp. Tox. 1; Aquatic Acute 2; Aquatic Chronic 3; H225, H315, H319, H340, H350, H372, H304, H401, H412	<= 100 %

For the full text of the H-Statements mentioned in this Section, see Section 16.

SECTION 4: First aid measures

4.1 Description of first aid measures

General advice
Consult a physician. Show this safety data sheet to the doctor in attendance.Move out of dangerous area.

If inhaled
If breathed in, move person into fresh air. If not breathing, give artificial respiration. Consult a physician.

In case of skin contact
Wash off with soap and plenty of water. Take victim immediately to hospital. Consult a physician.

In case of eye contact
Rinse thoroughly with plenty of water for at least 15 minutes and consult a physician.

If swallowed
Do NOT induce vomiting. Never give anything by mouth to an unconscious person. Rinse mouth with water. Consult a physician.

4.2 Most important symptoms and effects, both acute and delayed

The most important known symptoms and effects are described in the labelling (see section 2.2) and/or in section 11

4.3 Indication of any immediate medical attention and special treatment needed

No data available

SECTION 5: Firefighting measures

5.1 Extinguishing media

Suitable extinguishing media
Dry powder Dry sand

Unsuitable extinguishing media
Do NOT use water jet.

5.2 Special hazards arising from the substance or mixture
Carbon oxides

5.3 Advice for firefighters
Wear self-contained breathing apparatus for firefighting if necessary.

5.4 Further information
Use water spray to cool unopened containers.

SECTION 6: Accidental release measures

6.1 Personal precautions, protective equipment and emergency procedures
Use personal protective equipment. Avoid breathing vapours, mist or gas. Ensure adequate ventilation. Remove all sources of ignition. Evacuate personnel to safe areas. Beware of vapours accumulating to form explosive concentrations. Vapours can accumulate in low areas.
For personal protection see section 8.

6.2 Environmental precautions
Prevent further leakage or spillage if safe to do so. Do not let product enter drains. Discharge into the environment must be avoided.

6.3 Methods and materials for containment and cleaning up
Contain spillage, and then collect with non-combustible absorbent material, (e.g. sand, earth, diatomaceous earth, vermiculite) and place in container for disposal according to local / national regulations (see section 13).

6.4 Reference to other sections
For disposal see section 13.

SECTION 7: Handling and storage

7.1 Precautions for safe handling
Avoid contact with skin and eyes. Avoid inhalation of vapour or mist.
Use explosion-proof equipment.Keep away from sources of ignition - No smoking.Take measures to prevent the build up of electrostatic charge.
For precautions see section 2.2.

7.2 Conditions for safe storage, including any incompatibilities
Keep container tightly closed in a dry and well-ventilated place. Containers which are opened must be carefully resealed and kept upright to prevent leakage.
Storage class (TRGS 510): 3: Flammable liquids

7.3 Specific end use(s)
Apart from the uses mentioned in section 1.2 no other specific uses are stipulated

SECTION 8: Exposure controls/personal protection

8.1 Control parameters

Components with workplace control parameters

Component	CAS-No.	Value	Control parameters	Basis
Benzene	71-43-2	TWA	0.5 ppm	USA. ACGIH Threshold Limit Values (TLV)
	Remarks	Leukemia Substances for which there is a Biological Exposure Index or Indices (see BEI® section) Confirmed human carcinogen Danger of cutaneous absorption		
		STEL	2.5 ppm	USA. ACGIH Threshold Limit Values (TLV)
		Leukemia Substances for which there is a Biological Exposure Index or Indices (see BEI® section) Confirmed human carcinogen Danger of cutaneous absorption		
		TWA	10 ppm	USA. Occupational Exposure Limits (OSHA) - Table Z-2
		Z37.40-1969		
		CEIL	25 ppm	USA. Occupational Exposure Limits (OSHA) - Table Z-2
		Z37.40-1969		
		Peak	50 ppm	USA. Occupational Exposure Limits (OSHA) - Table Z-2
		Z37.40-1969		
		See 1910.1028. See Table Z-2 for the limits applicable in the operations or sectors excluded in 1910.1028 The final benzene standard in 1910.1028 applies to all occupational exposures to benzene except some subsegments of industry where exposures are consistently under the action level (i.e., distribution and sale of fuels, sealed containers and pipelines, coke production, oil and gas drilling and production, natural gas processing, and the percentage exclusion for liquid mixtures); for the excepted subsegments, the benzene limits in Table Z-2 apply.		
		TWA	0.1 ppm	USA. NIOSH Recommended Exposure Limits
		Potential Occupational Carcinogen See Appendix A		
		ST	1 ppm	USA. NIOSH Recommended Exposure Limits
		Potential Occupational Carcinogen See Appendix A		

8.2 Exposure controls

Appropriate engineering controls

Handle in accordance with good industrial hygiene and safety practice. Wash hands before breaks and at the end of workday.

Personal protective equipment

Eye/face protection

Face shield and safety glasses Use equipment for eye protection tested and approved under appropriate government standards such as NIOSH (US) or EN 166(EU).

Skin protection

Handle with gloves. Gloves must be inspected prior to use. Use proper glove removal technique (without touching glove's outer surface) to avoid skin contact with this product. Dispose of contaminated gloves after use in accordance with applicable laws and good laboratory practices. Wash and dry hands.

Full contact
Material: Fluorinated rubber
Minimum layer thickness: 0.7 mm
Break through time: 480 min
Material tested:Vitoject® (KCL 890 / Aldrich Z677698, Size M)

Splash contact
Material: Fluorinated rubber
Minimum layer thickness: 0.7 mm
Break through time: 480 min
Material tested:Vitoject® (KCL 890 / Aldrich Z677698, Size M)

data source: KCL GmbH, D-36124 Eichenzell, phone +49 (0)6659 87300, e-mail sales@kcl.de, test method: EN374
If used in solution, or mixed with other substances, and under conditions which differ from EN 374, contact the supplier of the CE approved gloves. This recommendation is advisory only and must be evaluated by an industrial hygienist and safety officer familiar with the specific situation of anticipated use by our customers. It should not be construed as offering an approval for any specific use scenario.

Handle with gloves. Gloves must be inspected prior to use. Use proper glove removal technique (without touching glove's outer surface) to avoid skin contact with this product. Dispose of contaminated gloves after use in accordance with applicable laws and good laboratory practices. Wash and dry hands.

Body Protection

Complete suit protecting against chemicals, Flame retardant antistatic protective clothing., The type of protective equipment must be selected according to the concentration and amount of the dangerous substance at the specific workplace.

Respiratory protection

Where risk assessment shows air-purifying respirators are appropriate use a full-face respirator with multi-purpose combination (US) or type ABEK (EN 14387) respirator cartridges as a backup to engineering controls. If the respirator is the sole means of protection, use a full-face supplied air respirator. Use respirators and components tested and approved under appropriate government standards such as NIOSH (US) or CEN (EU).

Control of environmental exposure

Prevent further leakage or spillage if safe to do so. Do not let product enter drains. Discharge into the environment must be avoided.

SECTION 9: Physical and chemical properties

9.1 Information on basic physical and chemical properties

a)	Appearance	Form: liquid Colour: colourless
b)	Odour	No data available
c)	Odour Threshold	No data available
d)	pH	No data available
e)	Melting point/freezing point	5.5 °C (41.9 °F)
f)	Initial boiling point and boiling range	80.1 °C 176.2 °F at 1,013 hPa
g)	Flash point	-11.0 °C (12.2 °F) - closed cup
h)	Evaporation rate	No data available
i)	Flammability (solid, gas)	No data available
j)	Upper/lower flammability or explosive limits	Upper explosion limit: 8.0 %(V) Lower explosion limit: 1.4 %(V)
k)	Vapour pressure	221.3 hPa at 37.7 °C (99.9 °F) 99.5 hPa at 20.0 °C(68.0 °F)
l)	Vapour density	No data available
m)	Relative density	0.88 g/cm3
n)	Water solubility	ca.1.88 g/l at 23.5 °C (74.3 °F) - soluble
o)	Partition coefficient: n-octanol/water	log Pow: 2.13 at 25 °C (77 °F) - Bioaccumulation is not expected.
p)	Auto-ignition temperature	498 °C (928 °F) at 1,013.5 hPa
q)	Decomposition temperature	No data available
r)	Viscosity	0.78 mm2/s at 20 °C (68 °F) -
s)	Explosive properties	No data available
t)	Oxidizing properties	No data available

9.2 Other safety information

No data available

SECTION 10: Stability and reactivity

10.1 Reactivity

No data available

10.2 Chemical stability

Stable under recommended storage conditions.

10.3 Possibility of hazardous reactions
Vapours may form explosive mixture with air.

10.4 Conditions to avoid
Heat, flames and sparks.

10.5 Incompatible materials
acids, Bases, Halogens, Strong oxidizing agents, Metallic salts

10.6 Hazardous decomposition products
Hazardous decomposition products formed under fire conditions. - Carbon oxides
Other decomposition products - No data available
In the event of fire: see section 5

SECTION 11: Toxicological information

11.1 Information on toxicological effects

Acute toxicity
LD50 Oral - Rat - male - > 2,000 mg/kg
(OECD Test Guideline 401)
LD50 Oral - Rat - male - 5,970 mg/kg
(OECD Test Guideline 401)
LC50 Inhalation - Rat - female - 4 h - 43.7 mg/l
(OECD Test Guideline 403)
LD50 Dermal - Rabbit - male and female - > 8,260 mg/kg
(OECD Test Guideline 402)
No data available

Skin corrosion/irritation
Skin - Rabbit
Result: Skin irritation - 4 h
(OECD Test Guideline 404)
Drying-out effect resulting in rough and chapped skin.

Serious eye damage/eye irritation
Eyes - Rabbit
Result: Eye irritation
Remarks: (ECHA)

Respiratory or skin sensitisation
Maximisation Test - Guinea pig
Result: Does not cause skin sensitisation.
(OECD Test Guideline 406)

Germ cell mutagenicity
May cause genetic defects.
Ames test
Salmonella typhimurium
Result: negative
In vitro mammalian cell gene mutation test
Result: negative
(ECHA)
Mutagenicity (mammal cell test): chromosome aberration.
Chinese hamster lung cells
Result: positive
OECD Test Guideline 474

Mouse - male - Bone marrow
Result: positive

Carcinogenicity
May cause cancer. Positive evidence from human epidemiological studies.

IARC: No component of this product present at levels greater than or equal to 0.1% is identified as probable, possible or confirmed human carcinogen by IARC.

NTP: No component of this product present at levels greater than or equal to 0.1% is identified as a known or anticipated carcinogen by NTP.

OSHA: No component of this product present at levels greater than or equal to 0.1% is on OSHA's list of regulated carcinogens.

Reproductive toxicity
No data available

Specific target organ toxicity - single exposure
No data available
Acute oral toxicity - Nausea
Acute inhalation toxicity - Possible damages:, mucosal irritations

Specific target organ toxicity - repeated exposure
Causes damage to organs through prolonged or repeated exposure. - Blood

Aspiration hazard
May be fatal if swallowed and enters airways.
Aspiration hazard, Aspiration may cause pulmonary oedema and pneumonitis.

Additional Information

Repeated dose toxicity - Rat - male and female - Oral - 120 d - No observed adverse effect level - 100 mg/kg - Lowest observed adverse effect level - 25 mg/kg
Subchronic toxicity
RTECS: CY1400000

Nausea, Dizziness, Headache, narcosis, Inhalation of high concentrations of benzene may have an initial stimulatory effect on the central nervous system characterized by exhilaration, nervous excitation and/or giddiness, depression, drowsiness, or fatigue. The victim may experience tightness in the chest, breathlessness, and loss of consciousness. Tremors, convulsions, and death due to respiratory paralysis or circulatory collapse can occur in a few minutes to several hours following severe exposures. Aspiration of small amounts of liquid immediately causes pulmonary edema and hemorrhage of pulmonary tissue. Direct skin contact may cause erythema. Repeated or prolonged skin contact may result in drying, scaling dermatitis, or development of secondary skin infections. The chief target organ is the hematopoietic system. Bleeding from the nose, gums, or mucous membranes and the development of purpuric spots, pancytopenia, leukopenia, thrombocytopenia, aplastic anemia, and leukemia may occur as the condition progresses. The bone marrow may appear normal, aplastic or hyperplastic, and may not correlate with peripheral blood-forming tissues. The onset of effects of prolonged benzene exposure may be delayed for many months or years after the actual exposure has ceased., Blood disorders
To the best of our knowledge, the chemical, physical, and toxicological properties have not been thoroughly investigated.

agitation, Headache, Dizziness, inebriation, Tiredness, CNS disorders, narcosis, respiratory arrest

Stomach - Irregularities - Based on Human Evidence
Stomach - Irregularities - Based on Human Evidence

SECTION 12: Ecological information

12.1 Toxicity

Toxicity to fish	flow-through test LC50 - Oncorhynchus mykiss (rainbow trout) - 5.3 mg/l - 96 h (OECD Test Guideline 203)
Toxicity to daphnia and other aquatic invertebrates	static test EC50 - Daphnia magna (Water flea) - 10 mg/l - 48 h (OECD Test Guideline 202)
Toxicity to algae	static test EC50 - Pseudokirchneriella subcapitata (green algae) - 100 mg/l - 72 h (OECD Test Guideline 201)
Toxicity to bacteria	static test IC50 - - 13 mg/l - 24 h Remarks: (ECHA)

12.2 Persistence and degradability

Biodegradability	aerobic - Exposure time 28 d Result: 96 % - Readily biodegradable. (OECD Test Guideline 301F)
Theoretical oxygen demand	3,100 mg/g Remarks: (Lit.)
Ratio BOD/ThBOD	71 % Remarks: (Lit.)
Ratio BOD/ThBOD	80 % Remarks: (Lit.)

12.3 Bioaccumulative potential

Bioaccumulation	Leuciscus idus (Golden orfe) - 3 d - 0.05 mg/l(Benzene) Bioconcentration factor (BCF): 10

12.4 Mobility in soil

No data available

12.5 Results of PBT and vPvB assessment

PBT/vPvB assessment not available as chemical safety assessment not required/not conducted

12.6 Other adverse effects

An environmental hazard cannot be excluded in the event of unprofessional handling or disposal.
Toxic to aquatic life.
Endangers drinking-water supplies if allowed to enter soil or water.
Discharge into the environment must be avoided.

SECTION 13: Disposal considerations

13.1 Waste treatment methods

Product
Offer surplus and non-recyclable solutions to a licensed disposal company. Burn in a chemical incinerator equipped with an afterburner and scrubber but exert extra care in igniting as this material is highly flammable. Contact a licensed professional waste disposal service to dispose of this material.

Contaminated packaging
Dispose of as unused product.

SECTION 14: Transport information

DOT (US)
UN number: 1114 Class: 3 Packing group: II
Proper shipping name: Benzene
Reportable Quantity (RQ): 10 lbs
Reportable Quantity (RQ): 10 lbs
Poison Inhalation Hazard: No

IMDG
UN number: 1114 Class: 3 Packing group: II EMS-No: F-E, S-D
Proper shipping name: BENZENE

IATA
UN number: 1114 Class: 3 Packing group: II
Proper shipping name: Benzene

SECTION 15: Regulatory information

SARA 302 Components
No chemicals in this material are subject to the reporting requirements of SARA Title III, Section 302.

SARA 313 Components
The following components are subject to reporting levels established by SARA Title III, Section 313:

	CAS-No.	Revision Date
Benzene	71-43-2	2007-07-01

SARA 311/312 Hazards
Fire Hazard, Acute Health Hazard, Chronic Health Hazard

Reportable Quantity : D018 lbs

Massachusetts Right To Know Components
No components are subject to the Massachusetts Right to Know Act.

Pennsylvania Right To Know Components

	CAS-No.	Revision Date
Benzene		

SECTION 16: Other information

Further information

The above information is believed to be correct but does not purport to be all inclusive and shall be used only as a guide. The information in this document is based on the present state of our knowledge and is applicable to the product with regard to appropriate safety precautions. It does not represent any guarantee of the properties of the product. Sigma-Aldrich Corporation and its Affiliates shall not be held liable for any damage resulting from handling or from contact with the above product. See www.sigma-aldrich.com and/or the reverse side of invoice or packing slip for additional terms and conditions of sale.

The branding on the header and/or footer of this document may temporarily not visually match the product purchased as we transition our branding. However, all of the information in the document regarding the product remains unchanged and matches the product ordered. For further information please contact mlsbranding@sial.com.

Version: 6.1 Revision Date: 10/05/2019 Print Date: 05/29/2020

SAFETY DATA SHEET

Version 6.6
Revision Date 10/08/2021
Print Date 04/09/2022

SECTION 1: Identification of the substance/mixture and of the company/undertaking

1.1 Product identifiers

Product name : Cadmium

Product Number : 265330
Brand : Aldrich
Index-No. : 048-002-00-0
CAS-No. : 7440-43-9

1.2 Relevant identified uses of the substance or mixture and uses advised against

Identified uses : Laboratory chemicals, Synthesis of substances

1.3 Details of the supplier of the safety data sheet

Company : Sigma-Aldrich Inc.
3050 SPRUCE ST
ST. LOUIS MO 63103
UNITED STATES

Telephone : +1 314 771-5765
Fax : +1 800 325-5052

1.4 Emergency telephone

Emergency Phone # : 800-424-9300 CHEMTREC (USA) +1-703-527-3887 CHEMTREC (International) 24 Hours/day; 7 Days/week

SECTION 2: Hazards identification

2.1 Classification of the substance or mixture

GHS Classification in accordance with 29 CFR 1910 (OSHA HCS)

Acute toxicity, Oral (Category 3), H301
Acute toxicity, Inhalation (Category 2), H330
Germ cell mutagenicity (Category 2), H341
Carcinogenicity (Category 1B), H350
Reproductive toxicity (Category 2), H361
Specific target organ toxicity - repeated exposure (Category 1), H372
Short-term (acute) aquatic hazard (Category 1), H400
Long-term (chronic) aquatic hazard (Category 1), H410

For the full text of the H-Statements mentioned in this Section, see Section 16.

2.2 GHS Label elements, including precautionary statements

Pictogram

Signal word Danger

Hazard statement(s)

H301	Toxic if swallowed.
H330	Fatal if inhaled.
H341	Suspected of causing genetic defects.
H350	May cause cancer.
H361	Suspected of damaging fertility or the unborn child.
H372	Causes damage to organs through prolonged or repeated exposure.
H410	Very toxic to aquatic life with long lasting effects.

Precautionary statement(s)

P201	Obtain special instructions before use.
P202	Do not handle until all safety precautions have been read and understood.
P260	Do not breathe dust/ fume/ gas/ mist/ vapors/ spray.
P264	Wash skin thoroughly after handling.
P270	Do not eat, drink or smoke when using this product.
P271	Use only outdoors or in a well-ventilated area.
P273	Avoid release to the environment.
P280	Wear protective gloves/ protective clothing/ eye protection/ face protection.
P284	Wear respiratory protection.
P301 + P310 + P330	IF SWALLOWED: Immediately call a POISON CENTER/ doctor. Rinse mouth.
P304 + P340 + P310	IF INHALED: Remove person to fresh air and keep comfortable for breathing. Immediately call a POISON CENTER/ doctor.
P308 + P313	IF exposed or concerned: Get medical advice/ attention.
P391	Collect spillage.
P403 + P233	Store in a well-ventilated place. Keep container tightly closed.
P405	Store locked up.
P501	Dispose of contents/ container to an approved waste disposal plant.

2.3 Hazards not otherwise classified (HNOC) or not covered by GHS - none

SECTION 3: Composition/information on ingredients

3.1 Substances

Formula	:	Cd
Molecular weight	:	112.41 g/mol
CAS-No.	:	7440-43-9
EC-No.	:	231-152-8
Index-No.	:	048-002-00-0

Component	Classification	Concentration
Cadmium		
	Acute Tox. 2; Muta. 2; Carc. 1B; Repr. 2; STOT RE 1; Aquatic Acute 1;	<= 100 %

	Aquatic Chronic 1; H330, H341, H350, H361, H372, H400, H410 M-Factor - Aquatic Acute: 1,000	

For the full text of the H-Statements mentioned in this Section, see Section 16.

SECTION 4: First aid measures

4.1 Description of first-aid measures

General advice
Consult a physician. Show this material safety data sheet to the doctor in attendance.Move out of dangerous area.

If inhaled
If breathed in, move person into fresh air. If not breathing, give artificial respiration. Consult a physician.

In case of skin contact
Wash off with soap and plenty of water. Take victim immediately to hospital. Consult a physician.

In case of eye contact
Flush eyes with water as a precaution.

If swallowed
Never give anything by mouth to an unconscious person. Rinse mouth with water. Consult a physician.

4.2 Most important symptoms and effects, both acute and delayed
The most important known symptoms and effects are described in the labelling (see section 2.2) and/or in section 11

4.3 Indication of any immediate medical attention and special treatment needed
No data available

SECTION 5: Firefighting measures

5.1 Extinguishing media

Suitable extinguishing media
Use water spray, alcohol-resistant foam, dry chemical or carbon dioxide.

5.2 Special hazards arising from the substance or mixture
Cadmium/cadmium oxides

5.3 Advice for firefighters
Wear self-contained breathing apparatus for firefighting if necessary.

5.4 Further information
No data available

SECTION 6: Accidental release measures

6.1 Personal precautions, protective equipment and emergency procedures

Use personal protective equipment. Avoid dust formation. Avoid breathing vapors, mist or gas. Ensure adequate ventilation. Evacuate personnel to safe areas. Avoid breathing dust. For personal protection see section 8.

6.2 Environmental precautions

Prevent further leakage or spillage if safe to do so. Do not let product enter drains. Discharge into the environment must be avoided.

6.3 Methods and materials for containment and cleaning up

Pick up and arrange disposal without creating dust. Sweep up and shovel. Keep in suitable, closed containers for disposal.

6.4 Reference to other sections

For disposal see section 13.

SECTION 7: Handling and storage

7.1 Precautions for safe handling

Advice on safe handling

Avoid contact with skin and eyes. Avoid formation of dust and aerosols.**Advice on safe handling**

Further processing of solid materials may result in the formation of combustible dusts. The potential for combustible dust formation should be taken into consideration before additional processing occurs.

Advice on protection against fire and explosion

Provide appropriate exhaust ventilation at places where dust is formed.

Hygiene measures

Handle in accordance with good industrial hygiene and safety practice. Wash hands before breaks and at the end of workday.
For precautions see section 2.2.

7.2 Conditions for safe storage, including any incompatibilities

Storage conditions

Keep container tightly closed in a dry and well-ventilated place.

Air sensitive.

Storage class

Storage class (TRGS 510): 6.1A: Combustible, acute toxic Cat. 1 and 2 / very toxic hazardous materials

7.3 Specific end use(s)

Apart from the uses mentioned in section 1.2 no other specific uses are stipulated

SECTION 8: Exposure controls/personal protection

8.1 Control parameters

Ingredients with workplace control parameters

Component	CAS-No.	Value	Control parameters	Basis
Cadmium	7440-43-9	TWA	0.1 mg/m3	USA. Occupational Exposure Limits (OSHA) - Table Z-2
		TWA	0.2 mg/m3	USA. Occupational Exposure Limits (OSHA) - Table Z-2
		CEIL	0.3 mg/m3	USA. Occupational Exposure Limits (OSHA) - Table Z-2
		CEIL	0.6 mg/m3	USA. Occupational Exposure Limits (OSHA) - Table Z-2
		PEL	0.005 mg/m3	California permissible exposure limits for chemical contaminants (Title 8, Article 107)
	Remarks	Potential Occupational Carcinogen		
		TWA	0.01 mg/m3	USA. ACGIH Threshold Limit Values (TLV)
		Suspected human carcinogen		
		TWA	0.002 mg/m3	USA. ACGIH Threshold Limit Values (TLV)
		Suspected human carcinogen		
		PEL	0.005 mg/m3	OSHA Specifically Regulated Chemicals/Carcinogens
		OSHA specifically regulated carcinogen		

Biological occupational exposure limits

Component	CAS-No.	Parameters	Value	Biological specimen	Basis
Cadmium	7440-43-9	cadmium	5 µg/l	In blood	ACGIH - Biological Exposure Indices (BEI)
	Remarks	Not critical			
		cadmium	5µg/g creatinine	Urine	ACGIH - Biological Exposure Indices (BEI)
		Not critical			

8.2 Exposure controls

Appropriate engineering controls
Handle in accordance with good industrial hygiene and safety practice. Wash hands before breaks and at the end of workday.

Personal protective equipment

Eye/face protection
Face shield and safety glasses Use equipment for eye protection tested and approved under appropriate government standards such as NIOSH (US) or EN 166(EU).

Skin protection
Handle with gloves. Gloves must be inspected prior to use. Use proper glove removal technique (without touching glove's outer surface) to avoid skin contact

with this product. Dispose of contaminated gloves after use in accordance with applicable laws and good laboratory practices. Wash and dry hands.

Full contact
Material: Nitrile rubber
Minimum layer thickness: 0.11 mm
Break through time: 480 min
Material tested:Dermatril® (KCL 740 / Aldrich Z677272, Size M)

Splash contact
Material: Nitrile rubber
Minimum layer thickness: 0.11 mm
Break through time: 480 min
Material tested:Dermatril® (KCL 740 / Aldrich Z677272, Size M)

data source: KCL GmbH, D-36124 Eichenzell, phone +49 (0)6659 87300, e-mail sales@kcl.de, test method: EN374
If used in solution, or mixed with other substances, and under conditions which differ from EN 374, contact the supplier of the EC approved gloves. This recommendation is advisory only and must be evaluated by an industrial hygienist and safety officer familiar with the specific situation of anticipated use by our customers. It should not be construed as offering an approval for any specific use scenario.

Body Protection
Complete suit protecting against chemicals, The type of protective equipment must be selected according to the concentration and amount of the dangerous substance at the specific workplace.

Respiratory protection
Where risk assessment shows air-purifying respirators are appropriate use a full-face particle respirator type N99 (US) or type P2 (EN 143) respirator cartridges as a backup to engineering controls. If the respirator is the sole means of protection, use a full-face supplied air respirator. Use respirators and components tested and approved under appropriate government standards such as NIOSH (US) or CEN (EU).

Control of environmental exposure
Prevent further leakage or spillage if safe to do so. Do not let product enter drains. Discharge into the environment must be avoided.

SECTION 9: Physical and chemical properties

9.1 Information on basic physical and chemical properties

a)	Appearance	Form: granular Color: metallic
b)	Odor	odorless
c)	Odor Threshold	No data available
d)	pH	No data available
e)	Melting point/freezing point	Melting point/range: 320.9 °C (609.6 °F) - lit.
f)	Initial boiling point and boiling range	765 °C 1409 °F - lit.
g)	Flash point	()Not applicable

h)	Evaporation rate	No data available
i)	Flammability (solid, gas)	No data available
j)	Upper/lower flammability or explosive limits	No data available
k)	Vapor pressure	1.3 hPa at 394 °C (741 °F)
l)	Vapor density	No data available
m)	Density	8.65 g/cm3 at 25 °C (77 °F) - lit.
	Relative density	8.6 at 22 °C (72 °F) - Regulation (EC) No. 440/2008, Annex, A.3
n)	Water solubility	2.3 g/l at 20 °C (68 °F) - OECD Test Guideline 105 - slightly soluble
o)	Partition coefficient: n-octanol/water	Not applicable for inorganic substances
p)	Autoignition temperature	No data available
q)	Decomposition temperature	No data available
r)	Viscosity	No data available
s)	Explosive properties	No data available
t)	Oxidizing properties	none

9.2 Other safety information
No data available

SECTION 10: Stability and reactivity

10.1 Reactivity
No data available

10.2 Chemical stability
Stable under recommended storage conditions.

10.3 Possibility of hazardous reactions
No data available

10.4 Conditions to avoid
No data available

10.5 Incompatible materials
Strong oxidizing agents, acids

10.6 Hazardous decomposition products
In the event of fire: see section 5

SECTION 11: Toxicological information

11.1 Information on toxicological effects

Acute toxicity
Oral: No data available
LC50 Inhalation - Rat - male - 4 h - > 0.5638 mg/l
Remarks: (ECHA)
Dermal: No data available
No data available

Skin corrosion/irritation
No data available

Serious eye damage/eye irritation
No data available

Respiratory or skin sensitization
No data available

Germ cell mutagenicity
Suspected of causing genetic defects.

Carcinogenicity
Presumed to have carcinogenic potential for humans

IARC: 1 - Group 1: Carcinogenic to humans (Cadmium)

NTP: No ingredient of this product present at levels greater than or equal to 0.1% is identified as a known or anticipated carcinogen by NTP.

OSHA: No component of this product present at levels greater than or equal to 0.1% is on OSHA's list of regulated carcinogens.

Reproductive toxicity
Suspected of damaging the unborn child.
Suspected of damaging fertility.

Specific target organ toxicity - single exposure
No data available

Specific target organ toxicity - repeated exposure
Causes damage to organs through prolonged or repeated exposure.**Aspiration hazard**
No data available

11.2 Additional Information

RTECS: EU9800000
Damage to the lungs., Kidney injury may occur., prolonged or repeated exposure can cause:, Vomiting, Diarrhea, Lung irritation
To the best of our knowledge, the chemical, physical, and toxicological properties have not been thoroughly investigated.

Stomach - Irregularities - Based on Human Evidence

Stomach - Irregularities - Based on Human Evidence

SECTION 12: Ecological information

12.1 Toxicity

Toxicity to fish	flow-through test LC50 - Pimephales promelas (fathead minnow) - 1.5 mg/l - 96 h Remarks: (ECHA)
Toxicity to daphnia and other aquatic invertebrates	static test LC50 - Daphnia magna (Water flea) - 0.11 mg/l - 48 h (OECD Test Guideline 202)
Toxicity to bacteria	static test NOEC - activated sludge - 0.2 mg/l - 3 h (OECD Test Guideline 209)

12.2 Persistence and degradability

The methods for determining the biological degradability are not applicable to inorganic substances.

12.3 Bioaccumulative potential

Bioaccumulation	Oncorhynchus mykiss (rainbow trout) - 72 d - 1.27 µg/l(Cadmium) Bioconcentration factor (BCF): 55

12.4 Mobility in soil

No data available

12.5 Results of PBT and vPvB assessment

PBT/vPvB assessment not available as chemical safety assessment not required/not conducted

12.6 Endocrine disrupting properties

No data available

12.7 Other adverse effects

An environmental hazard cannot be excluded in the event of unprofessional handling or disposal.
Very toxic to aquatic life with long lasting effects.

SECTION 13: Disposal considerations

13.1 Waste treatment methods

Product

Offer surplus and non-recyclable solutions to a licensed disposal company. Contact a licensed professional waste disposal service to dispose of this material. Dissolve or mix the material with a combustible solvent and burn in a chemical incinerator equipped with an afterburner and scrubber.

Contaminated packaging

Dispose of as unused product.

SECTION 14: Transport information

DOT (US)
UN number: 3288 Class: 6.1 Packing group: II
Proper shipping name: Toxic solid, inorganic, n.o.s. (Cadmium)
Reportable Quantity (RQ): 10 lbs
Reportable Quantity (RQ): 10 lbs
Poison Inhalation Hazard: No

IMDG
UN number: 3288 Class: 6.1 Packing group: II EMS-No: F-A, S-A
Proper shipping name: TOXIC SOLID, INORGANIC, N.O.S. (Cadmium)
Marine pollutant : yes

IATA
UN number: 3288 Class: 6.1 Packing group: II
Proper shipping name: Toxic solid, inorganic, n.o.s. (Cadmium)

SECTION 15: Regulatory information

SARA 302 Components
This material does not contain any components with a section 302 EHS TPQ.

SARA 313 Components
The following components are subject to reporting levels established by SARA Title III, Section 313:

	CAS-No.	Revision Date
Cadmium	7440-43-9	2007-07-01

SARA 311/312 Hazards
Acute Health Hazard, Chronic Health Hazard

Reportable Quantity : D006 lbs

Massachusetts Right To Know Components
No components are subject to the Massachusetts Right to Know Act.

SECTION 16: Other information

Further information

The above information is believed to be correct but does not purport to be all inclusive and shall be used only as a guide. The information in this document is based on the present state of our knowledge and is applicable to the product with regard to appropriate safety precautions. It does not represent any guarantee of the properties of the product. Sigma-Aldrich Corporation and its Affiliates shall not be held liable for any damage resulting from handling or from contact with the above product. See www.sigma-aldrich.com and/or the reverse side of invoice or packing slip for additional terms and conditions of sale.

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Version: 6.6 Revision Date: 10/08/2021 Print Date: 04/09/2022

SAFETY DATA SHEET

Version 6.1
Revision Date 01/15/2020
Print Date 05/29/2020

SECTION 1: Identification of the substance/mixture and of the company/undertaking

1.1 Product identifiers

Product name : Chromium

Product Number : 266299
Brand : Aldrich
CAS-No. : 7440-47-3

1.2 Relevant identified uses of the substance or mixture and uses advised against

Identified uses : Laboratory chemicals, Synthesis of substances

1.3 Details of the supplier of the safety data sheet

Company : Sigma-Aldrich Inc.
3050 Spruce Street
ST. LOUIS MO 63103
UNITED STATES

Telephone : +1 314 771-5765
Fax : +1 800 325-5052

1.4 Emergency telephone number

Emergency Phone # : +1-703-527-3887

SECTION 2: Hazards identification

2.1 Classification of the substance or mixture

Not a hazardous substance or mixture.

2.2 GHS Label elements, including precautionary statements

Not a hazardous substance or mixture.

2.3 Hazards not otherwise classified (HNOC) or not covered by GHS - none

SECTION 3: Composition/information on ingredients

3.1 Substances

Formula : Cr
Molecular weight : 52.00 g/mol
CAS-No. : 7440-47-3
EC-No. : 231-157-5

Component	Classification	Concentration

Chromium		
		<= 100 %

SECTION 4: First aid measures

4.1 Description of first aid measures

General advice
Move out of dangerous area.

If inhaled
If breathed in, move person into fresh air. If not breathing, give artificial respiration.

In case of skin contact
Wash off with soap and plenty of water.

In case of eye contact
Flush eyes with water as a precaution.

If swallowed
Never give anything by mouth to an unconscious person. Rinse mouth with water.

4.2 Most important symptoms and effects, both acute and delayed

The most important known symptoms and effects are described in the labelling (see section 2.2) and/or in section 11

4.3 Indication of any immediate medical attention and special treatment needed

No data available

SECTION 5: Firefighting measures

5.1 Extinguishing media

Suitable extinguishing media
Use water spray, alcohol-resistant foam, dry chemical or carbon dioxide.

5.2 Special hazards arising from the substance or mixture

Chromium oxides

5.3 Advice for firefighters

Wear self-contained breathing apparatus for firefighting if necessary.

5.4 Further information

No data available

SECTION 6: Accidental release measures

6.1 Personal precautions, protective equipment and emergency procedures

Avoid dust formation. Avoid breathing vapours, mist or gas.
For personal protection see section 8.

6.2 Environmental precautions

No special environmental precautions required.

6.3 Methods and materials for containment and cleaning up
Sweep up and shovel. Keep in suitable, closed containers for disposal.

6.4 Reference to other sections
For disposal see section 13.

SECTION 7: Handling and storage

7.1 Precautions for safe handling
Further processing of solid materials may result in the formation of combustible dusts. The potential for combustible dust formation should be taken into consideration before additional processing occurs.
Provide appropriate exhaust ventilation at places where dust is formed.
For precautions see section 2.2.

7.2 Conditions for safe storage, including any incompatibilities
Keep container tightly closed in a dry and well-ventilated place.

Air sensitive. Keep in a dry place.
Storage class (TRGS 510): 13: Non Combustible Solids

7.3 Specific end use(s)
Apart from the uses mentioned in section 1.2 no other specific uses are stipulated

SECTION 8: Exposure controls/personal protection

8.1 Control parameters

Components with workplace control parameters

Component	CAS-No.	Value	Control parameters	Basis
Chromium	7440-47-3	TWA	0.5 mg/m3	USA. ACGIH Threshold Limit Values (TLV)
	Remarks	respiratory tract irritation 2018 Adoption		
		PEL	0.5 mg/m3	California permissible exposure limits for chemical contaminants (Title 8, Article 107)
		see Sections 1532.2, 5206 & 8359		
		TWA	1 mg/m3	USA. Occupational Exposure Limits (OSHA) - Table Z-1 Limits for Air Contaminants

8.2 Exposure controls

Appropriate engineering controls
General industrial hygiene practice.

Personal protective equipment

Eye/face protection
Use equipment for eye protection tested and approved under appropriate government standards such as NIOSH (US) or EN 166(EU).

Skin protection
Handle with gloves. Gloves must be inspected prior to use. Use proper glove removal technique (without touching glove's outer surface) to avoid skin contact with this product. Dispose of contaminated gloves after use in accordance with applicable laws and good laboratory practices. Wash and dry hands.

Full contact
Material: Nitrile rubber
Minimum layer thickness: 0.11 mm
Break through time: 480 min
Material tested:Dermatril® (KCL 740 / Aldrich Z677272, Size M)

Splash contact
Material: Nitrile rubber
Minimum layer thickness: 0.11 mm
Break through time: 480 min
Material tested:Dermatril® (KCL 740 / Aldrich Z677272, Size M)

data source: KCL GmbH, D-36124 Eichenzell, phone +49 (0)6659 87300, e-mail sales@kcl.de, test method: EN374
If used in solution, or mixed with other substances, and under conditions which differ from EN 374, contact the supplier of the CE approved gloves. This recommendation is advisory only and must be evaluated by an industrial hygienist and safety officer familiar with the specific situation of anticipated use by our customers. It should not be construed as offering an approval for any specific use scenario.

Body Protection
Choose body protection in relation to its type, to the concentration and amount of dangerous substances, and to the specific work-place., The type of protective equipment must be selected according to the concentration and amount of the dangerous substance at the specific workplace.

Respiratory protection
Respiratory protection is not required. Where protection from nuisance levels of dusts are desired, use type N95 (US) or type P1 (EN 143) dust masks. Use respirators and components tested and approved under appropriate government standards such as NIOSH (US) or CEN (EU).

Control of environmental exposure
No special environmental precautions required.

SECTION 9: Physical and chemical properties

9.1 Information on basic physical and chemical properties

a)	Appearance	Form: powder Colour: light grey
b)	Odour	odourless
c)	Odour Threshold	No data available
d)	pH	No data available
e)	Melting point/freezing point	Melting point/range: 1,857 °C (3,375 °F) - lit.
f)	Initial boiling point and boiling range	2,672 °C 4,842 °F - lit.

g)	Flash point	()Not applicable
h)	Evaporation rate	No data available
i)	Flammability (solid, gas)	No data available
j)	Upper/lower flammability or explosive limits	No data available
k)	Vapour pressure	No data available
l)	Vapour density	No data available
m)	Relative density	7.14 g/mL at 25 °C (77 °F)
n)	Water solubility	insoluble
o)	Partition coefficient: n-octanol/water	Not applicable for inorganic substances
p)	Auto-ignition temperature	No data available
q)	Decomposition temperature	No data available
r)	Viscosity	No data available
s)	Explosive properties	No data available
t)	Oxidizing properties	No data available

9.2 Other safety information
No data available

SECTION 10: Stability and reactivity

10.1 Reactivity
No data available

10.2 Chemical stability
Stable under recommended storage conditions.

10.3 Possibility of hazardous reactions
No data available

10.4 Conditions to avoid
No data available

10.5 Incompatible materials
Strong acids, Strong oxidizing agents

10.6 Hazardous decomposition products
Hazardous decomposition products formed under fire conditions. - Chromium oxides
Other decomposition products - No data available
In the event of fire: see section 5

SECTION 11: Toxicological information

11.1 Information on toxicological effects

Acute toxicity
No data available
Inhalation: No data available
Dermal: No data available
No data available

Skin corrosion/irritation
No data available

Serious eye damage/eye irritation
No data available

Respiratory or skin sensitisation
No data available

Germ cell mutagenicity
No data available

Carcinogenicity

IARC: No component of this product present at levels greater than or equal to 0.1% is identified as probable, possible or confirmed human carcinogen by IARC.

NTP: No component of this product present at levels greater than or equal to 0.1% is identified as a known or anticipated carcinogen by NTP.

OSHA: No component of this product present at levels greater than or equal to 0.1% is on OSHA's list of regulated carcinogens.

Reproductive toxicity
No data available

Specific target organ toxicity - single exposure
No data available

Specific target organ toxicity - repeated exposure
No data available

Aspiration hazard
No data available

Additional Information
RTECS: GB4200000

To the best of our knowledge, the chemical, physical, and toxicological properties have not been thoroughly investigated.

Stomach - Irregularities - Based on Human Evidence
Stomach - Irregularities - Based on Human Evidence

SECTION 12: Ecological information

12.1 Toxicity

Toxicity to fish	LC50 - Cyprinus carpio (Carp) - 14.3 mg/l - 96 h
Toxicity to daphnia	EC50 - Daphnia magna (Water flea) - 0.07 mg/l - 48 h

and other aquatic invertebrates

12.2 Persistence and degradability
The methods for determining biodegradability are not applicable to inorganic substances.

12.3 Bioaccumulative potential
Bioaccumulation — Oncorhynchus mykiss (rainbow trout) - 30 d - 50 µg/l(Chromium)

Bioconcentration factor (BCF): 1.03 - 1.22

12.4 Mobility in soil
No data available

12.5 Results of PBT and vPvB assessment
PBT/vPvB assessment not available as chemical safety assessment not required/not conducted

12.6 Other adverse effects
No data available

SECTION 13: Disposal considerations

13.1 Waste treatment methods

Product
Offer surplus and non-recyclable solutions to a licensed disposal company.

Contaminated packaging
Dispose of as unused product.

SECTION 14: Transport information

DOT (US)
UN number: 3077 Class: 9 Packing group: III
Proper shipping name: Environmentally hazardous substance, solid, n.o.s. (Chromium)
Reportable Quantity (RQ): 5000 lbs
Reportable Quantity (RQ): 10 lbs
Poison Inhalation Hazard: No

IMDG
Not dangerous goods

IATA
Not dangerous goods

SECTION 15: Regulatory information

SARA 302 Components
This material does not contain any components with a section 302 EHS TPQ.

SARA 313 Components

The following components are subject to reporting levels established by SARA Title III, Section 313:

	CAS-No.	Revision Date
Chromium	7440-47-3	2007-07-01

SARA 311/312 Hazards
Chronic Health Hazard

Reportable Quantity : D007 lbs

Massachusetts Right To Know Components
No components are subject to the Massachusetts Right to Know Act.

Pennsylvania Right To Know Components

	CAS-No.	Revision Date
Chromium	7440-47-3	2007-07-01

SECTION 16: Other information

Further information

The above information is believed to be correct but does not purport to be all inclusive and shall be used only as a guide. The information in this document is based on the present state of our knowledge and is applicable to the product with regard to appropriate safety precautions. It does not represent any guarantee of the properties of the product. Sigma-Aldrich Corporation and its Affiliates shall not be held liable for any damage resulting from handling or from contact with the above product. See www.sigma-aldrich.com and/or the reverse side of invoice or packing slip for additional terms and conditions of sale.

The branding on the header and/or footer of this document may temporarily not visually match the product purchased as we transition our branding. However, all of the information in the document regarding the product remains unchanged and matches the product ordered. For further information please contact mlsbranding@sial.com.

Version: 6.1 Revision Date: 01/15/2020 Print Date: 05/29/2020

SAFETY DATA SHEET

Version 6.1
Revision Date 01/15/2020
Print Date 05/29/2020

SECTION 1: Identification of the substance/mixture and of the company/undertaking

1.1 Product identifiers

Product name : Ethylbenzene

Product Number : 296848
Brand : Sigma-Aldrich
Index-No. : 601-023-00-4
CAS-No. : 100-41-4

1.2 Relevant identified uses of the substance or mixture and uses advised against

Identified uses : Laboratory chemicals, Synthesis of substances

1.3 Details of the supplier of the safety data sheet

Company : Sigma-Aldrich Inc.
3050 Spruce Street
ST. LOUIS MO 63103
UNITED STATES

Telephone : +1 314 771-5765
Fax : +1 800 325-5052

1.4 Emergency telephone number

Emergency Phone # : +1-703-527-3887

SECTION 2: Hazards identification

2.1 Classification of the substance or mixture

GHS Classification in accordance with 29 CFR 1910 (OSHA HCS)

Flammable liquids (Category 2), H225
Acute toxicity, Inhalation (Category 4), H332
Carcinogenicity (Category 2), H351
Specific target organ toxicity - repeated exposure (Category 2), H373
Aspiration hazard (Category 1), H304
Short-term (acute) aquatic hazard (Category 2), H401

For the full text of the H-Statements mentioned in this Section, see Section 16.

2.2 GHS Label elements, including precautionary statements

Pictogram

Signal word Danger

Hazard statement(s)

H225	Highly flammable liquid and vapour.
H304	May be fatal if swallowed and enters airways.
H332	Harmful if inhaled.
H351	Suspected of causing cancer.
H373	May cause damage to organs through prolonged or repeated exposure.
H401	Toxic to aquatic life.

Precautionary statement(s)

P201	Obtain special instructions before use.
P202	Do not handle until all safety precautions have been read and understood.
P210	Keep away from heat/sparks/open flames/hot surfaces. No smoking.
P233	Keep container tightly closed.
P240	Ground/bond container and receiving equipment.
P241	Use explosion-proof electrical/ ventilating/ lighting equipment.
P242	Use only non-sparking tools.
P243	Take precautionary measures against static discharge.
P260	Do not breathe dust/ fume/ gas/ mist/ vapours/ spray.
P271	Use only outdoors or in a well-ventilated area.
P273	Avoid release to the environment.
P280	Wear protective gloves/ protective clothing/ eye protection/ face protection.
P301 + P310	IF SWALLOWED: Immediately call a POISON CENTER/doctor.
P303 + P361 + P353	IF ON SKIN (or hair): Take off immediately all contaminated clothing. Rinse skin with water/shower.
P304 + P340 + P312	IF INHALED: Remove person to fresh air and keep comfortable for breathing. Call a POISON CENTER/doctor if you feel unwell.
P308 + P313	IF exposed or concerned: Get medical advice/ attention.
P331	Do NOT induce vomiting.
P370 + P378	In case of fire: Use dry sand, dry chemical or alcohol-resistant foam to extinguish.
P403 + P235	Store in a well-ventilated place. Keep cool.
P405	Store locked up.
P501	Dispose of contents/ container to an approved waste disposal plant.

2.3 Hazards not otherwise classified (HNOC) or not covered by GHS - none

SECTION 3: Composition/information on ingredients

3.1 Substances

Formula	:	C_8H_{10}
Molecular weight	:	106.17 g/mol
CAS-No.	:	100-41-4
EC-No.	:	202-849-4
Index-No.	:	601-023-00-4

Component	Classification	Concentration
Ethylbenzene		
	Flam. Liq. 2; Acute Tox. 4;	<= 100 %

	Carc. 2; STOT RE 2; Asp. Tox. 1; Aquatic Acute 2; H225, H332, H351, H373, H304, H401	

For the full text of the H-Statements mentioned in this Section, see Section 16.

SECTION 4: First aid measures

4.1 Description of first aid measures

General advice
Consult a physician. Show this safety data sheet to the doctor in attendance.Move out of dangerous area.

If inhaled
If breathed in, move person into fresh air. If not breathing, give artificial respiration. Consult a physician.

In case of skin contact
Wash off with soap and plenty of water. Consult a physician.

In case of eye contact
Flush eyes with water as a precaution.

If swallowed
Do NOT induce vomiting. Never give anything by mouth to an unconscious person. Rinse mouth with water. Consult a physician.

4.2 Most important symptoms and effects, both acute and delayed
The most important known symptoms and effects are described in the labelling (see section 2.2) and/or in section 11

4.3 Indication of any immediate medical attention and special treatment needed
No data available

SECTION 5: Firefighting measures

5.1 Extinguishing media

Suitable extinguishing media
Use water spray, alcohol-resistant foam, dry chemical or carbon dioxide.

5.2 Special hazards arising from the substance or mixture
Carbon oxides

5.3 Advice for firefighters
Wear self-contained breathing apparatus for firefighting if necessary.

5.4 Further information
Use water spray to cool unopened containers.

SECTION 6: Accidental release measures

6.1 Personal precautions, protective equipment and emergency procedures

Use personal protective equipment. Avoid breathing vapours, mist or gas. Ensure adequate ventilation. Remove all sources of ignition. Evacuate personnel to safe areas. Beware of vapours accumulating to form explosive concentrations. Vapours can accumulate in low areas.
For personal protection see section 8.

6.2 Environmental precautions

Prevent further leakage or spillage if safe to do so. Do not let product enter drains. Discharge into the environment must be avoided.

6.3 Methods and materials for containment and cleaning up

Contain spillage, soak up with non-combustible absorbent material, (e.g. sand, earth, diatomaceous earth, vermiculite) and transfer to a container for disposal according to local / national regulations (see section 13).

6.4 Reference to other sections

For disposal see section 13.

SECTION 7: Handling and storage

7.1 Precautions for safe handling

Avoid contact with skin and eyes. Avoid inhalation of vapour or mist.
Use explosion-proof equipment.Keep away from sources of ignition - No smoking.Take measures to prevent the build up of electrostatic charge.
For precautions see section 2.2.

7.2 Conditions for safe storage, including any incompatibilities

Keep container tightly closed in a dry and well-ventilated place. Containers which are opened must be carefully resealed and kept upright to prevent leakage.

hygroscopic
Storage class (TRGS 510): 3: Flammable liquids

7.3 Specific end use(s)

Apart from the uses mentioned in section 1.2 no other specific uses are stipulated

SECTION 8: Exposure controls/personal protection

8.1 Control parameters

Components with workplace control parameters

Component	CAS-No.	Value	Control parameters	Basis
Ethylbenzene	100-41-4	TWA	100 ppm 435 mg/m3	USA. Occupational Exposure Limits (OSHA) - Table Z-1 Limits for Air Contaminants
	Remarks	The value in mg/m3 is approximate.		

		PEL	5 ppm 22 mg/m3	California permissible exposure limits for chemical contaminants (Title 8, Article 107)
		STEL	30 ppm 130 mg/m3	California permissible exposure limits for chemical contaminants (Title 8, Article 107)

Biological occupational exposure limits

Component	CAS-No.	Parameters	Value	Biological specimen	Basis
Ethylbenzene	100-41-4	Sum of mandelic acid and phenyl glyoxylic acid	0.15g/g creatinine	Urine	ACGIH - Biological Exposure Indices (BEI)
	Remarks	End of shift (As soon as possible after exposure ceases)			

8.2 Exposure controls

Appropriate engineering controls
Handle in accordance with good industrial hygiene and safety practice. Wash hands before breaks and at the end of workday.

Personal protective equipment

Eye/face protection
Face shield and safety glasses Use equipment for eye protection tested and approved under appropriate government standards such as NIOSH (US) or EN 166(EU).

Skin protection
Handle with gloves. Gloves must be inspected prior to use. Use proper glove removal technique (without touching glove's outer surface) to avoid skin contact with this product. Dispose of contaminated gloves after use in accordance with applicable laws and good laboratory practices. Wash and dry hands.

Full contact
Material: Fluorinated rubber
Minimum layer thickness: 0.7 mm
Break through time: 480 min
Material tested:Vitoject® (KCL 890 / Aldrich Z677698, Size M)

Splash contact
Material: Fluorinated rubber
Minimum layer thickness: 0.7 mm
Break through time: 480 min
Material tested:Vitoject® (KCL 890 / Aldrich Z677698, Size M)

data source: KCL GmbH, D-36124 Eichenzell, phone +49 (0)6659 87300, e-mail sales@kcl.de, test method: EN374
If used in solution, or mixed with other substances, and under conditions which differ from EN 374, contact the supplier of the CE approved gloves. This recommendation is advisory only and must be evaluated by an industrial hygienist and safety officer familiar with the specific situation of anticipated use by our

customers. It should not be construed as offering an approval for any specific use scenario.

Body Protection
Complete suit protecting against chemicals, Flame retardant antistatic protective clothing., The type of protective equipment must be selected according to the concentration and amount of the dangerous substance at the specific workplace.

Respiratory protection
Where risk assessment shows air-purifying respirators are appropriate use a full-face respirator with multi-purpose combination (US) or type ABEK (EN 14387) respirator cartridges as a backup to engineering controls. If the respirator is the sole means of protection, use a full-face supplied air respirator. Use respirators and components tested and approved under appropriate government standards such as NIOSH (US) or CEN (EU).

Control of environmental exposure
Prevent further leakage or spillage if safe to do so. Do not let product enter drains. Discharge into the environment must be avoided.

SECTION 9: Physical and chemical properties

9.1 Information on basic physical and chemical properties

a)	Appearance	Form: liquid Colour: colourless
b)	Odour	No data available
c)	Odour Threshold	No data available
d)	pH	No data available
e)	Melting point/freezing point	Melting point/range: -95 °C (-139 °F) - lit.
f)	Initial boiling point and boiling range	136 °C 277 °F - lit.
g)	Flash point	15.0 °C (59.0 °F) - closed cup
h)	Evaporation rate	No data available
i)	Flammability (solid, gas)	No data available
j)	Upper/lower flammability or explosive limits	Upper explosion limit: 6.7 %(V) Lower explosion limit: 1 %(V)
k)	Vapour pressure	13.3 hPa at 20.0 °C (68.0 °F)
l)	Vapour density	No data available
m)	Relative density	0.867 g/cm3 at 25 °C (77 °F)
n)	Water solubility	0.2 g/l at 25 °C (77 °F) - slightly soluble
o)	Partition coefficient: n-octanol/water	log Pow: 3.6 at 20 °C (68 °F)
p)	Auto-ignition temperature	432.0 °C (809.6 °F)
q)	Decomposition	No data available

	temperature	
r)	Viscosity	0.773 mm2/s at 20 °C (68 °F) -
s)	Explosive properties	No data available
t)	Oxidizing properties	No data available

9.2 Other safety information

Surface tension	71.2 mN/m at 23 °C (73 °F)

SECTION 10: Stability and reactivity

10.1 Reactivity
No data available

10.2 Chemical stability
Stable under recommended storage conditions.

10.3 Possibility of hazardous reactions
Vapours may form explosive mixture with air.

10.4 Conditions to avoid
Heat, flames and sparks.

10.5 Incompatible materials
Strong oxidizing agents

10.6 Hazardous decomposition products
Hazardous decomposition products formed under fire conditions. - Carbon oxides
Other decomposition products - No data available
In the event of fire: see section 5

SECTION 11: Toxicological information

11.1 Information on toxicological effects

Acute toxicity
LD50 Oral - Rat - male and female - 3,500 mg/kg
Inhalation: No data available
LD50 Dermal - Rabbit - 15,433 mg/kg
No data available

Skin corrosion/irritation
Skin - Rabbit
Result: Moderate skin irritation - 24 h

Serious eye damage/eye irritation
Eyes - Rabbit
Result: Mild eye irritation

Respiratory or skin sensitisation
No data available

Germ cell mutagenicity
Hamster

ovary
Result: negative

Mouse - male and female
Result: negative

Carcinogenicity

IARC: 2B - Group 2B: Possibly carcinogenic to humans (Ethylbenzene)

NTP: No component of this product present at levels greater than or equal to 0.1% is identified as a known or anticipated carcinogen by NTP.

OSHA: No component of this product present at levels greater than or equal to 0.1% is on OSHA's list of regulated carcinogens.

Reproductive toxicity
No data available

Specific target organ toxicity - single exposure
No data available

Specific target organ toxicity - repeated exposure
No data available

Aspiration hazard
May be fatal if swallowed and enters airways.

Additional Information

Repeated dose toxicity - Rat - male and female - No observed adverse effect level - 75 mg/kg
RTECS: DA0700000

Central nervous system depression, Nausea, Headache, Vomiting, Ataxia., Tremors

Stomach - Irregularities - Based on Human Evidence
Stomach - Irregularities - Based on Human Evidence

SECTION 12: Ecological information

12.1 Toxicity

Toxicity to fish	LC50 - Oncorhynchus mykiss (rainbow trout) - 4.2 mg/l - 96 h
Toxicity to daphnia and other aquatic invertebrates	static test EC50 - Daphnia magna (Water flea) - 1.8 - 2.4 mg/l - 48 h
Toxicity to algae	static test EC50 - Skeletonema costatum (marine diatom) - 4.9 mg/l - 72 h

12.2 Persistence and degradability

Biodegradability: aerobic - Exposure time 28 d
Result: 70 - 80 % - Readily biodegradable.

12.3 Bioaccumulative potential

Due to the distribution coefficient n-octanol/water, accumulation in organisms is not expected.

12.4 Mobility in soil

No data available

12.5 Results of PBT and vPvB assessment

PBT/vPvB assessment not available as chemical safety assessment not required/not conducted

12.6 Other adverse effects

An environmental hazard cannot be excluded in the event of unprofessional handling or disposal.
Harmful to aquatic life with long lasting effects.

SECTION 13: Disposal considerations

13.1 Waste treatment methods

Product
Burn in a chemical incinerator equipped with an afterburner and scrubber but exert extra care in igniting as this material is highly flammable. Offer surplus and non-recyclable solutions to a licensed disposal company. Contact a licensed professional waste disposal service to dispose of this material.

Contaminated packaging
Dispose of as unused product.

SECTION 14: Transport information

DOT (US)
UN number: 1175 Class: 3 Packing group: II
Proper shipping name: Ethylbenzene
Reportable Quantity (RQ): 1000 lbs
Poison Inhalation Hazard: No

IMDG
UN number: 1175 Class: 3 Packing group: II EMS-No: F-E, S-D
Proper shipping name: ETHYLBENZENE

IATA
UN number: 1175 Class: 3 Packing group: II
Proper shipping name: Ethylbenzene

SECTION 15: Regulatory information

SARA 302 Components
No chemicals in this material are subject to the reporting requirements of SARA Title III, Section 302.

SARA 313 Components
The following components are subject to reporting levels established by SARA Title III, Section 313:

	CAS-No.	Revision Date
Ethylbenzene	100-41-4	2007-07-01

SARA 311/312 Hazards
Fire Hazard, Chronic Health Hazard

Massachusetts Right To Know Components
No components are subject to the Massachusetts Right to Know Act.

Pennsylvania Right To Know Components

	CAS-No.	Revision Date
Ethylbenzene	100-41-4	2007-07-01

SECTION 16: Other information

Further information

The above information is believed to be correct but does not purport to be all inclusive and shall be used only as a guide. The information in this document is based on the present state of our knowledge and is applicable to the product with regard to appropriate safety precautions. It does not represent any guarantee of the properties of the product. Sigma-Aldrich Corporation and its Affiliates shall not be held liable for any damage resulting from handling or from contact with the above product. See www.sigma-aldrich.com and/or the reverse side of invoice or packing slip for additional terms and conditions of sale.

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Version: 6.1 Revision Date: 01/15/2020 Print Date: 05/29/2020

SAFETY DATA SHEET

Version 6.1
Revision Date 01/15/2020
Print Date 05/29/2020

SECTION 1: Identification of the substance/mixture and of the company/undertaking

1.1 Product identifiers

Product name : Lead

Product Number : 391352
Brand : Aldrich
CAS-No. : 7439-92-1

1.2 Relevant identified uses of the substance or mixture and uses advised against

Identified uses : Laboratory chemicals, Synthesis of substances

1.3 Details of the supplier of the safety data sheet

Company : Sigma-Aldrich Inc.
3050 Spruce Street
ST. LOUIS MO 63103
UNITED STATES

Telephone : +1 314 771-5765
Fax : +1 800 325-5052

1.4 Emergency telephone number

Emergency Phone # : +1-703-527-3887

SECTION 2: Hazards identification

2.1 Classification of the substance or mixture

GHS Classification in accordance with 29 CFR 1910 (OSHA HCS)

Acute toxicity, Oral (Category 4), H302
Carcinogenicity (Category 2), H351
Reproductive toxicity (Category 2), H361
Specific target organ toxicity - repeated exposure (Category 2), H373
Short-term (acute) aquatic hazard (Category 1), H400
Long-term (chronic) aquatic hazard (Category 1), H410

For the full text of the H-Statements mentioned in this Section, see Section 16.

2.2 GHS Label elements, including precautionary statements

Pictogram

Signal word Warning

Hazard statement(s)

H302	Harmful if swallowed.
H351	Suspected of causing cancer.
H361	Suspected of damaging fertility or the unborn child.
H373	May cause damage to organs through prolonged or repeated exposure.
H410	Very toxic to aquatic life with long lasting effects.

Precautionary statement(s)

P201	Obtain special instructions before use.
P202	Do not handle until all safety precautions have been read and understood.
P260	Do not breathe dust/ fume/ gas/ mist/ vapours/ spray.
P264	Wash skin thoroughly after handling.
P270	Do not eat, drink or smoke when using this product.
P273	Avoid release to the environment.
P280	Wear protective gloves/ protective clothing/ eye protection/ face protection.
P301 + P312 + P330	IF SWALLOWED: Call a POISON CENTER/doctor if you feel unwell. Rinse mouth.
P308 + P313	IF exposed or concerned: Get medical advice/ attention.
P391	Collect spillage.
P405	Store locked up.
P501	Dispose of contents/ container to an approved waste disposal plant.

2.3 Hazards not otherwise classified (HNOC) or not covered by GHS - none

SECTION 3: Composition/information on ingredients

3.1 Substances

Formula	:	Pb
Molecular weight	:	207.20 g/mol
CAS-No.	:	7439-92-1
EC-No.	:	231-100-4

Component	Classification	Concentration
Lead		
	Acute Tox. 4; Carc. 2; STOT RE 1; Aquatic Acute 1; Aquatic Chronic 1; H302, H351, H372, H400, H410 M-Factor - Aquatic Acute: 10	<= 100 %

For the full text of the H-Statements mentioned in this Section, see Section 16.

SECTION 4: First aid measures

4.1 Description of first aid measures

General advice
Consult a physician. Show this safety data sheet to the doctor in attendance.Move out of dangerous area.

If inhaled
If breathed in, move person into fresh air. If not breathing, give artificial respiration. Consult a physician.

In case of skin contact
Wash off with soap and plenty of water. Consult a physician.

In case of eye contact
Flush eyes with water as a precaution.

If swallowed
Never give anything by mouth to an unconscious person. Rinse mouth with water. Consult a physician.

4.2 Most important symptoms and effects, both acute and delayed
The most important known symptoms and effects are described in the labelling (see section 2.2) and/or in section 11

4.3 Indication of any immediate medical attention and special treatment needed
No data available

SECTION 5: Firefighting measures

5.1 Extinguishing media

Suitable extinguishing media
Use water spray, alcohol-resistant foam, dry chemical or carbon dioxide.

5.2 Special hazards arising from the substance or mixture
Lead oxides

5.3 Advice for firefighters
Wear self-contained breathing apparatus for firefighting if necessary.

5.4 Further information
No data available

SECTION 6: Accidental release measures

6.1 Personal precautions, protective equipment and emergency procedures
Use personal protective equipment. Avoid dust formation. Avoid breathing vapours, mist or gas. Ensure adequate ventilation. Evacuate personnel to safe areas. Avoid breathing dust.
For personal protection see section 8.

6.2 Environmental precautions
Prevent further leakage or spillage if safe to do so. Do not let product enter drains. Discharge into the environment must be avoided.

6.3 Methods and materials for containment and cleaning up

Pick up and arrange disposal without creating dust. Sweep up and shovel. Keep in suitable, closed containers for disposal.

6.4 Reference to other sections

For disposal see section 13.

SECTION 7: Handling and storage

7.1 Precautions for safe handling

Avoid contact with skin and eyes. Avoid formation of dust and aerosols.Further processing of solid materials may result in the formation of combustible dusts. The potential for combustible dust formation should be taken into consideration before additional processing occurs.
Provide appropriate exhaust ventilation at places where dust is formed.
For precautions see section 2.2.

7.2 Conditions for safe storage, including any incompatibilities

Keep container tightly closed in a dry and well-ventilated place.

Keep in a dry place.
Storage class (TRGS 510): 6.1D: Non-combustible, acute toxic Cat.3 / toxic hazardous materials or hazardous materials causing chronic effects

7.3 Specific end use(s)

Apart from the uses mentioned in section 1.2 no other specific uses are stipulated

SECTION 8: Exposure controls/personal protection

8.1 Control parameters

Components with workplace control parameters

Component	CAS-No.	Value	Control parameters	Basis
	Remarks	See 1910.1025		
Lead	7439-92-1	TWA	0.05 mg/m3	USA. ACGIH Threshold Limit Values (TLV)
		Confirmed animal carcinogen with unknown relevance to humans		
		TWA	0.05 mg/m3	USA. ACGIH Threshold Limit Values (TLV)
		Central Nervous System impairment Hematologic effects Peripheral Nervous System impairment Substances for which there is a Biological Exposure Index or Indices (see BEI® section) Confirmed animal carcinogen with unknown relevance to humans		
		TWA	0.05 mg/m3	USA. NIOSH Recommended Exposure Limits
		See Appendix C		

Biological occupational exposure limits

Component	CAS-No.	Parameters	Value	Biological specimen	Basis
Lead	7439-92-1	Lead	200 µg/l	In blood	ACGIH - Biological Exposure Indices (BEI)
	Remarks	Not critical			

8.2 Exposure controls

Appropriate engineering controls
Handle in accordance with good industrial hygiene and safety practice. Wash hands before breaks and at the end of workday.

Personal protective equipment

Eye/face protection
Safety glasses with side-shields conforming to EN166 Use equipment for eye protection tested and approved under appropriate government standards such as NIOSH (US) or EN 166(EU).

Skin protection
Handle with gloves. Gloves must be inspected prior to use. Use proper glove removal technique (without touching glove's outer surface) to avoid skin contact with this product. Dispose of contaminated gloves after use in accordance with applicable laws and good laboratory practices. Wash and dry hands.

Full contact
Material: Nitrile rubber
Minimum layer thickness: 0.11 mm
Break through time: 480 min
Material tested:Dermatril® (KCL 740 / Aldrich Z677272, Size M)

Splash contact
Material: Nitrile rubber
Minimum layer thickness: 0.11 mm
Break through time: 480 min
Material tested:Dermatril® (KCL 740 / Aldrich Z677272, Size M)

data source: KCL GmbH, D-36124 Eichenzell, phone +49 (0)6659 87300, e-mail sales@kcl.de, test method: EN374
If used in solution, or mixed with other substances, and under conditions which differ from EN 374, contact the supplier of the CE approved gloves. This recommendation is advisory only and must be evaluated by an industrial hygienist and safety officer familiar with the specific situation of anticipated use by our customers. It should not be construed as offering an approval for any specific use scenario.

Body Protection
Complete suit protecting against chemicals, The type of protective equipment must be selected according to the concentration and amount of the dangerous substance at the specific workplace.

Respiratory protection
Where risk assessment shows air-purifying respirators are appropriate use a full-face particle respirator type N100 (US) or type P3 (EN 143) respirator cartridges as a backup to engineering controls. If the respirator is the sole means of protection, use a full-face supplied air respirator. Use respirators and components tested and

approved under appropriate government standards such as NIOSH (US) or CEN (EU).

Control of environmental exposure
Prevent further leakage or spillage if safe to do so. Do not let product enter drains. Discharge into the environment must be avoided.

SECTION 9: Physical and chemical properties

9.1 Information on basic physical and chemical properties

a)	Appearance	Form: powder
b)	Odour	No data available
c)	Odour Threshold	No data available
d)	pH	No data available
e)	Melting point/freezing point	Melting point/range: 327.4 °C (621.3 °F) - lit.
f)	Initial boiling point and boiling range	1,740 °C 3,164 °F - lit.
g)	Flash point	()Not applicable
h)	Evaporation rate	No data available
i)	Flammability (solid, gas)	No data available
j)	Upper/lower flammability or explosive limits	No data available
k)	Vapour pressure	No data available
l)	Vapour density	No data available
m)	Relative density	No data available
n)	Water solubility	No data available
o)	Partition coefficient: n-octanol/water	No data available
p)	Auto-ignition temperature	No data available
q)	Decomposition temperature	No data available
r)	Viscosity	No data available
s)	Explosive properties	No data available
t)	Oxidizing properties	No data available

9.2 Other safety information

No data available

SECTION 10: Stability and reactivity

10.1 Reactivity
No data available

10.2 Chemical stability
Stable under recommended storage conditions.

10.3 Possibility of hazardous reactions
No data available

10.4 Conditions to avoid
No data available

10.5 Incompatible materials
Strong acids

10.6 Hazardous decomposition products
Hazardous decomposition products formed under fire conditions. - Lead oxides
Other decomposition products - No data available
In the event of fire: see section 5

SECTION 11: Toxicological information

11.1 Information on toxicological effects

Acute toxicity
No data available
Inhalation: No data available
Dermal: No data available
No data available

Skin corrosion/irritation
No data available

Serious eye damage/eye irritation
No data available

Respiratory or skin sensitisation
No data available

Germ cell mutagenicity

Rat
Cytogenetic analysis

Carcinogenicity
Limited evidence of carcinogenicity in animal studies

IARC:	2B - Group 2B: Possibly carcinogenic to humans (Lead)
NTP:	RAHC - Reasonably anticipated to be a human carcinogenThe reference note has been added by TD based on the background information of the NTP. (Lead)
OSHA:	OSHA specifically regulated carcinogen (Lead)

Reproductive toxicity
May damage fertility. May damage the unborn child.

Specific target organ toxicity - single exposure
No data available

Specific target organ toxicity - repeated exposure
Causes damage to organs through prolonged or repeated exposure.

Aspiration hazard
No data available

Additional Information
RTECS: OF7525000

anemia
To the best of our knowledge, the chemical, physical, and toxicological properties have not been thoroughly investigated.

Stomach - Irregularities - Based on Human Evidence
Stomach - Irregularities - Based on Human Evidence

SECTION 12: Ecological information

12.1 Toxicity

Toxicity to fish	mortality LOEC - Oncorhynchus mykiss (rainbow trout) - 1.19 mg/l - 96.0 h
	LC50 - Micropterus dolomieui - 2.2 mg/l - 96.0 h
	mortality NOEC - Salvelinus fontinalis - 1.7 mg/l - 10.0 d
Toxicity to daphnia and other aquatic invertebrates	mortality LOEC - Daphnia (water flea) - 0.17 mg/l - 24 h
	mortality NOEC - Daphnia (water flea) - 0.099 mg/l - 24 h
Toxicity to algae	mortality EC50 - Skeletonema costatum - 7.94 mg/l - 10 d

12.2 Persistence and degradability
No data available

12.3 Bioaccumulative potential
Bioaccumulation: Oncorhynchus kisutch - 2 Weeks - 150 µg/l(Lead)

Bioconcentration factor (BCF): 12

12.4 Mobility in soil
No data available

12.5 Results of PBT and vPvB assessment
PBT/vPvB assessment not available as chemical safety assessment not required/not conducted

12.6 Other adverse effects
An environmental hazard cannot be excluded in the event of unprofessional handling or disposal.
Very toxic to aquatic life with long lasting effects.

SECTION 13: Disposal considerations

13.1 Waste treatment methods

Product
Offer surplus and non-recyclable solutions to a licensed disposal company. Contact a licensed professional waste disposal service to dispose of this material. Dissolve or mix the material with a combustible solvent and burn in a chemical incinerator equipped with an afterburner and scrubber.

Contaminated packaging
Dispose of as unused product.

SECTION 14: Transport information

DOT (US)
UN number: 3077 Class: 9 Packing group: III
Proper shipping name: Environmentally hazardous substance, solid, n.o.s. (Lead)
Reportable Quantity (RQ): 10 lbs
Poison Inhalation Hazard: No

IMDG
UN number: 3077 Class: 9 Packing group: III EMS-No: F-A, S-F
Proper shipping name: ENVIRONMENTALLY HAZARDOUS SUBSTANCE, SOLID, N.O.S. (Lead)
Marine pollutant : yes

IATA
UN number: 3077 Class: 9 Packing group: III
Proper shipping name: Environmentally hazardous substance, solid, n.o.s. (Lead)
Further information
EHS-Mark required (ADR 2.2.9.1.10, IMDG code 2.10.3) for single packagings and combination packagings containing inner packagings with Dangerous Goods > 5L for liquids or > 5kg for solids.

SECTION 15: Regulatory information

SARA 302 Components
No chemicals in this material are subject to the reporting requirements of SARA Title III, Section 302.

SARA 313 Components
The following components are subject to reporting levels established by SARA Title III, Section 313:

	CAS-No.	Revision Date
Lead	7439-92-1	2015-11-23

SARA 311/312 Hazards
Acute Health Hazard, Chronic Health Hazard

Massachusetts Right To Know Components

	CAS-No.	Revision Date

Lead	7439-92-1	2015-11-23

Pennsylvania Right To Know Components

Lead	CAS-No. 7439-92-1	Revision Date 2015-11-23

New Jersey Right To Know Components

Lead	CAS-No. 7439-92-1	Revision Date 2015-11-23

California Prop. 65 Components

WARNING! This product contains a chemical known to the State of California to cause cancer.Lead	CAS-No. 7439-92-1	Revision Date 2009-02-01
WARNING: This product contains a chemical known to the State of California to cause birth defects or other reproductive harm.Lead	CAS-No. 7439-92-1	Revision Date 2009-02-01

SECTION 16: Other information

Further information

The above information is believed to be correct but does not purport to be all inclusive and shall be used only as a guide. The information in this document is based on the present state of our knowledge and is applicable to the product with regard to appropriate safety precautions. It does not represent any guarantee of the properties of the product. Sigma-Aldrich Corporation and its Affiliates shall not be held liable for any damage resulting from handling or from contact with the above product. See www.sigma-aldrich.com and/or the reverse side of invoice or packing slip for additional terms and conditions of sale.

The branding on the header and/or footer of this document may temporarily not visually match the product purchased as we transition our branding. However, all of the information in the document regarding the product remains unchanged and matches the product ordered. For further information please contact mlsbranding@sial.com.

Version: 6.1 Revision Date: 01/15/2020 Print Date: 05/29/2020

SAFETY DATA SHEET

Version 6.3
Revision Date 03/07/2020
Print Date 05/29/2020

SECTION 1: Identification of the substance/mixture and of the company/undertaking

1.1 Product identifiers

Product name : Mercury

Product Number : 261017
Brand : SIGALD
Index-No. : 080-001-00-0
CAS-No. : 7439-97-6

1.2 Relevant identified uses of the substance or mixture and uses advised against

Identified uses : Laboratory chemicals, Synthesis of substances

1.3 Details of the supplier of the safety data sheet

Company : Sigma-Aldrich Inc.
3050 Spruce Street
ST. LOUIS MO 63103
UNITED STATES

Telephone : +1 314 771-5765
Fax : +1 800 325-5052

1.4 Emergency telephone number

Emergency Phone # : +1-703-527-3887

SECTION 2: Hazards identification

2.1 Classification of the substance or mixture

GHS Classification in accordance with 29 CFR 1910 (OSHA HCS)

Acute toxicity, Inhalation (Category 2), H330
Reproductive toxicity (Category 1B), H360
Specific target organ toxicity - repeated exposure (Category 1), H372
Short-term (acute) aquatic hazard (Category 1), H400
Long-term (chronic) aquatic hazard (Category 1), H410

For the full text of the H-Statements mentioned in this Section, see Section 16.

2.2 GHS Label elements, including precautionary statements

Pictogram

Signal word Danger

Hazard statement(s)

H330	Fatal if inhaled.
H360	May damage fertility or the unborn child.
H372	Causes damage to organs through prolonged or repeated exposure.
H410	Very toxic to aquatic life with long lasting effects.

Precautionary statement(s)

P201	Obtain special instructions before use.
P202	Do not handle until all safety precautions have been read and understood.
P260	Do not breathe dust/ fume/ gas/ mist/ vapours/ spray.
P264	Wash skin thoroughly after handling.
P270	Do not eat, drink or smoke when using this product.
P271	Use only outdoors or in a well-ventilated area.
P273	Avoid release to the environment.
P280	Wear protective gloves/ protective clothing/ eye protection/ face protection.
P284	Wear respiratory protection.
P304 + P340 + P310	IF INHALED: Remove person to fresh air and keep comfortable for breathing. Immediately call a POISON CENTER/doctor.
P308 + P313	IF exposed or concerned: Get medical advice/ attention.
P391	Collect spillage.
P403 + P233	Store in a well-ventilated place. Keep container tightly closed.
P405	Store locked up.
P501	Dispose of contents/ container to an approved waste disposal plant.

2.3 Hazards not otherwise classified (HNOC) or not covered by GHS - none

SECTION 3: Composition/information on ingredients

3.1 Substances

Formula	:	Hg
Molecular weight	:	200.59 g/mol
CAS-No.	:	7439-97-6
EC-No.	:	231-106-7
Index-No.	:	080-001-00-0

Component	Classification	Concentration
Mercury		
	Acute Tox. 2; Repr. 1B; STOT RE 1; Aquatic Acute 1; Aquatic Chronic 1; H330, H360, H372, H400, H410 M-Factor - Aquatic Acute: 1 - Aquatic Chronic: 100	<= 100 %

For the full text of the H-Statements mentioned in this Section, see Section 16.

SECTION 4: First aid measures

4.1 Description of first aid measures

General advice
Consult a physician. Show this safety data sheet to the doctor in attendance.Move out of dangerous area.

If inhaled
If breathed in, move person into fresh air. If not breathing, give artificial respiration. Consult a physician.

In case of skin contact
Wash off with soap and plenty of water. Take victim immediately to hospital. Consult a physician.

In case of eye contact
Flush eyes with water as a precaution.

If swallowed
Never give anything by mouth to an unconscious person. Rinse mouth with water. Consult a physician.

4.2 Most important symptoms and effects, both acute and delayed
The most important known symptoms and effects are described in the labelling (see section 2.2) and/or in section 11

4.3 Indication of any immediate medical attention and special treatment needed
No data available

SECTION 5: Firefighting measures

5.1 Extinguishing media

Suitable extinguishing media
Use water spray, alcohol-resistant foam, dry chemical or carbon dioxide.

5.2 Special hazards arising from the substance or mixture
Mercury/mercury oxides.
Not combustible.

5.3 Advice for firefighters
Wear self-contained breathing apparatus for firefighting if necessary.

5.4 Further information
No data available

SECTION 6: Accidental release measures

6.1 Personal precautions, protective equipment and emergency procedures
Wear respiratory protection. Avoid breathing vapours, mist or gas. Ensure adequate ventilation. Evacuate personnel to safe areas.
For personal protection see section 8.

6.2 Environmental precautions
Prevent further leakage or spillage if safe to do so. Do not let product enter drains. Discharge into the environment must be avoided.

6.3 Methods and materials for containment and cleaning up

Soak up with inert absorbent material and dispose of as hazardous waste. Keep in suitable, closed containers for disposal.

6.4 Reference to other sections

For disposal see section 13.

SECTION 7: Handling and storage

7.1 Precautions for safe handling

Avoid contact with skin and eyes. Avoid inhalation of vapour or mist.
For precautions see section 2.2.

7.2 Conditions for safe storage, including any incompatibilities

Keep container tightly closed in a dry and well-ventilated place. Containers which are opened must be carefully resealed and kept upright to prevent leakage.

Store under inert gas.
Storage class (TRGS 510): 6.1A: Combustible, acute toxic Cat. 1 and 2 / very toxic hazardous materials

7.3 Specific end use(s)

Apart from the uses mentioned in section 1.2 no other specific uses are stipulated

SECTION 8: Exposure controls/personal protection

8.1 Control parameters

Components with workplace control parameters

Component	CAS-No.	Value	Control parameters	Basis
Mercury	7439-97-6	C	0.1 mg/m3	USA. NIOSH Recommended Exposure Limits
	Remarks	Potential for dermal absorption		
		CEIL	1.0mg/10m3	USA. Occupational Exposure Limits (OSHA) - Table Z-2
		TWA	0.05 mg/m3	USA. OSHA - TABLE Z-1 Limits for Air Contaminants - 1910.1000
		Skin notation		
		TWA	0.025 mg/m3	USA. ACGIH Threshold Limit Values (TLV)
		Central Nervous System impairment Kidney damage Substances for which there is a Biological Exposure Index or Indices (see BEI® section) Not classifiable as a human carcinogen Danger of cutaneous absorption		
		TWA	0.05 mg/m3	USA. NIOSH Recommended Exposure Limits
		Potential for dermal absorption		

8.2 Exposure controls

Appropriate engineering controls

Avoid contact with skin, eyes and clothing. Wash hands before breaks and immediately after handling the product.

Personal protective equipment

Eye/face protection

Face shield and safety glasses Use equipment for eye protection tested and approved under appropriate government standards such as NIOSH (US) or EN 166(EU).

Skin protection

Handle with gloves. Gloves must be inspected prior to use. Use proper glove removal technique (without touching glove's outer surface) to avoid skin contact with this product. Dispose of contaminated gloves after use in accordance with applicable laws and good laboratory practices. Wash and dry hands.

Full contact
Material: Nitrile rubber
Minimum layer thickness: 0.11 mm
Break through time: 480 min
Material tested:Dermatril® (KCL 740 / Aldrich Z677272, Size M)

Splash contact
Material: Nitrile rubber
Minimum layer thickness: 0.11 mm
Break through time: 480 min
Material tested:Dermatril® (KCL 740 / Aldrich Z677272, Size M)

data source: KCL GmbH, D-36124 Eichenzell, phone +49 (0)6659 87300, e-mail sales@kcl.de, test method: EN374
If used in solution, or mixed with other substances, and under conditions which differ from EN 374, contact the supplier of the CE approved gloves. This recommendation is advisory only and must be evaluated by an industrial hygienist and safety officer familiar with the specific situation of anticipated use by our customers. It should not be construed as offering an approval for any specific use scenario.

Body Protection

Complete suit protecting against chemicals, The type of protective equipment must be selected according to the concentration and amount of the dangerous substance at the specific workplace.

Respiratory protection

Where risk assessment shows air-purifying respirators are appropriate use a full-face respirator with multi-purpose combination (US) or type ABEK (EN 14387) respirator cartridges as a backup to engineering controls. If the respirator is the sole means of protection, use a full-face supplied air respirator. Use respirators and components tested and approved under appropriate government standards such as NIOSH (US) or CEN (EU).

Control of environmental exposure

Prevent further leakage or spillage if safe to do so. Do not let product enter drains. Discharge into the environment must be avoided.

SECTION 9: Physical and chemical properties

9.1 Information on basic physical and chemical properties

a)	Appearance	Form: liquid Colour: silver, white
b)	Odour	odourless
c)	Odour Threshold	No data available
d)	pH	No data available
e)	Melting point/freezing point	Melting point/range: -38.87 °C (-37.97 °F) - lit.
f)	Initial boiling point and boiling range	356.6 °C (673.9 °F)
g)	Flash point	()Not applicable
h)	Evaporation rate	No data available
i)	Flammability (solid, gas)	No data available
j)	Upper/lower flammability or explosive limits	No data available
k)	Vapour pressure	< 0.01 hPa at 20 °C (68 °F) 1 hPa at 126 °C(259 °F)
l)	Vapour density	6.93 - (Air = 1.0)
m)	Relative density	13.55 g/cm3 at 25 °C (77 °F)
n)	Water solubility	0.00006 g/l at 25 °C (77 °F)
o)	Partition coefficient: n-octanol/water	No data available
p)	Auto-ignition temperature	No data available
q)	Decomposition temperature	No data available
r)	Viscosity	No data available
s)	Explosive properties	No data available
t)	Oxidizing properties	No data available

9.2 Other safety information

Relative vapour density	6.93 - (Air = 1.0)

SECTION 10: Stability and reactivity

10.1 Reactivity

No data available

10.2 Chemical stability
Stable under recommended storage conditions.

10.3 Possibility of hazardous reactions
No data available

10.4 Conditions to avoid
No data available

10.5 Incompatible materials
Strong oxidizing agents, Ammonia, Azides, Nitrates, Chlorates, Copper

10.6 Hazardous decomposition products
Hazardous decomposition products formed under fire conditions. - Mercury/mercury oxides.
Other decomposition products - No data available
In the event of fire: see section 5

SECTION 11: Toxicological information

11.1 Information on toxicological effects

Acute toxicity
No data available
LC50 Inhalation - Rat - male - 2 h - < 27 mg/m3
Dermal: No data available
No data available

Skin corrosion/irritation
No data available

Serious eye damage/eye irritation
No data available

Respiratory or skin sensitisation
No data available

Germ cell mutagenicity
No data available

Carcinogenicity
This product is or contains a component that is not classifiable as to its carcinogenicity based on its IARC, ACGIH, NTP, or EPA classification.

IARC: No component of this product present at levels greater than or equal to 0.1% is identified as probable, possible or confirmed human carcinogen by IARC.

NTP: No component of this product present at levels greater than or equal to 0.1% is identified as a known or anticipated carcinogen by NTP.

OSHA: No component of this product present at levels greater than or equal to 0.1% is on OSHA's list of regulated carcinogens.

Reproductive toxicity
Presumed human reproductive toxicant

Specific target organ toxicity - single exposure
No data available

Specific target organ toxicity - repeated exposure
Causes damage to organs through prolonged or repeated exposure.

Aspiration hazard
No data available

Additional Information
RTECS: OV4550000

To the best of our knowledge, the chemical, physical, and toxicological properties have not been thoroughly investigated.

Stomach - Irregularities - Based on Human Evidence
Stomach - Irregularities - Based on Human Evidence

SECTION 12: Ecological information

12.1 Toxicity

Toxicity to fish	mortality LC50 - Cyprinus carpio (Carp) - 0.160 mg/l - 96 h

12.2 Persistence and degradability
No data available

12.3 Bioaccumulative potential

Bioaccumulation	Carassius auratus (goldfish) - 1,789 d - 0.25 µg/l(Mercury)
	Bioconcentration factor (BCF): 155,986

12.4 Mobility in soil
No data available

12.5 Results of PBT and vPvB assessment
PBT/vPvB assessment not available as chemical safety assessment not required/not conducted

12.6 Other adverse effects
An environmental hazard cannot be excluded in the event of unprofessional handling or disposal.
Very toxic to aquatic life with long lasting effects.
No data available

SECTION 13: Disposal considerations

13.1 Waste treatment methods

Product
Offer surplus and non-recyclable solutions to a licensed disposal company. Contact a licensed professional waste disposal service to dispose of this material.

Contaminated packaging
Dispose of as unused product.

SECTION 14: Transport information

DOT (US)
UN number: 2809 Class: 8 (6.1) Packing group: III

Proper shipping name: A. W. Mercury
Reportable Quantity (RQ): 1 lbs
Reportable Quantity (RQ): 1 lbs
Poison Inhalation Hazard: No

IMDG
UN number: 2809 Class: 8 (6.1) Packing group: III EMS-No: F-A, S-B
Proper shipping name: MERCURY
Marine pollutant : yes

IATA
UN number: 2809 Class: 8 (6.1) Packing group: III
Proper shipping name: Mercury

SECTION 15: Regulatory information

SARA 302 Components
This material does not contain any components with a section 302 EHS TPQ.

SARA 313 Components
The following components are subject to reporting levels established by SARA Title III, Section 313:

	CAS-No.	Revision Date
Mercury	7439-97-6	2007-03-01

SARA 311/312 Hazards
Acute Health Hazard, Chronic Health Hazard

Reportable Quantity : D009 lbs

Massachusetts Right To Know Components
No components are subject to the Massachusetts Right to Know Act.

SECTION 16: Other information

Further information

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SAFETY DATA SHEET

Version 6.3
Revision Date 01/10/2020
Print Date 06/02/2020

SECTION 1: Identification of the substance/mixture and of the company/undertaking

1.1 Product identifiers

Product name : PAHs by HPLC - PT

Product Number : SPE017
Brand : Sigma-Aldrich

1.2 Relevant identified uses of the substance or mixture and uses advised against

Identified uses : Laboratory chemicals, Synthesis of substances

1.3 Details of the supplier of the safety data sheet

Company : Sigma-Aldrich Inc.
3050 Spruce Street
ST. LOUIS MO 63103
UNITED STATES

Telephone : +1 314 771-5765
Fax : +1 800 325-5052

1.4 Emergency telephone number

Emergency Phone # : +1-703-527-3887

SECTION 2: Hazards identification

2.1 Classification of the substance or mixture

GHS Classification in accordance with 29 CFR 1910 (OSHA HCS)

Short-term (acute) aquatic hazard (Category 3), H402
Long-term (chronic) aquatic hazard (Category 3), H412

For the full text of the H-Statements mentioned in this Section, see Section 16.

2.2 GHS Label elements, including precautionary statements

Pictogram: none

Signal word: none

Hazard statement(s)
H412: Harmful to aquatic life with long lasting effects.

Precautionary statement(s)
P273: Avoid release to the environment.
P501: Dispose of contents/ container to an approved waste disposal plant.

2.3 Hazards not otherwise classified (HNOC) or not covered by GHS - none

SECTION 3: Composition/information on ingredients

3.2 Mixtures

Component		Classification	Concentration
Quartz (SiO2)			
CAS-No. EC-No.	14808-60-7 238-878-4		>= 90 - <= 100 %
Anthracene			
CAS-No. EC-No.	120-12-7 204-371-1	Carc. 1A; Aquatic Acute 1; Aquatic Chronic 1; H350, H400, H410 M-Factor - Aquatic Acute: 1,000 M-Factor - Aquatic Chronic: 1,000	< 0.1 %
Benzo[ghi]perylene Included in the Candidate List of Substances of Very High Concern (SVHC) according to Regulation (EC) No. 1907/2006 (REACH)			
CAS-No. EC-No.	191-24-2 205-883-8	Aquatic Acute 1; Aquatic Chronic 1; H400, H410 M-Factor - Aquatic Acute: 1,000 - Aquatic Chronic: 1,000	< 0.1 %

For the full text of the H-Statements mentioned in this Section, see Section 16.

SECTION 4: First aid measures

4.1 Description of first aid measures

General advice
Consult a physician. Show this safety data sheet to the doctor in attendance.Move out of dangerous area.

If inhaled
If breathed in, move person into fresh air. If not breathing, give artificial respiration. Consult a physician.

In case of skin contact
Wash off with soap and plenty of water. Consult a physician.

In case of eye contact
Flush eyes with water as a precaution.

If swallowed
Never give anything by mouth to an unconscious person. Rinse mouth with water. Consult a physician.

4.2 Most important symptoms and effects, both acute and delayed

The most important known symptoms and effects are described in the labelling (see section 2.2) and/or in section 11

4.3 Indication of any immediate medical attention and special treatment needed
No data available

SECTION 5: Firefighting measures

5.1 Extinguishing media

Suitable extinguishing media
Use water spray, alcohol-resistant foam, dry chemical or carbon dioxide.

5.2 Special hazards arising from the substance or mixture
silicon oxides

5.3 Advice for firefighters
Wear self-contained breathing apparatus for firefighting if necessary.

5.4 Further information
No data available

SECTION 6: Accidental release measures

6.1 Personal precautions, protective equipment and emergency procedures
Avoid dust formation. Avoid breathing vapours, mist or gas. Ensure adequate ventilation.
For personal protection see section 8.

6.2 Environmental precautions
Prevent further leakage or spillage if safe to do so. Do not let product enter drains.
Discharge into the environment must be avoided.

6.3 Methods and materials for containment and cleaning up
Pick up and arrange disposal without creating dust. Sweep up and shovel. Keep in suitable, closed containers for disposal.

6.4 Reference to other sections
For disposal see section 13.

SECTION 7: Handling and storage

7.1 Precautions for safe handling
Further processing of solid materials may result in the formation of combustible dusts. The potential for combustible dust formation should be taken into consideration before additional processing occurs.
Provide appropriate exhaust ventilation at places where dust is formed.
For precautions see section 2.2.

7.2 Conditions for safe storage, including any incompatibilities
Keep container tightly closed in a dry and well-ventilated place.

Recommended storage temperature 2 - 8 °C
Storage class (TRGS 510): 13: Non Combustible Solids

7.3 Specific end use(s)
Apart from the uses mentioned in section 1.2 no other specific uses are stipulated

SECTION 8: Exposure controls/personal protection

8.1 Control parameters

Components with workplace control parameters

Component	CAS-No.	Value	Control parameters	Basis
Quartz (SiO2)	14808-60-7	TWA	0.05 mg/m3	USA. Occupational Exposure Limits (OSHA) - Table Z-1 Limits for Air Contaminants
	Remarks	Substance listed; for more information see OSHA document 1910.1053 See Table Z-3 for the exposure limit for any operations or sectors where the exposure limit in § 1910.1053 is stayed or is otherwise not in effect.		
		TWA	10mg/m3 / %SiO2+2	USA. Occupational Exposure Limits (OSHA) - Table Z-3 Mineral Dusts
		This standard applies to any operations or sectors for which the respirable crystalline silica standard, 1910.1053, is stayed or is otherwise not in effect. Both concentration and percent quartz for the application of this limit are to be determined from the fraction passing a size-selector with the following characteristics: Aerodynamic diameter (unit density sphere): 2; Percent passing selector: 90 Aerodynamic diameter (unit density sphere): 2,5; Percent passing selector: 75 Aerodynamic diameter (unit density sphere): 3,5; Percent passing selector: 50 Aerodynamic diameter (unit density sphere): 5,0; Percent passing selector: 25 Aerodynamic diameter (unit density sphere): 10; Percent passing selector: 0 The measurements under this note refer to the use of an AEC (now NRC) instrument. The respirable fraction of coal dust is determined with an MRE; the figure corresponding to that of 2.4 mg/m3 in the table for coal dust is 4.5 mg/m3.		
		TWA	250mppcf / %SiO2+5	USA. Occupational Exposure Limits (OSHA) - Table Z-3 Mineral Dusts
		This standard applies to any operations or sectors for which the respirable crystalline silica standard, 1910.1053, is stayed or is otherwise not in effect. Millions of particles per cubic foot of air, based on impinger samples counted by light-field techniques. The percentage of crystalline silica in the formula is the amount determined from airborne samples, except in those instances in which other methods have been shown to be applicable.		
		PEL	0.05 mg/m3	California permissible exposure limits for chemical contaminants (Title 8, Article 107)
		The concentration and percentage of the particulate used for this limit are determined from the fraction passing a size		

		selector with the following characteristics: Aerodynamic Diameter in Micrometers (unit density sphere)..... Percent Passing Selector 0 ..... 100 1 ..... 97 2 ..... 91 3 ..... 74 4 ..... 50 5 ..... 30 6 ..... 17 7 ..... 9 8 ..... 5 10 ..... 1 see also Sections 1532.3 & 5204		
		TWA	0.025 mg/m3	USA. ACGIH Threshold Limit Values (TLV)
		Lung cancer Pulmonary fibrosis Suspected human carcinogen		
		TWA	0.05 mg/m3	USA. NIOSH Recommended Exposure Limits
		Potential Occupational Carcinogen See Appendix A		
Anthracene	120-12-7	TWA	0.2 mg/m3	USA. Occupational Exposure Limits (OSHA) - Table Z-1 Limits for Air Contaminants
		1910.1002 As used in §1910.1000 (Table Z-1), coal tar pitch volatiles include the fused polycyclic hydrocarbons which volatilize from the distillation residues of coal, petroleum (excluding asphalt), wood, and other organic matter. Asphalt (CAS 8052-42-4, and CAS 64742-93-4) is not covered under the 'coal tar pitch volatiles' standard OSHA specifically regulated carcinogen		
		TWA	0.1 mg/m3	USA. NIOSH Recommended Exposure Limits
		Potential Occupational Carcinogen NIOSH considers coal tar, coal tar pitch, and creosote to be coal tar products. cyclohexane-extractable fraction See Appendix C See Appendix A		
		PEL	0.2 mg/m3	California permissible exposure limits for chemical contaminants (Title 8, Article 107)
		Coal tar pitch volatiles (benzene or cyclohexane-soluble fraction) include fused polycyclic hydrocarbons (some of which are known carcinogens) which volatilize from the distillation residues of coal, petroleum (excluding asphalt), wood, and other organic matter. Asphalt (CAS 8052-42-4, and CAS 64742-93-4) is not covered under the 'coal tar pitch volatiles' standard.		

		PEL	0.2 mg/m3	California permissible exposure limits for chemical contaminants (Title 8, Article 107)
		Coal tar pitch volatiles (benzene or cyclohexane-soluble fraction) include fused polycyclic hydrocarbons (some of which are known carcinogens) which volatilize from the distillation residues of coal, petroleum (excluding asphalt), wood, and other organic matter. Asphalt (CAS 8052-42-4, and CAS 64742-93-4) is not covered under the 'coal tar pitch volatiles' standard.		
Benzo[ghi]perylene	191-24-2	PEL	0.2 mg/m3	California permissible exposure limits for chemical contaminants (Title 8, Article 107)
		Coal tar pitch volatiles (benzene or cyclohexane-soluble fraction) include fused polycyclic hydrocarbons (some of which are known carcinogens) which volatilize from the distillation residues of coal, petroleum (excluding asphalt), wood, and other organic matter. Asphalt (CAS 8052-42-4, and CAS 64742-93-4) is not covered under the 'coal tar pitch volatiles' standard.		

Biological occupational exposure limits

Component	CAS-No.	Parameters	Value	Biological specimen	Basis
Anthracene	120-12-7	1-Hydroxypyrene	2.5 µg/l	Urine	ACGIH - Biological Exposure Indices (BEI)
	Remarks	End of shift at end of workweek			
		3-hydroxybenzo(a)pyrene		Urine	ACGIH - Biological Exposure Indices (BEI)
		End of shift at end of workweek			
Benzo[ghi]perylene	191-24-2	1-Hydroxypyrene	2.5 µg/l	Urine	ACGIH - Biological Exposure Indices (BEI)
		End of shift at end of workweek			
		3-hydroxybenzo(a)pyrene		Urine	ACGIH - Biological Exposure Indices (BEI)
		End of shift at end of workweek			

8.2 Exposure controls

Appropriate engineering controls

Handle in accordance with good industrial hygiene and safety practice. Wash hands before breaks and at the end of workday.

Personal protective equipment

Eye/face protection
Use equipment for eye protection tested and approved under appropriate government standards such as NIOSH (US) or EN 166(EU).

Skin protection
Handle with gloves. Gloves must be inspected prior to use. Use proper glove removal technique (without touching glove's outer surface) to avoid skin contact with this product. Dispose of contaminated gloves after use in accordance with applicable laws and good laboratory practices. Wash and dry hands.

Body Protection
Choose body protection in relation to its type, to the concentration and amount of dangerous substances, and to the specific work-place., The type of protective equipment must be selected according to the concentration and amount of the dangerous substance at the specific workplace.

Respiratory protection
Respiratory protection is not required. Where protection from nuisance levels of dusts are desired, use type N95 (US) or type P1 (EN 143) dust masks. Use respirators and components tested and approved under appropriate government standards such as NIOSH (US) or CEN (EU).

Control of environmental exposure
Prevent further leakage or spillage if safe to do so. Do not let product enter drains. Discharge into the environment must be avoided.

SECTION 9: Physical and chemical properties

9.1 Information on basic physical and chemical properties

a)	Appearance	Form: solid
b)	Odour	No data available
c)	Odour Threshold	No data available
d)	pH	No data available
e)	Melting point/freezing point	No data available
f)	Initial boiling point and boiling range	No data available
g)	Flash point	()No data available
h)	Evaporation rate	No data available
i)	Flammability (solid, gas)	No data available
j)	Upper/lower flammability or explosive limits	No data available
k)	Vapour pressure	No data available
l)	Vapour density	No data available
m)	Relative density	No data available
n)	Water solubility	No data available

o)	Partition coefficient: n-octanol/water	No data available
p)	Auto-ignition temperature	No data available
q)	Decomposition temperature	No data available
r)	Viscosity	No data available
s)	Explosive properties	No data available
t)	Oxidizing properties	No data available

9.2 Other safety information
No data available

SECTION 10: Stability and reactivity

10.1 Reactivity
No data available

10.2 Chemical stability
Stable under recommended storage conditions.

10.3 Possibility of hazardous reactions
No data available

10.4 Conditions to avoid
No data available

10.5 Incompatible materials
Strong oxidizing agents

10.6 Hazardous decomposition products
Hazardous decomposition products formed under fire conditions. - silicon oxides
Other decomposition products - No data available
In the event of fire: see section 5

SECTION 11: Toxicological information

11.1 Information on toxicological effects

Acute toxicity
No data available

Inhalation: No data available

Dermal: No data available

No data available

Skin corrosion/irritation
No data available

Serious eye damage/eye irritation
No data available

Respiratory or skin sensitisation
No data available

Germ cell mutagenicity
No data available

Carcinogenicity

NTP: No component of this product present at levels greater than or equal to 0.1% is identified as a known or anticipated carcinogen by NTP.

OSHA: No component of this product present at levels greater than or equal to 0.1% is on OSHA's list of regulated carcinogens.

Reproductive toxicity
No data available

Specific target organ toxicity - single exposure
No data available

Specific target organ toxicity - repeated exposure
No data available

Aspiration hazard
No data available

Additional Information
RTECS: Not available

To the best of our knowledge, the chemical, physical, and toxicological properties have not been thoroughly investigated.

Liver - Irregularities - Based on Human Evidence

SECTION 12: Ecological information

12.1 Toxicity
No data available

12.2 Persistence and degradability
No data available

12.3 Bioaccumulative potential
No data available

12.4 Mobility in soil
No data available

12.5 Results of PBT and vPvB assessment
PBT/vPvB assessment not available as chemical safety assessment not required/not conducted

12.6 Other adverse effects
Harmful to aquatic life with long lasting effects.
An environmental hazard cannot be excluded in the event of unprofessional handling or disposal.

SECTION 13: Disposal considerations

13.1 Waste treatment methods

Product
Contact a licensed professional waste disposal service to dispose of this material. Dissolve or mix the material with a combustible solvent and burn in a chemical incinerator equipped with an afterburner and scrubber. Offer surplus and non-recyclable solutions to a licensed disposal company.

Contaminated packaging
Dispose of as unused product.

SECTION 14: Transport information

DOT (US)
Not dangerous goods

IMDG
Not dangerous goods

IATA
Not dangerous goods

SECTION 15: Regulatory information

SARA 302 Components
This material does not contain any components with a section 302 EHS TPQ.

SARA 313 Components
This material does not contain any chemical components with known CAS numbers that exceed the threshold (De Minimis) reporting levels established by SARA Title III, Section 313.

SARA 311/312 Hazards
Chronic Health Hazard

Massachusetts Right To Know Components
No components are subject to the Massachusetts Right to Know Act.

Pennsylvania Right To Know Components

Quartz (SiO2)	CAS-No. 14808-60-7	Revision Date 1989-08-11
Quartz (SiO2)	CAS-No. 14808-60-7	Revision Date 1989-08-11

New Jersey Right To Know Components

Quartz (SiO2)	CAS-No. 14808-60-7	Revision Date 1989-08-11

California Prop. 65 Components
This product does not contain any chemicals known to State of California to cause cancer, birth defects, or any other reproductive harm.

SECTION 16: Other information

Further information

The above information is believed to be correct but does not purport to be all inclusive and shall be used only as a guide. The information in this document is based on the present state of our knowledge and is applicable to the product with regard to appropriate safety precautions. It does not represent any guarantee of the properties of the product. Sigma-Aldrich Corporation and its Affiliates shall not be held liable for any damage resulting from handling or from contact with the above product. See www.sigma-aldrich.com and/or the reverse side of invoice or packing slip for additional terms and conditions of sale.

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Version: 6.3 Revision Date: 01/10/2020 Print Date: 06/02/2020

SAFETY DATA SHEET

Version 8.1
Revision Date 03/28/2020
Print Date 06/02/2020

SECTION 1: Identification of the substance/mixture and of the company/undertaking

1.1 Product identifiers

Product name : PCBs in Soil

Product Number : SQC010
Brand : Sigma-Aldrich

1.2 Relevant identified uses of the substance or mixture and uses advised against

Identified uses : Laboratory chemicals, Synthesis of substances

1.3 Details of the supplier of the safety data sheet

Company : Sigma-Aldrich Inc.
3050 Spruce Street
ST. LOUIS MO 63103
UNITED STATES

Telephone : +1 314 771-5765
Fax : +1 800 325-5052

1.4 Emergency telephone number

Emergency Phone # : +1-703-527-3887

SECTION 2: Hazards identification

2.1 Classification of the substance or mixture

GHS Classification in accordance with 29 CFR 1910 (OSHA HCS)

Short-term (acute) aquatic hazard (Category 2), H401
Long-term (chronic) aquatic hazard (Category 2), H411

For the full text of the H-Statements mentioned in this Section, see Section 16.

2.2 GHS Label elements, including precautionary statements

Pictogram

Signal word none

Hazard statement(s)
H411 Toxic to aquatic life with long lasting effects.

Precautionary statement(s)
P273 Avoid release to the environment.
P391 Collect spillage.
P501 Dispose of contents/ container to an approved waste disposal

plant.

2.3 Hazards not otherwise classified (HNOC) or not covered by GHS - none

SECTION 3: Composition/information on ingredients

3.2 Mixtures

No components need to be disclosed according to the applicable regulations.

For the full text of the H-Statements mentioned in this Section, see Section 16.

SECTION 4: First aid measures

4.1 Description of first aid measures

General advice
Consult a physician. Show this safety data sheet to the doctor in attendance.

If inhaled
If breathed in, move person into fresh air. If not breathing, give artificial respiration. Consult a physician.

In case of skin contact
Wash off with soap and plenty of water. Consult a physician.

In case of eye contact
Flush eyes with water as a precaution.

If swallowed
Never give anything by mouth to an unconscious person. Rinse mouth with water. Consult a physician.

4.2 Most important symptoms and effects, both acute and delayed

The most important known symptoms and effects are described in the labelling (see section 2.2) and/or in section 11

4.3 Indication of any immediate medical attention and special treatment needed

No data available

SECTION 5: Firefighting measures

5.1 Extinguishing media

Suitable extinguishing media
Use water spray, alcohol-resistant foam, dry chemical or carbon dioxide.

5.2 Special hazards arising from the substance or mixture

5.3 Advice for firefighters

Wear self-contained breathing apparatus for firefighting if necessary.

5.4 Further information

No data available

SECTION 6: Accidental release measures

6.1 Personal precautions, protective equipment and emergency procedures

Avoid dust formation. Avoid breathing vapours, mist or gas. Ensure adequate ventilation.
For personal protection see section 8.

6.2 Environmental precautions

Prevent further leakage or spillage if safe to do so. Do not let product enter drains.
Discharge into the environment must be avoided.

6.3 Methods and materials for containment and cleaning up

Pick up and arrange disposal without creating dust. Sweep up and shovel. Keep in suitable, closed containers for disposal.

6.4 Reference to other sections

For disposal see section 13.

SECTION 7: Handling and storage

7.1 Precautions for safe handling

Further processing of solid materials may result in the formation of combustible dusts. The potential for combustible dust formation should be taken into consideration before additional processing occurs.
Provide appropriate exhaust ventilation at places where dust is formed.
For precautions see section 2.2.

7.2 Conditions for safe storage, including any incompatibilities

Keep container tightly closed in a dry and well-ventilated place.

Store at Room Temperature.
Storage class (TRGS 510): 13: Non Combustible Solids

7.3 Specific end use(s)

Apart from the uses mentioned in section 1.2 no other specific uses are stipulated

SECTION 8: Exposure controls/personal protection

8.1 Control parameters

Components with workplace control parameters
Contains no substances with occupational exposure limit values.

8.2 Exposure controls

Appropriate engineering controls
Handle in accordance with good industrial hygiene and safety practice. Wash hands before breaks and at the end of workday.

Personal protective equipment

Eye/face protection
Use equipment for eye protection tested and approved under appropriate government standards such as NIOSH (US) or EN 166(EU).

Skin protection
Handle with gloves. Gloves must be inspected prior to use. Use proper glove removal technique (without touching glove's outer surface) to avoid skin contact

with this product. Dispose of contaminated gloves after use in accordance with applicable laws and good laboratory practices. Wash and dry hands.

Body Protection
Choose body protection in relation to its type, to the concentration and amount of dangerous substances, and to the specific work-place., The type of protective equipment must be selected according to the concentration and amount of the dangerous substance at the specific workplace.

Respiratory protection
Respiratory protection is not required. Where protection from nuisance levels of dusts are desired, use type N95 (US) or type P1 (EN 143) dust masks. Use respirators and components tested and approved under appropriate government standards such as NIOSH (US) or CEN (EU).

Control of environmental exposure
Prevent further leakage or spillage if safe to do so. Do not let product enter drains. Discharge into the environment must be avoided.

SECTION 9: Physical and chemical properties

9.1 Information on basic physical and chemical properties

a)	Appearance	Form: solid
b)	Odour	No data available
c)	Odour Threshold	No data available
d)	pH	No data available
e)	Melting point/freezing point	No data available
f)	Initial boiling point and boiling range	No data available
g)	Flash point	()No data available
h)	Evaporation rate	No data available
i)	Flammability (solid, gas)	No data available
j)	Upper/lower flammability or explosive limits	No data available
k)	Vapour pressure	No data available
l)	Vapour density	No data available
m)	Relative density	No data available
n)	Water solubility	No data available
o)	Partition coefficient: n-octanol/water	No data available
p)	Auto-ignition temperature	No data available
q)	Decomposition temperature	No data available
r)	Viscosity	No data available

s) Explosive properties No data available

t) Oxidizing properties No data available

9.2 Other safety information
No data available

SECTION 10: Stability and reactivity

10.1 Reactivity
No data available

10.2 Chemical stability
Stable under recommended storage conditions.

10.3 Possibility of hazardous reactions
No data available

10.4 Conditions to avoid
No data available

10.5 Incompatible materials
Strong oxidizing agents

10.6 Hazardous decomposition products
Other decomposition products - No data available
In the event of fire: see section 5

SECTION 11: Toxicological information

11.1 Information on toxicological effects

Acute toxicity
Inhalation: No data available

Dermal: No data available

No data available

Skin corrosion/irritation
No data available

Serious eye damage/eye irritation
No data available

Respiratory or skin sensitisation
No data available

Germ cell mutagenicity
No data available

Carcinogenicity

IARC: No component of this product present at levels greater than or equal to 0.1% is identified as probable, possible or confirmed human carcinogen by IARC.

ACGIH: No component of this product present at levels greater than or equal to 0.1% is identified as a carcinogen or potential carcinogen by ACGIH.

NTP: No component of this product present at levels greater than or equal to 0.1% is

identified as a known or anticipated carcinogen by NTP.

OSHA: No component of this product present at levels greater than or equal to 0.1% is on OSHA's list of regulated carcinogens.

Reproductive toxicity
No data available

Specific target organ toxicity - single exposure
No data available

Specific target organ toxicity - repeated exposure
No data available

Aspiration hazard
No data available

Additional Information
RTECS: Not available

To the best of our knowledge, the chemical, physical, and toxicological properties have not been thoroughly investigated.

SECTION 12: Ecological information

12.1 Toxicity
No data available

12.2 Persistence and degradability
No data available

12.3 Bioaccumulative potential
No data available

12.4 Mobility in soil
No data available

12.5 Results of PBT and vPvB assessment
PBT/vPvB assessment not available as chemical safety assessment not required/not conducted

12.6 Other adverse effects
An environmental hazard cannot be excluded in the event of unprofessional handling or disposal.
Toxic to aquatic life with long lasting effects.

SECTION 13: Disposal considerations

13.1 Waste treatment methods

Product
Offer surplus and non-recyclable solutions to a licensed disposal company.

Contaminated packaging
Dispose of as unused product.

SECTION 14: Transport information

DOT (US)
Not dangerous goods

IMDG
UN number: 3077 Class: 9 Packing group: III EMS-No: F-A, S-F
Proper shipping name: ENVIRONMENTALLY HAZARDOUS SUBSTANCE, SOLID, N.O.S. (Aroclor 1016, Aroclor 1254)
Marine pollutant : yes

IATA
UN number: 3077 Class: 9 Packing group: III
Proper shipping name: Environmentally hazardous substance, solid, n.o.s. (Aroclor 1016, Aroclor 1254)

Further information
EHS-Mark required (ADR 2.2.9.1.10, IMDG code 2.10.3) for single packagings and combination packagings containing inner packagings with Dangerous Goods > 5L for liquids or > 5kg for solids.

SECTION 15: Regulatory information

SARA 302 Components
No chemicals in this material are subject to the reporting requirements of SARA Title III, Section 302.

SARA 313 Components
This material does not contain any chemical components with known CAS numbers that exceed the threshold (De Minimis) reporting levels established by SARA Title III, Section 313.

SARA 311/312 Hazards
No SARA Hazards

Massachusetts Right To Know Components

	CAS-No.	Revision Date
Aroclor 1254	11097-69-1	1993-02-16
Aroclor 1242	53469-21-9	1993-02-16

Pennsylvania Right To Know Components

	CAS-No.	Revision Date
Soil	-	

New Jersey Right To Know Components

	CAS-No.	Revision Date
Soil	-	

California Prop. 65 Components

	CAS-No.	Revision Date
WARNING! This product contains a chemical known to the State of California to cause cancer.Aroclor 1260	11096-82-5	2008-08-01

Aroclor 1254	11097-69-1	2008-08-01
PCB - Aroclor 1221	11104-28-2	2008-08-01
Aroclor 1232	11141-16-5	2008-08-01
Aroclor 1248	12672-29-6	2008-08-01
Aroclor 1016	12674-11-2	2008-08-01
PCB - Aroclor 1262	37324-23-5	2008-08-01
PCB- Aroclor 1268	11100-14-4	2008-08-01
Aroclor 1242	53469-21-9	2008-08-01

WARNING: This product contains a chemical known to the State of California to cause birth defects or other reproductive harm.Aroclor 1260	CAS-No. 11096-82-5	Revision Date 2008-08-01
Aroclor 1254	11097-69-1	2008-08-01
PCB - Aroclor 1221	11104-28-2	2008-08-01
Aroclor 1232	11141-16-5	2008-08-01
Aroclor 1248	12672-29-6	2008-08-01
Aroclor 1016	12674-11-2	2008-08-01
PCB - Aroclor 1262	37324-23-5	2008-08-01
PCB- Aroclor 1268	11100-14-4	2008-08-01
Aroclor 1242	53469-21-9	2008-08-01

SECTION 16: Other information

Further information

The above information is believed to be correct but does not purport to be all inclusive and shall be used only as a guide. The information in this document is based on the present state of our knowledge and is applicable to the product with regard to appropriate safety precautions. It does not represent any guarantee of the properties of the product. Sigma-Aldrich Corporation and its Affiliates shall not be held liable for any damage resulting from handling or from contact with the above product. See www.sigma-aldrich.com and/or the reverse side of invoice or packing slip for additional terms and conditions of sale.

The branding on the header and/or footer of this document may temporarily not visually match the product purchased as we transition our branding. However, all of the information in the document regarding the product remains unchanged and matches the product ordered. For further information please contact mlsbranding@sial.com.

Version: 8.1 Revision Date: 03/28/2020 Print Date: 06/02/2020

SAFETY DATA SHEET

Version 6.7
Revision Date 09/06/2022
Print Date 10/08/2022

SECTION 1: Identification of the substance/mixture and of the company/undertaking

1.1 Product identifiers

Product name : Selenium

Product Number : 209651
Brand : Aldrich
Index-No. : 034-001-00-2
CAS-No. : 7782-49-2

1.2 Relevant identified uses of the substance or mixture and uses advised against

Identified uses : Laboratory chemicals, Synthesis of substances

1.3 Details of the supplier of the safety data sheet

Company : Sigma-Aldrich Inc.
3050 SPRUCE ST
ST. LOUIS MO 63103
UNITED STATES

Telephone : +1 314 771-5765
Fax : +1 800 325-5052

1.4 Emergency telephone

Emergency Phone # : 800-424-9300 CHEMTREC (USA) +1-703-527-3887 CHEMTREC (International) 24 Hours/day; 7 Days/week

SECTION 2: Hazards identification

2.1 Classification of the substance or mixture

GHS Classification in accordance with 29 CFR 1910 (OSHA HCS)

Acute toxicity, Oral (Category 3), H301
Acute toxicity, Inhalation (Category 3), H331
Specific target organ toxicity - repeated exposure (Category 2), H373
Long-term (chronic) aquatic hazard (Category 4), H413

For the full text of the H-Statements mentioned in this Section, see Section 16.

2.2 GHS Label elements, including precautionary statements

Pictogram

Signal Word Danger

Hazard statement(s)

H301 + H331	Toxic if swallowed or if inhaled.
H373	May cause damage to organs through prolonged or repeated exposure.
H413	May cause long lasting harmful effects to aquatic life.

Precautionary statement(s)

P260	Do not breathe dust.
P264	Wash skin thoroughly after handling.
P270	Do not eat, drink or smoke when using this product.
P271	Use only outdoors or in a well-ventilated area.
P273	Avoid release to the environment.
P301 + P310 + P330	IF SWALLOWED: Immediately call a POISON CENTER/ doctor. Rinse mouth.
P304 + P340 + P311	IF INHALED: Remove person to fresh air and keep comfortable for breathing. Call a POISON CENTER/ doctor.
P314	Get medical advice/ attention if you feel unwell.
P403 + P233	Store in a well-ventilated place. Keep container tightly closed.
P405	Store locked up.
P501	Dispose of contents/ container to an approved waste disposal plant.

2.3 Hazards not otherwise classified (HNOC) or not covered by GHS - none

SECTION 3: Composition/information on ingredients

3.1 Substances

Formula	:	Se
Molecular weight	:	78.96 g/mol
CAS-No.	:	7782-49-2
EC-No.	:	231-957-4
Index-No.	:	034-001-00-2

Component	Classification	Concentration
Selenium		
	Acute Tox. 3; STOT RE 2; Aquatic Chronic 4; H301, H331, H373, H413	<= 100 %

For the full text of the H-Statements mentioned in this Section, see Section 16.

SECTION 4: First aid measures

4.1 Description of first-aid measures

General advice
First aiders need to protect themselves. Show this material safety data sheet to the doctor in attendance.

If inhaled
After inhalation: fresh air. Immediately call in physician. If breathing stops: immediately apply artificial respiration, if necessary also oxygen.

In case of skin contact
In case of skin contact: Take off immediately all contaminated clothing. Rinse skin with water/ shower. Consult a physician.

In case of eye contact
After eye contact: rinse out with plenty of water. Call in ophthalmologist. Remove contact lenses.

If swallowed
If swallowed: give water to drink (two glasses at most). Seek medical advice immediately. In exceptional cases only, if medical care is not available within one hour, induce vomiting (only in persons who are wide awake and fully conscious), administer activated charcoal (20 - 40 g in a 10% slurry) and consult a doctor as quickly as possible.

4.2 Most important symptoms and effects, both acute and delayed
The most important known symptoms and effects are described in the labelling (see section 2.2) and/or in section 11

4.3 Indication of any immediate medical attention and special treatment needed
No data available

SECTION 5: Firefighting measures

5.1 Extinguishing media

Suitable extinguishing media
Use extinguishing measures that are appropriate to local circumstances and the surrounding environment.

Unsuitable extinguishing media
For this substance/mixture no limitations of extinguishing agents are given.

5.2 Special hazards arising from the substance or mixture
Selenium/selenium oxides
Not combustible.
Ambient fire may liberate hazardous vapours.

5.3 Advice for firefighters
Stay in danger area only with self-contained breathing apparatus. Prevent skin contact by keeping a safe distance or by wearing suitable protective clothing.

5.4 Further information
Suppress (knock down) gases/vapors/mists with a water spray jet. Prevent fire extinguishing water from contaminating surface water or the ground water system.

SECTION 6: Accidental release measures

6.1 Personal precautions, protective equipment and emergency procedures
Advice for non-emergency personnel: Avoid generation and inhalation of dusts in all circumstances. Avoid substance contact. Ensure adequate ventilation. Evacuate the danger area, observe emergency procedures, consult an expert.
For personal protection see section 8.

6.2 Environmental precautions
Do not let product enter drains.

6.3 Methods and materials for containment and cleaning up

Cover drains. Collect, bind, and pump off spills. Observe possible material restrictions (see sections 7 and 10). Take up carefully. Dispose of properly. Clean up affected area. Avoid generation of dusts.

6.4 Reference to other sections

For disposal see section 13.

SECTION 7: Handling and storage

7.1 Precautions for safe handling

Advice on safe handling

Work under hood. Do not inhale substance/mixture.

Hygiene measures

Change contaminated clothing. Preventive skin protection recommended. Wash hands after working with substance.
For precautions see section 2.2.

7.2 Conditions for safe storage, including any incompatibilities

Storage conditions

Tightly closed. Dry. Keep in a well-ventilated place. Keep locked up or in an area accessible only to qualified or authorized persons.

Store under inert gas.

Storage class

Storage class (TRGS 510): 6.1C: Combustible, acute toxic Cat.3 / toxic compounds or compounds which causing chronic effects

7.3 Specific end use(s)

Apart from the uses mentioned in section 1.2 no other specific uses are stipulated

SECTION 8: Exposure controls/personal protection

8.1 Control parameters

Ingredients with workplace control parameters

Component	CAS-No.	Value	Control parameters	Basis
Selenium	7782-49-2	TWA	0.2 mg/m3	USA. Occupational Exposure Limits (OSHA) - Table Z-1 Limits for Air Contaminants
		TWA	0.2 mg/m3	USA. ACGIH Threshold Limit Values (TLV)
		TWA	0.2 mg/m3	USA. NIOSH Recommended Exposure Limits
		PEL	0.2 mg/m3	California permissible exposure limits for chemical contaminants (Title 8, Article 107)

8.2 Exposure controls

Appropriate engineering controls
Change contaminated clothing. Preventive skin protection recommended. Wash hands after working with substance.

Personal protective equipment

Eye/face protection
Use equipment for eye protection tested and approved under appropriate government standards such as NIOSH (US) or EN 166(EU). Safety glasses

Skin protection
This recommendation applies only to the product stated in the safety data sheet, supplied by us and for the designated use. When dissolving in or mixing with other substances and under conditions deviating from those stated in EN374 please contact the supplier of CE-approved gloves (e.g. KCL GmbH, D-36124 Eichenzell, Internet: www.kcl.de).
Full contact
Material: Nitrile rubber
Minimum layer thickness: 0.11 mm
Break through time: 480 min
Material tested:KCL 741 Dermatril® L

This recommendation applies only to the product stated in the safety data sheet, supplied by us and for the designated use. When dissolving in or mixing with other substances and under conditions deviating from those stated in EN374 please contact the supplier of CE-approved gloves (e.g. KCL GmbH, D-36124 Eichenzell, Internet: www.kcl.de).
Splash contact
Material: Nitrile rubber
Minimum layer thickness: 0.11 mm
Break through time: 480 min
Material tested:KCL 741 Dermatril® L

Body Protection
protective clothing

Respiratory protection
required when dusts are generated.
Our recommendations on filtering respiratory protection are based on the following standards: DIN EN 143, DIN 14387 and other accompanying standards relating to the used respiratory protection system.

Control of environmental exposure
Do not let product enter drains.

SECTION 9: Physical and chemical properties

9.1 Information on basic physical and chemical properties

a)	Appearance	Form: powder Color: light gray
b)	Odor	No data available
c)	Odor Threshold	No data available
d)	pH	No data available
e)	Melting point/freezing point	Melting point/range: 217 °C (423 °F) - lit.
f)	Initial boiling point and boiling range	684.9 °C 1264.8 °F - lit.
g)	Flash point	()Not applicable
h)	Evaporation rate	No data available
i)	Flammability (solid, gas)	No data available
j)	Upper/lower flammability or explosive limits	No data available
k)	Vapor pressure	> 0.001 hPa at 20 °C (68 °F)
l)	Vapor density	No data available
m)	Density	4.81 g/cm3 at 25 °C (77 °F) - lit.
	Relative density	4.825 °C
n)	Water solubility	0.1 g/l at 20.9 °C (69.6 °F) - insoluble
o)	Partition coefficient: n-octanol/water	log Pow: 5Not applicable for inorganic substances
p)	Autoignition temperature	220 - 250 °C (428 - 482 °F) at 1,013.25 hPa - Relative self-ignition temperature for solids
q)	Decomposition temperature	No data available
r)	Viscosity	No data available
s)	Explosive properties	No data available
t)	Oxidizing properties	none

9.2 Other safety information
No data available

SECTION 10: Stability and reactivity

10.1 Reactivity
No data available

10.2 Chemical stability
The product is chemically stable under standard ambient conditions (room temperature) .

10.3 Possibility of hazardous reactions
Risk of explosion with:
alkali amides
Metals
Oxygen
amides
cadmium
Potassium
sodium
nitrogen oxides
Tin
nitrogen trichloride
Risk of ignition or formation of inflammable gases or vapours with:
carbides
peroxi compounds
halogen-halogen compounds
halogen oxides
Fluorine
lithium silicide
barium peroxide
Uranium
Generates dangerous gases or fumes in contact with:
hydrochloric acid
sulfuric acid
Exothermic reaction with:
powdered aluminium
Beryllium
bromates
chromium(VI) oxide
chlorates
Nickel
Oxidizing agents
phosphorus
platinum
Nitric acid
silver oxide
Zinc
Alkali metals

10.4 Conditions to avoid
no information available

10.5 Incompatible materials
No data available

10.6 Hazardous decomposition products
In the event of fire: see section 5

SECTION 11: Toxicological information

11.1 Information on toxicological effects

Acute toxicity
Acute toxicity estimate Oral - Expert judgment - 100.1 mg/kg
Symptoms: Gastrointestinal disturbance
Acute toxicity estimate Inhalation - Expert judgment - 4 h - 0.51 mg/l - dust/mist

Dermal: No data available
No data available

Skin corrosion/irritation
Skin - reconstructed human epidermis (RhE)
Result: No skin irritation
(OECD Test Guideline 431)

Serious eye damage/eye irritation
Eyes - Bovine cornea
Result: No eye irritation - 4 h
(OECD Test Guideline 437)

Respiratory or skin sensitization
Local lymph node assay (LLNA) - Mouse
Result: negative
(OECD Test Guideline 429)

Germ cell mutagenicity
Test Type: Ames test
Test system: Salmonella typhimurium
Metabolic activation: with and without metabolic activation
Method: OECD Test Guideline 471
Result: negative

Test Type: Chromosome aberration test
Species: Mouse
Cell type: Bone marrow
Application Route: Intraperitoneal

Result: negative
Remarks: (ECHA)

Carcinogenicity
This product is or contains a component that is not classifiable as to its carcinogenicity based on its IARC, ACGIH, NTP, or EPA classification.

IARC: No ingredient of this product present at levels greater than or equal to 0.1% is identified as probable, possible or confirmed human carcinogen by IARC.

NTP: No ingredient of this product present at levels greater than or equal to 0.1% is identified as a known or anticipated carcinogen by NTP.

OSHA: No component of this product present at levels greater than or equal to 0.1% is on OSHA's list of regulated carcinogens.

Reproductive toxicity
No data available

Specific target organ toxicity - single exposure

No data available

Specific target organ toxicity - repeated exposure
May cause damage to organs through prolonged or repeated exposure.**Aspiration hazard**
No data available

11.2 Additional Information

Repeated dose toxicity - Rat - male and female - Oral - 13 Weeks - NOAEL (No observed adverse effect level) - 0.4 mg/kg
Remarks: (in analogy to similar products)
The value is given in analogy to the following substances: Sodium selenite

RTECS: VS7700000
anemia, Vomiting, Diarrhea, Cough, Difficulty in breathing, Acute selenium poisoning produces central nervous system effects, which include nervousness, convulsions, and drowsiness. Other signs of intoxication can include skin eruptions, lassitude, gastrointestinal distress, teeth that are discolored or decayed, odorous ("garlic") breath, and partial loss of hair and nails. Chronic exposure by inhalation can produce symptoms that include pallor, coating of the tongue, anemia, irritation of the mucosa, lumbar pain, liver and spleen damage, as well as any of the other previously mentioned symptoms. Chronic contact with selenium compounds may cause garlic odor of breath and sweat, dermatitis, and moderate emotional instability., Dermatitis, garlic-like breath odor, pallor, nervousness, depression
To the best of our knowledge, the chemical, physical, and toxicological properties have not been thoroughly investigated.

After absorption:

CNS disorders
Dizziness
muscular weakness
Headache
cardiovascular disorders
Shortness of breath
somnolence
Cough
Unconsciousness

Other dangerous properties can not be excluded.

Handle in accordance with good industrial hygiene and safety practice.

Stomach - Irregularities - Based on Human Evidence

Stomach - Irregularities - Based on Human Evidence

SECTION 12: Ecological information

12.1 Toxicity

Toxicity to fish	semi-static test LC50 - Oncorhynchus mykiss (rainbow trout) - > 100 mg/l - 96 h (OECD Test Guideline 203)

Remarks: (above the solubility limit in the test medium)

Toxicity to daphnia and other aquatic invertebrates	static test EC50 - Daphnia magna (Water flea) - > 100 mg/l - 48 h (OECD Test Guideline 202) Remarks: (above the solubility limit in the test medium)
Toxicity to algae	static test ErC50 - Pseudokirchneriella subcapitata (algae) - > 100 mg/l - 72 h (OECD Test Guideline 201) Remarks: (above the solubility limit in the test medium)
Toxicity to bacteria	static test EC50 - activated sludge - > 3,200 mg/l - 3 h (OECD Test Guideline 209)

12.2 Persistence and degradability

The methods for determining the biological degradability are not applicable to inorganic substances.

12.3 Bioaccumulative potential

Bioaccumulation	Lepomis macrochirus - 60 d - 640 µg/l(Selenium)
	Bioconcentration factor (BCF): 7.7

12.4 Mobility in soil

No data available

12.5 Results of PBT and vPvB assessment

PBT/vPvB assessment not available as chemical safety assessment not required/not conducted

12.6 Endocrine disrupting properties

No data available

12.7 Other adverse effects

Discharge into the environment must be avoided.

SECTION 13: Disposal considerations

13.1 Waste treatment methods

Product

Waste material must be disposed of in accordance with the national and local regulations. Leave chemicals in original containers. No mixing with other waste. Handle uncleaned containers like the product itself. See www.retrologistik.com for processes regarding the return of chemicals and containers, or contact us there if you have further questions.

SECTION 14: Transport information

DOT (US)

UN number: 3288 Class: 6.1 Packing group: III
Proper shipping name: Toxic solid, inorganic, n.o.s. (Selenium)
Reportable Quantity (RQ): 100 lbs
Reportable Quantity (RQ): 10 lbs

Poison Inhalation Hazard: No

IMDG
UN number: 3288 Class: 6.1 Packing group: III EMS-No: F-A, S-A
Proper shipping name: TOXIC SOLID, INORGANIC, N.O.S. (Selenium)

IATA
UN number: 3288 Class: 6.1 Packing group: III
Proper shipping name: Toxic solid, inorganic, n.o.s. (Selenium)

SECTION 15: Regulatory information

SARA 302 Components
This material does not contain any components with a section 302 EHS TPQ.

SARA 313 Components
The following components are subject to reporting levels established by SARA Title III, Section 313:

	CAS-No.	Revision Date
Selenium	7782-49-2	2007-07-01

SARA 311/312 Hazards
Acute Health Hazard, Chronic Health Hazard

Reportable Quantity : D010 lbs

Massachusetts Right To Know Components
No components are subject to the Massachusetts Right to Know Act.

SECTION 16: Other information

Further information
The above information is believed to be correct but does not purport to be all inclusive and shall be used only as a guide. The information in this document is based on the present state of our knowledge and is applicable to the product with regard to appropriate safety precautions. It does not represent any guarantee of the properties of the product. Sigma-Aldrich Corporation and its Affiliates shall not be held liable for any damage resulting from handling or from contact with the above product. See www.sigma-aldrich.com and/or the reverse side of invoice or packing slip for additional terms and conditions of sale.

The branding on the header and/or footer of this document may temporarily not visually match the product purchased as we transition our branding. However, all of the information in the document regarding the product remains unchanged and matches the product ordered. For further information please contact mlsbranding@sial.com.

Version: 6.7 Revision Date: 09/06/2022 Print Date: 10/08/2022

SAFETY DATA SHEET

Version 6.3
Revision Date 04/18/2021
Print Date 11/21/2022

SECTION 1: Identification of the substance/mixture and of the company/undertaking

1.1 Product identifiers

Product name : Silver

Product Number : 295744
Brand : Aldrich
CAS-No. : 7440-22-4

1.2 Relevant identified uses of the substance or mixture and uses advised against

Identified uses : Laboratory chemicals, Synthesis of substances

1.3 Details of the supplier of the safety data sheet

Company : Sigma-Aldrich Inc.
3050 SPRUCE ST
ST. LOUIS MO 63103
UNITED STATES

Telephone : +1 314 771-5765
Fax : +1 800 325-5052

1.4 Emergency telephone

Emergency Phone # : 800-424-9300 CHEMTREC (USA) +1-703-527-3887 CHEMTREC (International) 24 Hours/day; 7 Days/week

SECTION 2: Hazards identification

2.1 Classification of the substance or mixture

GHS Classification in accordance with 29 CFR 1910 (OSHA HCS)

Short-term (acute) aquatic hazard (Category 1), H400
Long-term (chronic) aquatic hazard (Category 1), H410

For the full text of the H-Statements mentioned in this Section, see Section 16.

2.2 GHS Label elements, including precautionary statements

Pictogram

Signal word Warning

Hazard statement(s)
H410 Very toxic to aquatic life with long lasting effects.

Precautionary statement(s)

P273	Avoid release to the environment.
P391	Collect spillage.
P501	Dispose of contents/ container to an approved waste disposal plant.

2.3 Hazards not otherwise classified (HNOC) or not covered by GHS - none

SECTION 3: Composition/information on ingredients

3.1 Substances

Formula	:	Ag
Molecular weight	:	107.87 g/mol
CAS-No.	:	7440-22-4
EC-No.	:	231-131-3

Component	Classification	Concentration
colloidal silver		
	Aquatic Acute 1; Aquatic Chronic 1; H400, H410 M-Factor - Aquatic Acute: 100 - Aquatic Chronic: 100	<= 100 %

For the full text of the H-Statements mentioned in this Section, see Section 16.

SECTION 4: First aid measures

4.1 Description of first-aid measures

General advice
Consult a physician. Show this material safety data sheet to the doctor in attendance.

If inhaled
If breathed in, move person into fresh air. If not breathing, give artificial respiration. Consult a physician.

In case of skin contact
Wash off with soap and plenty of water. Consult a physician.

In case of eye contact
Flush eyes with water as a precaution.

If swallowed
Never give anything by mouth to an unconscious person. Rinse mouth with water. Consult a physician.

4.2 Most important symptoms and effects, both acute and delayed
The most important known symptoms and effects are described in the labelling (see section 2.2) and/or in section 11

4.3 Indication of any immediate medical attention and special treatment needed
No data available

SECTION 5: Firefighting measures

5.1 Extinguishing media

Suitable extinguishing media
Use water spray, alcohol-resistant foam, dry chemical or carbon dioxide.

5.2 Special hazards arising from the substance or mixture

Silver/silver oxides
Not combustible.

5.3 Advice for firefighters

Wear self-contained breathing apparatus for firefighting if necessary.

5.4 Further information

No data available

SECTION 6: Accidental release measures

6.1 Personal precautions, protective equipment and emergency procedures

Avoid dust formation. Avoid breathing vapors, mist or gas. Ensure adequate ventilation.
For personal protection see section 8.

6.2 Environmental precautions

Prevent further leakage or spillage if safe to do so. Do not let product enter drains.
Discharge into the environment must be avoided.

6.3 Methods and materials for containment and cleaning up

Pick up and arrange disposal without creating dust. Sweep up and shovel. Keep in suitable, closed containers for disposal.

6.4 Reference to other sections

For disposal see section 13.

SECTION 7: Handling and storage

7.1 Precautions for safe handling

Advice on safe handling
Further processing of solid materials may result in the formation of combustible dusts. The potential for combustible dust formation should be taken into consideration before additional processing occurs.

Advice on protection against fire and explosion
Provide appropriate exhaust ventilation at places where dust is formed.

Hygiene measures
Handle in accordance with good industrial hygiene and safety practice. Wash hands before breaks and at the end of workday.
For precautions see section 2.2.

7.2 Conditions for safe storage, including any incompatibilities

Storage conditions
Keep container tightly closed in a dry and well-ventilated place.

Air sensitive.
Storage class (TRGS 510): 13: Non Combustible Solids

7.3 Specific end use(s)

Apart from the uses mentioned in section 1.2 no other specific uses are stipulated

SECTION 8: Exposure controls/personal protection

8.1 Control parameters

Ingredients with workplace control parameters

Component	CAS-No.	Value	Control parameters	Basis
colloidal silver	7440-22-4	TWA	0.1 mg/m3	USA. ACGIH Threshold Limit Values (TLV)
		PEL	0.01 mg/m3	California permissible exposure limits for chemical contaminants (Title 8, Article 107)
		TWA	0.01 mg/m3	USA. Occupational Exposure Limits (OSHA) - Table Z-1 Limits for Air Contaminants

8.2 Exposure controls

Appropriate engineering controls

Handle in accordance with good industrial hygiene and safety practice. Wash hands before breaks and at the end of workday.

Personal protective equipment

Eye/face protection

Use equipment for eye protection tested and approved under appropriate government standards such as NIOSH (US) or EN 166(EU).

Skin protection

Handle with gloves. Gloves must be inspected prior to use. Use proper glove removal technique (without touching glove's outer surface) to avoid skin contact with this product. Dispose of contaminated gloves after use in accordance with applicable laws and good laboratory practices. Wash and dry hands.

Full contact
Material: Nitrile rubber
Minimum layer thickness: 0.11 mm
Break through time: 480 min
Material tested:Dermatril® (KCL 740 / Aldrich Z677272, Size M)

Splash contact
Material: Nitrile rubber
Minimum layer thickness: 0.11 mm
Break through time: 480 min
Material tested:Dermatril® (KCL 740 / Aldrich Z677272, Size M)

data source: KCL GmbH, D-36124 Eichenzell, phone +49 (0)6659 87300, e-mail sales@kcl.de, test method: EN374
If used in solution, or mixed with other substances, and under conditions which differ from EN 374, contact the supplier of the EC approved gloves. This recommendation is advisory only and must be evaluated by an industrial hygienist and safety officer familiar with the specific situation of anticipated use by our customers. It should not be construed as offering an approval for any specific use scenario.

Body Protection
Choose body protection in relation to its type, to the concentration and amount of dangerous substances, and to the specific work-place., The type of protective equipment must be selected according to the concentration and amount of the dangerous substance at the specific workplace.

Respiratory protection
Respiratory protection is not required. Where protection from nuisance levels of dusts are desired, use type N95 (US) or type P1 (EN 143) dust masks. Use respirators and components tested and approved under appropriate government standards such as NIOSH (US) or CEN (EU).

Control of environmental exposure
Prevent further leakage or spillage if safe to do so. Do not let product enter drains. Discharge into the environment must be avoided.

SECTION 9: Physical and chemical properties

9.1 Information on basic physical and chemical properties

a)	Appearance	Form: solid
b)	Odor	No data available
c)	Odor Threshold	No data available
d)	pH	No data available
e)	Melting point/freezing point	Melting point/range: 960 °C (1760 °F) - lit.
f)	Initial boiling point and boiling range	2,212 °C 4,014 °F - lit.
g)	Flash point	()Not applicable
h)	Evaporation rate	No data available
i)	Flammability (solid, gas)	The product is not flammable.
j)	Upper/lower flammability or explosive limits	No data available
k)	Vapor pressure	No data available
l)	Vapor density	No data available
m)	Relative density	No data available
n)	Water solubility	insoluble
o)	Partition coefficient: n-octanol/water	Not applicable for inorganic substances
p)	Autoignition temperature	No data available
q)	Decomposition temperature	No data available
r)	Viscosity	No data available
s)	Explosive properties	No data available

t) Oxidizing properties No data available

9.2 Other safety information
No data available

SECTION 10: Stability and reactivity

10.1 Reactivity
No data available

10.2 Chemical stability
Stable under recommended storage conditions.

10.3 Possibility of hazardous reactions
No data available

10.4 Conditions to avoid
No data available

10.5 Incompatible materials
Strong oxidizing agents

10.6 Hazardous decomposition products
In the event of fire: see section 5

SECTION 11: Toxicological information

11.1 Information on toxicological effects

Acute toxicity
No data available

No data available

Inhalation: No data available

Inhalation: No data available

Dermal: No data available

Dermal: No data available
No data available
No data available

Skin corrosion/irritation
No data available

Skin - Rabbit
Result: No skin irritation
(OECD Test Guideline 404)

Serious eye damage/eye irritation
No data available
Eyes - Rabbit
Result: No eye irritation

(OECD Test Guideline 405)

Respiratory or skin sensitization
No data available
No data available

Germ cell mutagenicity
No data available
No data available

Carcinogenicity

IARC: No ingredient of this product present at levels greater than or equal to 0.1% is identified as probable, possible or confirmed human carcinogen by IARC.

NTP: No ingredient of this product present at levels greater than or equal to 0.1% is identified as a known or anticipated carcinogen by NTP.

OSHA: No component of this product present at levels greater than or equal to 0.1% is on OSHA's list of regulated carcinogens.

Reproductive toxicity
No data available
No data available

Specific target organ toxicity - single exposure
No data available**Specific target organ toxicity - repeated exposure**
No data available

Aspiration hazard
No data available

11.2 Additional Information
Not available

To the best of our knowledge, the chemical, physical, and toxicological properties have not been thoroughly investigated.

Hazardous properties cannot be excluded but are unlikely when the product is handled appropriately.

Further data:

Handle in accordance with good industrial hygiene and safety practice.

SECTION 12: Ecological information

12.1 Toxicity
No data available

Toxicity to fish semi-static test LC50 - Pimephales promelas (fathead minnow) -

0.0021 mg/l - 96 h
Remarks: (ECOTOX Database)

12.2 Persistence and degradability
No data available

12.3 Bioaccumulative potential
No data available

12.4 Mobility in soil
No data available

12.5 Results of PBT and vPvB assessment
PBT/vPvB assessment not available as chemical safety assessment not required/not conducted

12.6 Other adverse effects
An environmental hazard cannot be excluded in the event of unprofessional handling or disposal.
Very toxic to aquatic life with long lasting effects.
Discharge into the environment must be avoided.

SECTION 13: Disposal considerations

13.1 Waste treatment methods

Product
Offer surplus and non-recyclable solutions to a licensed disposal company.

Contaminated packaging
Dispose of as unused product.

SECTION 14: Transport information

DOT (US)
UN number: 3077 Class: 9 Packing group: III
Proper shipping name: Environmentally hazardous substance, solid, n.o.s.
Reportable Quantity (RQ): 1 lbs
Poison Inhalation Hazard: No

IMDG
UN number: 3077 Class: 9 Packing group: III EMS-No: F-A, S-F
Proper shipping name: ENVIRONMENTALLY HAZARDOUS SUBSTANCE, SOLID, N.O.S.
Marine pollutant : yes

IATA
UN number: 3077 Class: 9 Packing group: III
Proper shipping name: Environmentally hazardous substance, solid, n.o.s.
Further information
EHS-Mark required (ADR 2.2.9.1.10, IMDG code 2.10.3) for single packagings and combination packagings containing inner packagings with Dangerous Goods > 5L for liquids or > 5kg for solids.

SECTION 15: Regulatory information

SARA 302 Components
No chemicals in this material are subject to the reporting requirements of SARA Title III, Section 302.

SARA 313 Components
The following components are subject to reporting levels established by SARA Title III, Section 313:

	CAS-No.	Revision Date
colloidal silver	7440-22-4	2007-07-01

SARA 311/312 Hazards
No SARA Hazards

Massachusetts Right To Know Components
No components are subject to the Massachusetts Right to Know Act.

Pennsylvania Right To Know Components

	CAS-No.	Revision Date
colloidal silver	7440-22-4	2007-07-01

SECTION 16: Other information

Further information

The above information is believed to be correct but does not purport to be all inclusive and shall be used only as a guide. The information in this document is based on the present state of our knowledge and is applicable to the product with regard to appropriate safety precautions. It does not represent any guarantee of the properties of the product. Sigma-Aldrich Corporation and its Affiliates shall not be held liable for any damage resulting from handling or from contact with the above product. See www.sigma-aldrich.com and/or the reverse side of invoice or packing slip for additional terms and conditions of sale.

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Version: 6.3 Revision Date: 04/18/2021 Print Date: 11/21/2022

SAFETY DATA SHEET

Version 6.10
Revision Date 03/21/2020
Print Date 05/29/2020

SECTION 1: Identification of the substance/mixture and of the company/undertaking

1.1 Product identifiers

Product name : Toluene

Product Number : 244511
Brand : Sigma-Aldrich
Index-No. : 601-021-00-3
CAS-No. : 108-88-3

1.2 Relevant identified uses of the substance or mixture and uses advised against

Identified uses : Laboratory chemicals, Synthesis of substances

1.3 Details of the supplier of the safety data sheet

Company : Sigma-Aldrich Inc.
3050 Spruce Street
ST. LOUIS MO 63103
UNITED STATES

Telephone : +1 314 771-5765
Fax : +1 800 325-5052

1.4 Emergency telephone number

Emergency Phone # : +1-703-527-3887

SECTION 2: Hazards identification

2.1 Classification of the substance or mixture

GHS Classification in accordance with 29 CFR 1910 (OSHA HCS)

Flammable liquids (Category 2), H225
Skin irritation (Category 2), H315
Reproductive toxicity (Category 2), H361
Specific target organ toxicity - single exposure (Category 3), Central nervous system, H336
Specific target organ toxicity - repeated exposure (Category 2), Central nervous system, H373
Aspiration hazard (Category 1), H304
Short-term (acute) aquatic hazard (Category 2), H401
Long-term (chronic) aquatic hazard (Category 3), H412

For the full text of the H-Statements mentioned in this Section, see Section 16.

2.2 GHS Label elements, including precautionary statements

Pictogram	
Signal word	Danger

Hazard statement(s)

H225	Highly flammable liquid and vapour.
H304	May be fatal if swallowed and enters airways.
H315	Causes skin irritation.
H336	May cause drowsiness or dizziness.
H361	Suspected of damaging fertility or the unborn child.
H373	May cause damage to organs (Central nervous system) through prolonged or repeated exposure.
H401	Toxic to aquatic life.
H412	Harmful to aquatic life with long lasting effects.

Precautionary statement(s)

P201	Obtain special instructions before use.
P202	Do not handle until all safety precautions have been read and understood.
P210	Keep away from heat/sparks/open flames/hot surfaces. No smoking.
P233	Keep container tightly closed.
P240	Ground/bond container and receiving equipment.
P241	Use explosion-proof electrical/ ventilating/ lighting equipment.
P242	Use only non-sparking tools.
P243	Take precautionary measures against static discharge.
P260	Do not breathe dust/ fume/ gas/ mist/ vapours/ spray.
P264	Wash skin thoroughly after handling.
P271	Use only outdoors or in a well-ventilated area.
P273	Avoid release to the environment.
P280	Wear protective gloves/ protective clothing/ eye protection/ face protection.
P301 + P310	IF SWALLOWED: Immediately call a POISON CENTER/doctor.
P303 + P361 + P353	IF ON SKIN (or hair): Take off immediately all contaminated clothing. Rinse skin with water/shower.
P304 + P340 + P312	IF INHALED: Remove person to fresh air and keep comfortable for breathing. Call a POISON CENTER/doctor if you feel unwell.
P308 + P313	IF exposed or concerned: Get medical advice/ attention.
P331	Do NOT induce vomiting.
P332 + P313	If skin irritation occurs: Get medical advice/ attention.
P362	Take off contaminated clothing and wash before reuse.
P370 + P378	In case of fire: Use dry sand, dry chemical or alcohol-resistant foam to extinguish.
P403 + P233	Store in a well-ventilated place. Keep container tightly closed.
P403 + P235	Store in a well-ventilated place. Keep cool.
P405	Store locked up.
P501	Dispose of contents/ container to an approved waste disposal plant.

2.3 Hazards not otherwise classified (HNOC) or not covered by GHS - none

SECTION 3: Composition/information on ingredients

3.1 Substances

Formula	:	C_7H_8
Molecular weight	:	92.14 g/mol
CAS-No.	:	108-88-3
EC-No.	:	203-625-9
Index-No.	:	601-021-00-3

Component	Classification	Concentration
Toluene		
	Flam. Liq. 2; Skin Irrit. 2; Repr. 2; STOT SE 3; STOT RE 2; Asp. Tox. 1; Aquatic Acute 2; Aquatic Chronic 3; H225, H315, H361, H336, H373, H304, H401, H412 Concentration limits: 20 %: STOT SE 3, H336;	<= 100 %

For the full text of the H-Statements mentioned in this Section, see Section 16.

SECTION 4: First aid measures

4.1 Description of first aid measures

General advice
Consult a physician. Show this safety data sheet to the doctor in attendance.Move out of dangerous area.

If inhaled
If breathed in, move person into fresh air. If not breathing, give artificial respiration. Consult a physician.

In case of skin contact
Wash off with soap and plenty of water. Consult a physician.

In case of eye contact
Flush eyes with water as a precaution.

If swallowed
Do NOT induce vomiting. Never give anything by mouth to an unconscious person. Rinse mouth with water. Consult a physician.

4.2 Most important symptoms and effects, both acute and delayed

The most important known symptoms and effects are described in the labelling (see section 2.2) and/or in section 11

4.3 Indication of any immediate medical attention and special treatment needed

No data available

SECTION 5: Firefighting measures

5.1 Extinguishing media

Suitable extinguishing media
Dry powder Dry sand

Unsuitable extinguishing media
Do NOT use water jet.

5.2 Special hazards arising from the substance or mixture
Carbon oxides
Combustible.

5.3 Advice for firefighters
Wear self-contained breathing apparatus for firefighting if necessary.

5.4 Further information
Use water spray to cool unopened containers.

SECTION 6: Accidental release measures

6.1 Personal precautions, protective equipment and emergency procedures
Use personal protective equipment. Avoid breathing vapours, mist or gas. Ensure adequate ventilation. Remove all sources of ignition. Evacuate personnel to safe areas. Beware of vapours accumulating to form explosive concentrations. Vapours can accumulate in low areas.
For personal protection see section 8.

6.2 Environmental precautions
Prevent further leakage or spillage if safe to do so. Do not let product enter drains. Discharge into the environment must be avoided.

6.3 Methods and materials for containment and cleaning up
Contain spillage, and then collect with non-combustible absorbent material, (e.g. sand, earth, diatomaceous earth, vermiculite) and place in container for disposal according to local / national regulations (see section 13).

6.4 Reference to other sections
For disposal see section 13.

SECTION 7: Handling and storage

7.1 Precautions for safe handling
Avoid contact with skin and eyes. Avoid inhalation of vapour or mist.
Use explosion-proof equipment.Keep away from sources of ignition - No smoking.Take measures to prevent the build up of electrostatic charge.
For precautions see section 2.2.

7.2 Conditions for safe storage, including any incompatibilities
Keep container tightly closed in a dry and well-ventilated place. Containers which are opened must be carefully resealed and kept upright to prevent leakage.

Handle and store under inert gas.
Storage class (TRGS 510): 3: Flammable liquids

7.3 Specific end use(s)

Apart from the uses mentioned in section 1.2 no other specific uses are stipulated

SECTION 8: Exposure controls/personal protection

8.1 Control parameters

Components with workplace control parameters

Component	CAS-No.	Value	Control parameters	Basis
Toluene	108-88-3	TWA	100 ppm 375 mg/m3	USA. OSHA - TABLE Z-1 Limits for Air Contaminants - 1910.1000
		STEL	150 ppm 560 mg/m3	USA. OSHA - TABLE Z-1 Limits for Air Contaminants - 1910.1000
		TWA	200 ppm	USA. Occupational Exposure Limits (OSHA) - Table Z-2
	Remarks	Z37.12-1967		
		CEIL	300 ppm	USA. Occupational Exposure Limits (OSHA) - Table Z-2
		Z37.12-1967		
		Peak	500 ppm	USA. Occupational Exposure Limits (OSHA) - Table Z-2
		Z37.12-1967		
		TWA	20 ppm	USA. ACGIH Threshold Limit Values (TLV)
		Visual impairment Female reproductive Pregnancy loss 2018 Adoption Substances for which there is a Biological Exposure Index or Indices (see BEI® section) Not classifiable as a human carcinogen		
		TWA	100 ppm 375 mg/m3	USA. NIOSH Recommended Exposure Limits
		ST	150 ppm 560 mg/m3	USA. NIOSH Recommended Exposure Limits

Biological occupational exposure limits

Component	CAS-No.	Parameters	Value	Biological specimen	Basis
Toluene	108-88-3	Toluene	0.02 mg/l	In blood	ACGIH - Biological Exposure Indices (BEI)
	Remarks	Prior to last shift of workweek			
		Toluene	0.03 mg/l	Urine	ACGIH - Biological Exposure Indices (BEI)
		End of shift (As soon as possible after exposure ceases)			

		o-Cresol	0.3mg/g Creatinine	Urine	ACGIH - Biological Exposure Indices (BEI)
		End of shift (As soon as possible after exposure ceases)			

Derived No Effect Level (DNEL)

Application Area	Exposure routes	Health effect	Value
Workers	Inhalation	Acute systemic effects	384 mg/m3
Workers	Inhalation	Acute local effects	384 mg/m3
Workers	Skin contact	Long-term systemic effects	384mg/kg BW/d
Workers	Inhalation	Long-term systemic effects	192 mg/m3
Workers	Inhalation	Long-term local effects	192 mg/m3
Consumers	Inhalation	Acute systemic effects	226 mg/m3
Consumers	Inhalation	Acute local effects	226 mg/m3
Consumers	Skin contact	Long-term systemic effects	226mg/kg BW/d
Consumers	Inhalation	Long-term systemic effects	56.5 mg/m3
Consumers	Ingestion	Long-term systemic effects	8.13mg/kg BW/d

Predicted No Effect Concentration (PNEC)

Compartment	Value
Soil	2.89 mg/kg
Marine water	0.68 mg/l
Fresh water	0.68 mg/l
Marine sediment	16.39 mg/kg
Fresh water sediment	16.39 mg/kg
Sewage treatment plant	13.61 mg/l
Aquatic intermittent release	0.68 mg/l

8.2 Exposure controls

Appropriate engineering controls
Handle in accordance with good industrial hygiene and safety practice. Wash hands before breaks and at the end of workday.

Personal protective equipment

Eye/face protection
Face shield and safety glasses Use equipment for eye protection tested and approved under appropriate government standards such as NIOSH (US) or EN 166(EU).

Skin protection
Handle with gloves. Gloves must be inspected prior to use. Use proper glove removal technique (without touching glove's outer surface) to avoid skin contact with this product. Dispose of contaminated gloves after use in accordance with applicable laws and good laboratory practices. Wash and dry hands.

Full contact
Material: Fluorinated rubber
Minimum layer thickness: 0.7 mm
Break through time: 480 min
Material tested:Vitoject® (KCL 890 / Aldrich Z677698, Size M)

Splash contact
Material: Fluorinated rubber

Minimum layer thickness: 0.7 mm
Break through time: 480 min
Material tested:Vitoject® (KCL 890 / Aldrich Z677698, Size M)

data source: KCL GmbH, D-36124 Eichenzell, phone +49 (0)6659 87300, e-mail sales@kcl.de, test method: EN374
If used in solution, or mixed with other substances, and under conditions which differ from EN 374, contact the supplier of the CE approved gloves. This recommendation is advisory only and must be evaluated by an industrial hygienist and safety officer familiar with the specific situation of anticipated use by our customers. It should not be construed as offering an approval for any specific use scenario.

Body Protection
Complete suit protecting against chemicals, Flame retardant antistatic protective clothing., The type of protective equipment must be selected according to the concentration and amount of the dangerous substance at the specific workplace.

Respiratory protection
Where risk assessment shows air-purifying respirators are appropriate use a full-face respirator with multi-purpose combination (US) or type ABEK (EN 14387) respirator cartridges as a backup to engineering controls. If the respirator is the sole means of protection, use a full-face supplied air respirator. Use respirators and components tested and approved under appropriate government standards such as NIOSH (US) or CEN (EU).

Control of environmental exposure
Prevent further leakage or spillage if safe to do so. Do not let product enter drains. Discharge into the environment must be avoided.

SECTION 9: Physical and chemical properties

9.1 Information on basic physical and chemical properties

a)	Appearance	Form: liquid
b)	Odour	benzene-like
c)	Odour Threshold	No data available
d)	pH	Not applicable
e)	Melting point/freezing point	Melting point/range: -93 °C (-135 °F)
f)	Initial boiling point and boiling range	110 - 111 °C 230 - 232 °F
g)	Flash point	4.0 °C (39.2 °F) - c.c.
h)	Evaporation rate	No data available
i)	Flammability (solid, gas)	No data available
j)	Upper/lower flammability or explosive limits	Upper explosion limit: 7.1 %(V) Lower explosion limit: 1.2 %(V)
k)	Vapour pressure	30.88 hPa at 21.1 °C (70.0 °F)
l)	Vapour density	3.18
m)	Relative density	0.865 g/mL at 25 °C (77 °F)

n)	Water solubility	0.58 g/l at 25 °C (77 °F) - partly soluble
o)	Partition coefficient: n-octanol/water	log Pow: 2.73 at 20 °C (68 °F) - Bioaccumulation is not expected.
p)	Auto-ignition temperature	535.0 °C (995.0 °F)
q)	Decomposition temperature	No data available
r)	Viscosity	No data available
s)	Explosive properties	No data available
t)	Oxidizing properties	No data available

9.2 Other safety information

Conductivity	< 0.01 µS/cm
Surface tension	27.73 mN/m at 0.516g/l at 25 °C (77 °F)
Relative vapour density	3.18

SECTION 10: Stability and reactivity

10.1 Reactivity
No data available

10.2 Chemical stability
Stable under recommended storage conditions.

10.3 Possibility of hazardous reactions
Vapours may form explosive mixture with air.

10.4 Conditions to avoid
Heat, flames and sparks.

10.5 Incompatible materials
Strong oxidizing agents

10.6 Hazardous decomposition products
Hazardous decomposition products formed under fire conditions. - Carbon oxides
Other decomposition products - No data available
In the event of fire: see section 5

SECTION 11: Toxicological information

11.1 Information on toxicological effects

Acute toxicity
LD50 Oral - Rat - male - 5,580 mg/kg
(Tested according to Directive 92/69/EEC.)
LC50 Inhalation - Rat - male and female - 4 h - 25.7 mg/l
(OECD Test Guideline 403)
LD50 Dermal - Rabbit - > 5,000 mg/kg
Remarks: (ECHA)
No data available

Skin corrosion/irritation
Skin - Rabbit
Result: irritating - 4 h
Remarks: (ECHA)

Serious eye damage/eye irritation
Eyes - Rabbit
Result: slight irritation
(OECD Test Guideline 405)

Respiratory or skin sensitisation
Maximisation Test - Guinea pig
Result: negative
(Regulation (EC) No. 440/2008, Annex, B.6)

Germ cell mutagenicity
In vitro mammalian cell gene mutation test
Mouse lymphoma test
Result: negative
Ames test
S. typhimurium
Result: negative

Rat - Bone marrow
Result: negative
(ECHA)

Carcinogenicity

IARC:	No component of this product present at levels greater than or equal to 0.1% is identified as probable, possible or confirmed human carcinogen by IARC.
NTP:	No component of this product present at levels greater than or equal to 0.1% is identified as a known or anticipated carcinogen by NTP.
OSHA:	No component of this product present at levels greater than or equal to 0.1% is on OSHA's list of regulated carcinogens.

Reproductive toxicity
Suspected of damaging the unborn child.

Specific target organ toxicity - single exposure
May cause drowsiness or dizziness. - Central nervous system

Specific target organ toxicity - repeated exposure
May cause damage to organs through prolonged or repeated exposure. - Central nervous system

Aspiration hazard
Aspiration hazard, Aspiration may cause pulmonary oedema and pneumonitis.

Additional Information
RTECS: XS5250000

Drowsiness, irritant effects, Dizziness, Convulsions, Headache, Nausea, Vomiting, Circulatory collapse, somnolence, inebriation, Unconsciousness, respiratory arrest, CNS disorders, respiratory paralysis, death
To the best of our knowledge, the chemical, physical, and toxicological properties have not been thoroughly investigated.

Stomach - Irregularities - Based on Human Evidence

Stomach - Irregularities - Based on Human Evidence

SECTION 12: Ecological information

12.1 Toxicity

Toxicity to fish	flow-through test LC50 - Oncorhynchus kisutch (coho salmon) - 5.5 mg/l - 96 h Remarks: (ECHA)
Toxicity to daphnia and other aquatic invertebrates	EC50 - Ceriodaphnia dubia (water flea) - 3.78 mg/l - 48 h (US-EPA)
Toxicity to bacteria	static test EC50 - Bacteria - 84 mg/l - 24 h Remarks: (ECHA)

12.2 Persistence and degradability

Biodegradability	aerobic - Exposure time 20 d Result: 86 % - Readily biodegradable. Remarks: (IUCLID)
Theoretical oxygen demand	3,130 mg/g Remarks: (Lit.)

12.3 Bioaccumulative potential

Bioaccumulation	Leuciscus idus (Golden orfe) - 3 d - 0.05 mg/l(Toluene) Bioconcentration factor (BCF): 90

12.4 Mobility in soil

No data available

12.5 Results of PBT and vPvB assessment

PBT/vPvB assessment not available as chemical safety assessment not required/not conducted

12.6 Other adverse effects

An environmental hazard cannot be excluded in the event of unprofessional handling or disposal.
Toxic to aquatic life.
No data available

SECTION 13: Disposal considerations

13.1 Waste treatment methods

Product

Contact a licensed professional waste disposal service to dispose of this material. Offer surplus and non-recyclable solutions to a licensed disposal company. Burn in a chemical incinerator equipped with an afterburner and scrubber but exert extra care in igniting as this material is highly flammable.

Contaminated packaging

Dispose of as unused product.

SECTION 14: Transport information

DOT (US)
UN number: 1294 Class: 3 Packing group: II
Proper shipping name: Toluene
Reportable Quantity (RQ): 1000 lbs
Poison Inhalation Hazard: No

IMDG
UN number: 1294 Class: 3 Packing group: II EMS-No: F-E, S-D
Proper shipping name: TOLUENE

IATA
UN number: 1294 Class: 3 Packing group: II
Proper shipping name: Toluene

SECTION 15: Regulatory information

SARA 302 Components
This material does not contain any components with a section 302 EHS TPQ.

SARA 313 Components
The following components are subject to reporting levels established by SARA Title III, Section 313:

	CAS-No.	Revision Date
Toluene	108-88-3	2007-07-01

SARA 311/312 Hazards
Fire Hazard, Acute Health Hazard, Chronic Health Hazard

Massachusetts Right To Know Components
No components are subject to the Massachusetts Right to Know Act.

SECTION 16: Other information

Further information

The above information is believed to be correct but does not purport to be all inclusive and shall be used only as a guide. The information in this document is based on the present state of our knowledge and is applicable to the product with regard to appropriate safety precautions. It does not represent any guarantee of the properties of the product. Sigma-Aldrich Corporation and its Affiliates shall not be held liable for any damage resulting from handling or from contact with the above product. See www.sigma-aldrich.com and/or the reverse side of invoice or packing slip for additional terms and conditions of sale.

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information in the document regarding the product remains unchanged and matches the product ordered. For further information please contact mlsbranding@sial.com.

Version: 6.10 Revision Date: 03/21/2020 Print Date: 05/29/2020

SAFETY DATA SHEET

Version 8.1
Revision Date 03/28/2020
Print Date 11/20/2020

SECTION 1: Identification of the substance/mixture and of the company/undertaking

1.1 Product identifiers

Product name : Xylenes

Product Number : 534056
Brand : SIGALD

1.2 Relevant identified uses of the substance or mixture and uses advised against

Identified uses : Laboratory chemicals, Synthesis of substances

1.3 Details of the supplier of the safety data sheet

Company : Sigma-Aldrich Inc.
3050 Spruce Street
ST. LOUIS MO 63103
UNITED STATES

Telephone : +1 314 771-5765
Fax : +1 800 325-5052

1.4 Emergency telephone number

Emergency Phone # : 800-424-9300 CHEMTREC (USA) +1-703-527-3887 CHEMTREC (International) 24 Hours/day; 7 Days/week

SECTION 2: Hazards identification

2.1 Classification of the substance or mixture

GHS Classification in accordance with 29 CFR 1910 (OSHA HCS)

Flammable liquids (Category 3), H226
Acute toxicity, Inhalation (Category 4), H332
Skin irritation (Category 2), H315
Eye irritation (Category 2A), H319
Carcinogenicity (Category 2), H351
Specific target organ toxicity - single exposure (Category 3), Respiratory system, H335
Specific target organ toxicity - repeated exposure (Category 2), H373
Specific target organ toxicity - repeated exposure, Inhalation (Category 2), Central nervous system, Liver, Kidney, H373
Aspiration hazard (Category 1), H304
Short-term (acute) aquatic hazard (Category 2), H401

For the full text of the H-Statements mentioned in this Section, see Section 16.

2.2 GHS Label elements, including precautionary statements

Pictogram	
Signal word	Danger

Hazard statement(s)

H226	Flammable liquid and vapour.
H304	May be fatal if swallowed and enters airways.
H315	Causes skin irritation.
H319	Causes serious eye irritation.
H332	Harmful if inhaled.
H335	May cause respiratory irritation.
H351	Suspected of causing cancer.
H373	May cause damage to organs through prolonged or repeated exposure.
H373	May cause damage to organs (Central nervous system, Liver, Kidney) through prolonged or repeated exposure if inhaled.
H401	Toxic to aquatic life.

Precautionary statement(s)

P201	Obtain special instructions before use.
P202	Do not handle until all safety precautions have been read and understood.
P210	Keep away from heat/sparks/open flames/hot surfaces. No smoking.
P233	Keep container tightly closed.
P240	Ground/bond container and receiving equipment.
P241	Use explosion-proof electrical/ ventilating/ lighting equipment.
P242	Use only non-sparking tools.
P243	Take precautionary measures against static discharge.
P260	Do not breathe dust/ fume/ gas/ mist/ vapours/ spray.
P264	Wash skin thoroughly after handling.
P271	Use only outdoors or in a well-ventilated area.
P273	Avoid release to the environment.
P280	Wear protective gloves/ protective clothing/ eye protection/ face protection.
P301 + P310	IF SWALLOWED: Immediately call a POISON CENTER/doctor.
P303 + P361 + P353	IF ON SKIN (or hair): Take off immediately all contaminated clothing. Rinse skin with water/shower.
P304 + P340 + P312	IF INHALED: Remove person to fresh air and keep comfortable for breathing. Call a POISON CENTER/doctor if you feel unwell.
P305 + P351 + P338	IF IN EYES: Rinse cautiously with water for several minutes. Remove contact lenses, if present and easy to do. Continue rinsing.
P308 + P313	IF exposed or concerned: Get medical advice/ attention.
P331	Do NOT induce vomiting.
P332 + P313	If skin irritation occurs: Get medical advice/ attention.
P337 + P313	If eye irritation persists: Get medical advice/ attention.
P362	Take off contaminated clothing and wash before reuse.
P370 + P378	In case of fire: Use dry sand, dry chemical or alcohol-resistant foam to extinguish.
P403 + P233	Store in a well-ventilated place. Keep container tightly closed.
P403 + P235	Store in a well-ventilated place. Keep cool.
P405	Store locked up.
P501	Dispose of contents/ container to an approved waste disposal plant.

2.3 Hazards not otherwise classified (HNOC) or not covered by GHS - none

SECTION 3: Composition/information on ingredients

3.1 Substances

Synonyms : Xylene mixture of isomers

Formula : C_8H_{10}

Molecular weight : 106.17 g/mol

Component	Classification	Concentration
Xylene		
	Flam. Liq. 2; Acute Tox. 4; Skin Irrit. 2; Eye Irrit. 2A; STOT SE 3; STOT RE 2; Asp. Tox. 1; Aquatic Acute 2; Aquatic Chronic 3; H225, H332, H312, H315, H319, H335, H373, H304, H401, H412	<= 100 %

Ethylbenzene		
	Flam. Liq. 2; Acute Tox. 4; Carc. 2; STOT RE 2; Asp. Tox. 1; Aquatic Acute 2; H225, H332, H351, H373, H304, H401	>= 20 - < 30 %

For the full text of the H-Statements mentioned in this Section, see Section 16.

SECTION 4: First aid measures

4.1 Description of first aid measures

General advice
Consult a physician. Show this safety data sheet to the doctor in attendance.Move out of dangerous area.

If inhaled
If breathed in, move person into fresh air. If not breathing, give artificial respiration. Consult a physician.

In case of skin contact
Wash off with soap and plenty of water. Consult a physician.

In case of eye contact
Rinse thoroughly with plenty of water for at least 15 minutes and consult a physician.

If swallowed
Do NOT induce vomiting. Never give anything by mouth to an unconscious person. Rinse mouth with water. Consult a physician.

4.2 Most important symptoms and effects, both acute and delayed
The most important known symptoms and effects are described in the labelling (see section 2.2) and/or in section 11

4.3 Indication of any immediate medical attention and special treatment needed
No data available

SECTION 5: Firefighting measures

5.1 Extinguishing media

Suitable extinguishing media
Use water spray, alcohol-resistant foam, dry chemical or carbon dioxide.

5.2 Special hazards arising from the substance or mixture
Carbon oxides

5.3 Advice for firefighters
Wear self-contained breathing apparatus for firefighting if necessary.

5.4 Further information
Use water spray to cool unopened containers.

SECTION 6: Accidental release measures

6.1 Personal precautions, protective equipment and emergency procedures
Use personal protective equipment. Avoid breathing vapours, mist or gas. Ensure adequate ventilation. Remove all sources of ignition. Evacuate personnel to safe areas. Beware of vapours accumulating to form explosive concentrations. Vapours can accumulate in low areas.
For personal protection see section 8.

6.2 Environmental precautions
Prevent further leakage or spillage if safe to do so. Do not let product enter drains. Discharge into the environment must be avoided.

6.3 Methods and materials for containment and cleaning up
Contain spillage, and then collect with an electrically protected vacuum cleaner or by wet-brushing and place in container for disposal according to local regulations (see section 13).

6.4 Reference to other sections
For disposal see section 13.

SECTION 7: Handling and storage

7.1 Precautions for safe handling
Avoid contact with skin and eyes. Avoid inhalation of vapour or mist.
Keep away from sources of ignition - No smoking.Take measures to prevent the build up of electrostatic charge.
For precautions see section 2.2.

7.2 Conditions for safe storage, including any incompatibilities
Keep container tightly closed in a dry and well-ventilated place. Containers which are opened must be carefully resealed and kept upright to prevent leakage.

Storage class (TRGS 510): 3: Flammable liquids

7.3 Specific end use(s)

Apart from the uses mentioned in section 1.2 no other specific uses are stipulated

SECTION 8: Exposure controls/personal protection

8.1 Control parameters

Components with workplace control parameters

Component	CAS-No.	Value	Control parameters	Basis
Xylene	1330-20-7	STEL	150 ppm 655 mg/m3	California permissible exposure limits for chemical contaminants (Title 8, Article 107)
		C	300 ppm	California permissible exposure limits for chemical contaminants (Title 8, Article 107)
		PEL	100 ppm 435 mg/m3	California permissible exposure limits for chemical contaminants (Title 8, Article 107)
		TWA	100 ppm 435 mg/m3	USA. Occupational Exposure Limits (OSHA) - Table Z-1 Limits for Air Contaminants
	Remarks	The value in mg/m3 is approximate.		
		TWA	100 ppm	USA. ACGIH Threshold Limit Values (TLV)
		Central Nervous System impairment Upper Respiratory Tract irritation Eye irritation Substances for which there is a Biological Exposure Index or Indices (see BEI® section) Not classifiable as a human carcinogen		
		STEL	150 ppm	USA. ACGIH Threshold Limit Values (TLV)
		Central Nervous System impairment Upper Respiratory Tract irritation Eye irritation Substances for which there is a Biological Exposure Index or Indices (see BEI® section) Not classifiable as a human carcinogen		
Ethylbenzene	100-41-4	TWA	100 ppm 435 mg/m3	USA. Occupational Exposure Limits (OSHA) - Table Z-1 Limits for Air Contaminants
		The value in mg/m3 is approximate.		

		PEL	5 ppm 22 mg/m3	California permissible exposure limits for chemical contaminants (Title 8, Article 107)
		STEL	30 ppm 130 mg/m3	California permissible exposure limits for chemical contaminants (Title 8, Article 107)

Biological occupational exposure limits

Component	CAS-No.	Parameters	Value	Biological specimen	Basis
Xylene	1330-20-7	Methylhippuric acids	1.5g/g creatinine	Urine	ACGIH - Biological Exposure Indices (BEI)
	Remarks	End of shift (As soon as possible after exposure ceases)			
Ethylbenzene	100-41-4	Sum of mandelic acid and phenyl glyoxylic acid	0.15g/g creatinine	Urine	ACGIH - Biological Exposure Indices (BEI)
		End of shift (As soon as possible after exposure ceases)			

8.2 Exposure controls

Appropriate engineering controls
Handle in accordance with good industrial hygiene and safety practice. Wash hands before breaks and at the end of workday.

Personal protective equipment

Eye/face protection
Face shield and safety glasses Use equipment for eye protection tested and approved under appropriate government standards such as NIOSH (US) or EN 166(EU).

Skin protection
Handle with gloves. Gloves must be inspected prior to use. Use proper glove removal technique (without touching glove's outer surface) to avoid skin contact with this product. Dispose of contaminated gloves after use in accordance with applicable laws and good laboratory practices. Wash and dry hands.

Full contact
Material: Fluorinated rubber
Minimum layer thickness: 0.7 mm
Break through time: 480 min
Material tested:Vitoject® (KCL 890 / Aldrich Z677698, Size M)

Splash contact
Material: Nitrile rubber
Minimum layer thickness: 0.4 mm
Break through time: 30 min
Material tested:Camatril® (KCL 730 / Aldrich Z677442, Size M)

data source: KCL GmbH, D-36124 Eichenzell, phone +49 (0)6659 87300, e-mail sales@kcl.de, test method: EN374

If used in solution, or mixed with other substances, and under conditions which differ from EN 374, contact the supplier of the CE approved gloves. This recommendation is advisory only and must be evaluated by an industrial hygienist and safety officer familiar with the specific situation of anticipated use by our customers. It should not be construed as offering an approval for any specific use scenario.

Body Protection
Complete suit protecting against chemicals, Flame retardant antistatic protective clothing., The type of protective equipment must be selected according to the concentration and amount of the dangerous substance at the specific workplace.

Respiratory protection
Where risk assessment shows air-purifying respirators are appropriate use a full-face respirator with multi-purpose combination (US) or type ABEK (EN 14387) respirator cartridges as a backup to engineering controls. If the respirator is the sole means of protection, use a full-face supplied air respirator. Use respirators and components tested and approved under appropriate government standards such as NIOSH (US) or CEN (EU).

Control of environmental exposure
Prevent further leakage or spillage if safe to do so. Do not let product enter drains. Discharge into the environment must be avoided.

SECTION 9: Physical and chemical properties

9.1 Information on basic physical and chemical properties

a)	Appearance	Form: clear, liquid Colour: colourless
b)	Odour	No data available
c)	Odour Threshold	No data available
d)	pH	No data available
e)	Melting point/freezing point	< 0 °C (< 32 °F)
f)	Initial boiling point and boiling range	136 - 140 °C 277 - 284 °F at 1,013 hPa
g)	Flash point	25 °C (77 °F) - closed cup
h)	Evaporation rate	No data available
i)	Flammability (solid, gas)	No data available
j)	Upper/lower flammability or explosive limits	Upper explosion limit: 7 %(V) Lower explosion limit: 1.1 %(V)
k)	Vapour pressure	24 hPa at 37.70 °C (99.86 °F)
l)	Vapour density	3.67 - (Air = 1.0)
m)	Relative density	0.865 g/cm3 at 20 °C (68 °F)
n)	Water solubility	No data available
o)	Partition coefficient: n-octanol/water	No data available

p)	Auto-ignition temperature	No data available
q)	Decomposition temperature	No data available
r)	Viscosity	No data available
s)	Explosive properties	No data available
t)	Oxidizing properties	No data available

9.2 Other safety information

Relative vapour density	3.67 - (Air = 1.0)

SECTION 10: Stability and reactivity

10.1 Reactivity
No data available

10.2 Chemical stability
Stable under recommended storage conditions.

10.3 Possibility of hazardous reactions
Vapours may form explosive mixture with air.

10.4 Conditions to avoid
Heat, flames and sparks.

10.5 Incompatible materials
Strong oxidizing agents

10.6 Hazardous decomposition products
Hazardous decomposition products formed under fire conditions. - Carbon oxides
Other decomposition products - No data available
In the event of fire: see section 5

SECTION 11: Toxicological information

11.1 Information on toxicological effects

Acute toxicity
LD50 Oral - Rat - male - 3,523 mg/kg
(EC Directive 92/69/EEC B.1 Acute Toxicity (Oral))
Remarks: (ECHA)
LC50 Inhalation - Rat - male - 4 h - 29 mg/l
(Regulation (EC) No. 440/2008, Annex, B.2)
Remarks: (Regulation (EC) No 1272/2008, Annex VI)
LD50 Dermal - Rabbit - male - 12,126 mg/kg
No data available
No data available

Skin corrosion/irritation
Skin - Rabbit
Result: Moderate skin irritation - 24 h
Remarks: (IUCLID)

Drying-out effect resulting in rough and chapped skin. After long-term exposure to the chemical: Dermatitis

Serious eye damage/eye irritation
Eyes - Rabbit
Result: Causes serious eye irritation. - 24 h
Remarks: (RTECS)

Respiratory or skin sensitisation
Local lymph node assay (LLNA) - Mouse
Result: negative
(OECD Test Guideline 429)

Germ cell mutagenicity
Mutagenicity (mammal cell test): chromosome aberration.
Chinese hamster ovary cells
Result: negative
(National Toxicology Program)
Ames test
Salmonella typhimurium
Result: negative
sister chromatid exchange assay
Chinese hamster ovary cells
Result: negative
OECD Test Guideline 478
Mouse - male and female
Result: negative

Carcinogenicity

IARC:	2B - Group 2B: Possibly carcinogenic to humans (Ethylbenzene)
NTP:	No component of this product present at levels greater than or equal to 0.1% is identified as a known or anticipated carcinogen by NTP.
OSHA:	No component of this product present at levels greater than or equal to 0.1% is on OSHA's list of regulated carcinogens.

Reproductive toxicity
No data available

Specific target organ toxicity - single exposure
No data available
Acute oral toxicity - Gastrointestinal disturbance
Acute inhalation toxicity - mucosal irritations, Cough, Shortness of breath, Possible damages:, damage of respiratory tract, Inhalation may lead to the formation of oedemas in the respiratory tract.

Specific target organ toxicity - repeated exposure
No data available

Aspiration hazard
May be fatal if swallowed and enters airways.

Additional Information

Repeated dose toxicity - Rat - male and female - Oral - 90 d - No observed adverse effect level - 150 mg/kg - Lowest observed adverse effect level - 150 mg/kg
RTECS: Not available

Blurred vision, Incoordination., Headache, Nausea, Vomiting, Dizziness, Weakness, anemia, Prolonged or repeated exposure to skin causes defatting and dermatitis.
To the best of our knowledge, the chemical, physical, and toxicological properties have not been thoroughly investigated.

After absorption:
Systemic effects:
Headache, somnolence, Dizziness, agitation, spasms, narcosis, inebriation
Effect potentiated by: ethanol
Other dangerous properties can not be excluded.
Handle in accordance with good industrial hygiene and safety practice.

Stomach - Irregularities - Based on Human Evidence
Stomach - Irregularities - Based on Human Evidence

SECTION 12: Ecological information

12.1 Toxicity

No data available

Toxicity to fish	static test LC50 - Oncorhynchus mykiss (rainbow trout) - 2.60 mg/l - 96 h (OECD Test Guideline 203)
Toxicity to algae	static test EC50 - Pseudokirchneriella subcapitata - 4.36 mg/l - 73 h (OECD Test Guideline 201)
Toxicity to bacteria	Remarks: (ECHA)(Xylene)

12.2 Persistence and degradability

No data available

12.3 Bioaccumulative potential

No data available

12.4 Mobility in soil

No data available

12.5 Results of PBT and vPvB assessment

PBT/vPvB assessment not available as chemical safety assessment not required/not conducted

12.6 Other adverse effects

An environmental hazard cannot be excluded in the event of unprofessional handling or disposal.
Toxic to aquatic life.
No data available

SECTION 13: Disposal considerations

13.1 Waste treatment methods

Product

Burn in a chemical incinerator equipped with an afterburner and scrubber but exert extra care in igniting as this material is highly flammable. Offer surplus and non-recyclable solutions to a licensed disposal company. Contact a licensed professional waste disposal service to dispose of this material.

Contaminated packaging
Dispose of as unused product.

SECTION 14: Transport information

DOT (US)
UN number: 1307 Class: 3 Packing group: III
Proper shipping name: Xylenes
Reportable Quantity (RQ): 100 lbs
Reportable Quantity (RQ): 100 lbs
Poison Inhalation Hazard: No

IMDG
UN number: 1307 Class: 3 Packing group: III EMS-No: F-E, S-D
Proper shipping name: XYLENES

IATA
UN number: 1307 Class: 3 Packing group: III
Proper shipping name: Xylenes

SECTION 15: Regulatory information

US TSCA Section 3
This chemical/product is not and cannot be distributed in commerce (as defined in TSCA section 3(5)) or processed (as defined in TSCA section 3(13)) for consumer paint or coating removal.

SARA 302 Components
No chemicals in this material are subject to the reporting requirements of SARA Title III, Section 302.

SARA 313 Components
The following components are subject to reporting levels established by SARA Title III, Section 313:

	CAS-No.	Revision Date
Ethylbenzene	100-41-4	2007-07-01
Xylene	1330-20-7	1993-04-24

SARA 311/312 Hazards
Fire Hazard, Acute Health Hazard, Chronic Health Hazard

Reportable Quantity : F003 lbs

Massachusetts Right To Know Components

	CAS-No.	Revision Date
Xylene	1330-20-7	1993-04-24
	100-41-4	2007-07-01

Ethylbenzene

Pennsylvania Right To Know Components

Xylene	CAS-No. 1330-20-7	Revision Date 1993-04-24
Ethylbenzene	100-41-4	2007-07-01

California Prop. 65 Components

, which is/are known to the State of California to cause cancer. For more information go to www.P65Warnings.ca.gov.Ethylbenzene	CAS-No. 100-41-4	Revision Date 2007-09-28

SECTION 16: Other information

Further information

The above information is believed to be correct but does not purport to be all inclusive and shall be used only as a guide. The information in this document is based on the present state of our knowledge and is applicable to the product with regard to appropriate safety precautions. It does not represent any guarantee of the properties of the product. Sigma-Aldrich Corporation and its Affiliates shall not be held liable for any damage resulting from handling or from contact with the above product. See www.sigma-aldrich.com and/or the reverse side of invoice or packing slip for additional terms and conditions of sale.

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Version: 8.1 Revision Date: 03/28/2020 Print Date: 11/20/2020

ATTACHMENT B

WEST NILE VIRUS/ST. LOUIS ENCEPHALITIS PREVENTION

WEST NILE VIRUS/ST. LOUIS ENCEPHALITIS PREVENTION

The following section is based upon information provided by the CDC Division of Vector-Borne Infectious Diseases. Symptoms of West Nile Virus include fever, headache, and body aches, occasionally with skin rash and swollen lymph glands, with most infections being mild. More severe infection may be marked by headache, high fever, neck stiffness, stupor, disorientation, coma, tremors, convulsions, muscle weakness, paralysis, and, rarely, death. Most infections of St. Louis encephalitis are mild without apparent symptoms other than fever with headache. More severe infection is marked by headache, high fever, neck stiffness, stupor, disorientation, coma, tremors, occasional convulsions (especially infants) and spastic (but rarely flaccid) paralysis. The only way to avoid infection of West Nile Virus and St. Louis encephalitis is to avoid mosquito bites. To reduce the chance of mosquito contact:

- Stay indoors at dawn, dusk, and in the early evening.
- Wear long-sleeved shirts and long pants whenever you are outdoors.
- Spray clothing with repellents containing permethrin or DEET (N, N-diethyl-meta-toluamide), since mosquitoes may bite through thin clothing.
- Apply insect repellent sparingly to exposed skin. An effective repellent will contain 35% DEET. DEET in high concentrations (greater than 35%) provides no additional protection.
- Repellents may irritate the eyes and mouth.
- Whenever you use an insecticide or insect repellent, be sure to read and follow the manufacturer's directions for use, as printed on the product.

ATTACHMENT C

REPORT FORMS

WEEKLY SAFETY REPORT FORM

Week Ending: _____ Project Name/Number: _____

Report Date: _____ Project Manager Name: _____

Summary of any violations of procedures occurring that week:

_____

_____

_____

Summary of any job related injuries, illnesses, or near misses that week:

_____

_____

_____

_____

Summary of air monitoring data that week (include and sample analyses, action levels exceeded, and actions taken):

_____

_____

_____

Comments:

_____

_____

_____

Name: _____ Company: _____

Signature: _____ Title: _____

INCIDENT REPORT FORM

Date of Report: _____

Injured: _____

Employer: _____

Site: _____ Site Location: _____

Report Prepared By: _____ _____
Signature Title

ACCIDENT/INCIDENT CATEGORY (check all that applies)

___Injury	___ Illness	___ Near Miss
__ Property Damage	___ Fire	___ Chemical Exposure
__ On-site Equipment	___ Motor Vehicle	___ Electrical
__ Mechanical	___ Spill	___ Other

DATE AND TIME OF ACCIDENT/INCIDENT: Narrative report of Accident/Incident: Identify: 1) actions leading to or contributing to the accident/incident; 2) the accident/incident occurrence; and 3) actions following the accident/incident.

_____

_____

_____

_____

_____

_____

_____

_____

_____

WITNESS TO ACCIDENT/INCIDENT:

Name:	_____	Company:	_____
Address:	_____	Address:	_____
Phone No.:	_____	Phone No.:	_____
Name:	_____	Company:	_____
Address:	_____	Address:	_____
Phone No.:	_____	Phone No.:	_____

INJURED - ILL:

Name: _____ SSN: _____

Address: _____ Age: _____

Length of Service: _____ Time on Present Job: _____

Time/Classification: _____

SEVERITY OF INJURY OR ILLNESS:

____ Disabling ___ Non-disabling ___ Fatality

____ Medical Treatment ___ First Aid Only

ESTIMATED NUMBER OF DAYS AWAY FROM JOB: _____

NATURE OF INJURY OR ILLNESS: _____

_____

_____

_____

CLASSIFICATION OF INJURY:

__ Abrasions _____ Dislocations ____ Punctures

__ Bites _____ Faint/Dizziness ____ Radiation Burns

__ Blisters _____ Fractures ____ Respiratory Allergy

__ Bruises _____ Frostbite ____ Sprains

__ Chemical Burns _____ Heat Burns ____ Toxic Resp. Exposure

__ Cold Exposure _____ Heat Exhaustion ____ Toxic Ingestion

__ Concussion _____ Heat Stroke ____ Dermal Allergy

__ Lacerations

Part of Body Affected: _____

Degree of Disability: _____

Date Medical Care was Received: _____

Where Medical Care was Received: _____

Address (if off-site): _____

(If two or more injuries, record on separate sheets)

PROPERTY DAMAGE:

Description of Damage: _____

_____

Cost of Damage: $ _____

ACCIDENT/INCIDENT LOCATION: _____

ACCIDENT/INCIDENT ANALYSIS: Causative agent most directly related to accident/incident (Object, substance, material, machinery, equipment, conditions)

_____

_____

_____

_____

Was weather a factor?:_____

_____

Unsafe mechanical/physical/environmental condition at time of accident/incident (Be specific):

_____

_____

_____

Personal factors (Attitude, knowledge or skill, reaction time, fatigue):

_____

_____

ON-SITE ACCIDENTS/INCIDENTS:

Level of personal protection equipment required in Site Safety Plan:

_____

_____

Modifications:

_____

Was injured using required equipment?:

_____

_____

If not, how did actual equipment use differ from plan?:

_____

_____

ACTION TAKEN TO PREVENT RECURRENCE: (Be specific. What has or will be done? When will it be done? Who is the responsible party to insure that the correction is made?

_____

_____

_____

_____

_____

ACCIDENT/INCIDENT REPORT REVIEWED BY:

_____ _____

SSO Name Printed SSO Signature

OTHERS PARTICIPATING IN INVESTIGATION:

_____ _____

Signature Title

_____ _____

Signature Title

_____ _____

Signature Title

ACCIDENT/INCIDENT FOLLOW-UP: Date: _____

Outcome of accident/incident: _____

_____

_____

_____

Physician's recommendations: _____

_____

_____

_____

Date injured returned to work: _____

Follow-up performed by:

_____ _____

Signature Title

ATTACH ANY ADDITIONAL INFORMATION TO THIS FORM

ATTACHMENT D
EMERGENCY HAND SIGNALS

EMERGENCY SIGNALS

In most cases, field personnel will carry portable radios for communication. If this is the case, a transmission that indicates an emergency will take priority over all other transmissions. All other site radios will yield the frequency to the emergency transmissions.

Where radio communications is not available, the following air-horn and/or hand signals will be used:

EMERGENCY HAND SIGNALS

OUT OF AIR, CAN'T BREATHE!

Hand gripping throat

LEAVE AREA IMMEDIATELY, NO DEBATE!

(No Picture) Grip partner's wrist or place both hands around waist

NEED ASSISTANCE!

Hands on top of head

OKAY! – I'M ALL RIGHT!

- I UNDERSTAND!

Thumbs up

NO! - NEGATIVE!

Thumbs down

ATTACHMENT E
COVID-19 CONSIDERATIONS

COVID-19 GENERAL PROCEDURES FOR SAR STAFF

(Revised April 22, 2020)

As part of our COVID-19 response measures, SAR has prepared this draft guidance document to supplement existing Health and Safety Plan (HASPs), New York City and State procedures, and OSHA and CDC guidelines with additional information and procedures to limit contamination and the potential spread of COVID-19.

This document is a working draft and will be updated and improved to remain relevant and viable as conditions change.

Section A - ON-SITE AND OFF-SITE PROCEDURES TO LIMIT CONTAMINATION AND POTENTIAL SPREAD OF COVID-19

CDC - COVID-19 Spread and Prevention Information

OSHA - Workplace Preparation Guidance

CDC - Guidance on Extended Use/Limited Reuse of Respiratory Protection

Let your PM know if there are any difficulties or issues with any of the following at sites

1) Maintain minimum 6-foot separation from others whenever possible (social distancing).

   The virus is thought to spread mainly from person-to-person, between people who are in close contact, through respiratory droplets produced when an infected person coughs or sneezes.

2) Wash your hands frequently with soap and water. Wash for at least 20 seconds and, if no soap is present, use a hand sanitizer that contains at least 60% alcohol.

3) Wear nitrile gloves whenever possible and be especially mindful of touching common surfaces.

4) Disinfect commonly touched surfaces frequently, and items frequently used in public immediately upon returning home

5) Face Coverings and Masks:

   a) On-site: Wear a cloth face covering or mask at all times when there is no issue with maintaining social distancing. N95 masks or respirators should be reserved for situations where social distancing on-site is difficult or impossible. Appropriate circumstances for donning an N95 mask or respirator on-site include, but are not necessarily limited to, going inside the Site trailer; and/or entering, exiting, or traversing the Site if proper social distancing cannot be achieved. This tiered approach will help maintain the supply of N95 masks so they are available for the highest risk scenarios.

b) Off-site During Work-related Commute: The CDC now recommends wearing cloth face coverings in public settings where other social distancing measures are difficult to maintain (https://www.cdc.gov/coronavirus/2019-ncov/prevent-getting-sick/cloth-face-cover.html). A mask or cloth face covering should worn during your commute to and from your field sites if you are unable to achieve proper social distancing. Appropriate times to wear a mask or cloth face covering include, but are not necessarily limited to, walking on crowded sidewalks, traveling in a shared vehicle (e.g. an AKRF vehicle or a Zipcar), and/or if you are required to enter an occupied indoor space to acquire supplies for your field site.

6) Wear safety glasses or goggles at all times while on-site and some form of eye covering (e.g., sunglasses, prescription and non-prescription glasses, or safety glasses) should be considered when commuting.

7) Avoid touching your face (eyes, nose, and mouth).

8) Cover your nose and mouth when coughing, sneezing, etc./ cough into elbow.

9) Do not spit.

10) Try to take your temperature regularly.

Certain job sites will have a station to have your temperature taken.

11) Talk to your PM/supervisor if you, your friends or family members that you live with or spend time with have displayed symptoms of COVID-19, tested positive, or are afflicted with even the common cold/flu.

12) Talk to your PM if anyone you know at your site tested positive for the COVID-19.

13) Follow any additional health & safety protocols required at your site or elsewhere.

Section B - OTHER FIELD PRACTICES

1) Maintain social distancing during field procedures, including:

a) Holding tailgate safety meetings;

b) Inside any construction trailers. Limit your time inside the trailer, if possible;

c) Interacting with other AKRF staff;

d) Interacting with drillers and other subcontractors;

e) Interacting with couriers/delivery services.

2) Review site access and conditions with your PM to assess health and safety precautions that must be taken before conducting work.

3) Talk to your PM if staffing by other contractors, consultants, or companies at your site is changed due to impacts caused by COVID-19.

4) Consider long-term safety and organization of equipment in the event of full lockdown of New York City:

   a) Take extra precautions to ensure that all of your materials, tools, equipment, etc. are properly stored and secured in the event of a long term shutdown.

Section C - SUPPLIES AND DOCUMENTATION

Check Google docs for latest supply inventory:
Link: NYC and WP inventory
*Link: Additional inventory at Tim McClintock's home***

1) Additional documentation to maintain during all field work:

   a) General Letter of Authorization for construction work from AKRF (google drive)

   b) Site-specific Letter of Authorization for construction work from AKRF (provided by PM)

   c) New York State Executive Order 202.7 (google drive)

   d) AKRF Protocols and Procedures related to COVID-19, dated March 17, 2020 (google drive)

2) Minimum supplies you should have with you:

   a) Standard PPE as required in HASP for your site --PLUS-

   b) N95 mask for on-site use (minimum 1)

   c) Hand sanitizer and/or disinfectant wipes (minimum 1)

   d) Soap and water in container (if working at a site with limited access to soap and running water)

   e) Safety glasses or googles (minimum 1)

   f) Mask or cloth face covering for off-site use during commute and to wear during working on job site when social distancing can be maintained (minimum 1)*

   * Cloth face coverings or masks can be purchased online or made at home. The CDC has provided a tutorial on how to make a cloth face covering at home with supplies you likely already have (https://www.cdc.gov/coronavirus/2019-ncov/prevent-getting-sick/diy-cloth-face-coverings.html). If you need to purchase supplies for making a cloth face covering, AKRF will reimburse you (up to $25). Consult with your supervisor before making any purchases.

3) Accessing offices:

Please limit your excursions to the office as part of limiting your moving and traveling in general

a) NYC (per Edythe):

i. Additional procedures for setting the alarm: The alarm will not be able to arm because of a default zone on the 9th floor.

ii. To bypass, press the alarm code number then the bypass button which is 6 then the 3 digit zone number (I think it is 060) which is shown on the keypad. The ready light will turn green. Then turn on system as normal.

b) WP: No modification to access procedures.

c) NJ: No modification to access procedures. Contact Jeff Entin for access cards.

d) Philadelphia: No modification to access procedures.

Section D - COMMUTING

The following commuting options are presented in preferred order.

For all vehicles, decontaminate all controls, steering wheel, etc. upon entering and wash hands/use sanitizer right after leaving car

Discuss cost to project with PM for all atypical options

1) Personal car (consider PPE and decontamination prior to driving)

2) SAR field vehicle (wear face covering and nitrile gloves

3) Zipcar (wear face covering and nitrile gloves)

4) Public transportation (wear face covering and nitrile gloves)

Section E - REMINDERS ABOUT SCHEDULING, HOURS, BILLABILITY, ETC.

1) Conduct all site-follow-up work at home, unless otherwise directed.

2) Talk to your PM/supervisor if you are scheduled to work longer than 40 hours in one week, or longer than 12 hours in one day.

3) Talk to your PM/supervisor if you might have difficulty accruing 40 hours of work in a week.

4) Talk to your PM about procedures regarding COVID-19 at your site, including any documentation or official information that was shared with you, any ongoing discussions about general practices, or any changes in the way consultants, contractors, or clients' staff or operate at the site.

Use of Cloth Face Coverings to Help Slow the Spread of COVID-19

How to Wear Cloth Face Coverings

Cloth face coverings should—

- fit snugly but comfortably against the side of the face
- be secured with ties or ear loops
- include multiple layers of fabric
- allow for breathing without restriction
- be able to be laundered and machine dried without damage or change to shape

CDC on Homemade Cloth Face Coverings

CDC recommends wearing cloth face coverings in public settings where other social distancing measures are difficult to maintain (e.g., grocery stores and pharmacies), **especially** in areas of significant community-based transmission.

CDC also advises the use of simple cloth face coverings to slow the spread of the virus and help people who may have the virus and do not know it from transmitting it to others. Cloth face coverings fashioned from household items or made at home from common materials at low cost can be used as an additional, voluntary public health measure.

Cloth face coverings should not be placed on young children under age 2, anyone who has trouble breathing, or is unconscious, incapacitated or otherwise unable to remove the cloth face covering without assistance.

The cloth face coverings recommended are not surgical masks or N-95 respirators. Those are critical supplies that must continue to be reserved for healthcare workers and other medical first responders, as recommended by current CDC guidance.

Should cloth face coverings be washed or otherwise cleaned regularly? How regularly?

Yes. They should be routinely washed depending on the frequency of use.

How does one safely sterilize/clean a cloth face covering?

A washing machine should suffice in properly washing a cloth face covering.

How does one safely remove a used cloth face covering?

Individuals should be careful not to touch their eyes, nose, and mouth when removing their cloth face covering and wash hands immediately after removing.

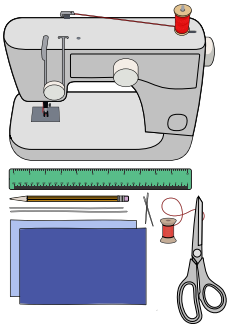
cdc.gov/coronavirus

CS316353B 04/04/2020, 12:22 PM

Sewn Cloth Face Covering

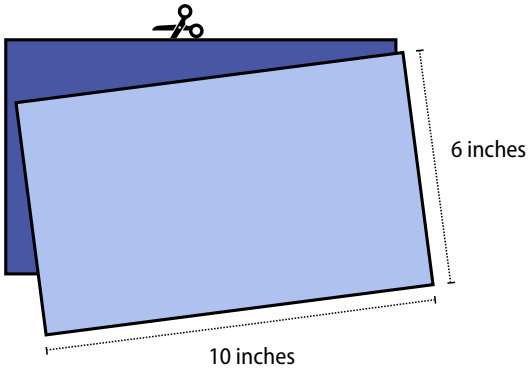
Materials

- Two 10”x6” rectangles of cotton fabric
- Two 6” pieces of elastic (or rubber bands, string, cloth strips, or hair ties)
- Needle and thread (or bobby pin)
- Scissors
- Sewing machine

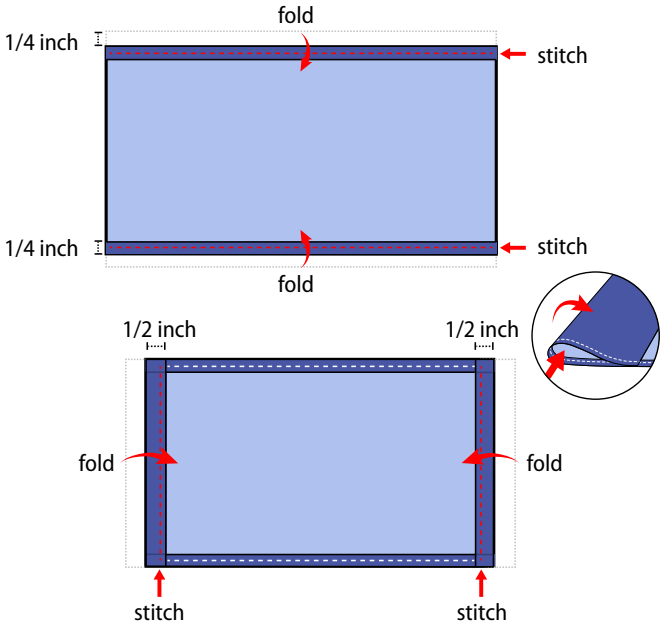


Tutorial

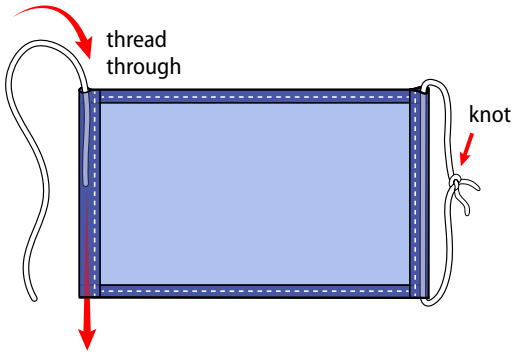
1. Cut out two 10-by-6-inch rectangles of cotton fabric. Use tightly woven cotton, such as quilting fabric or cotton sheets. T-shirt fabric will work in a pinch. Stack the two rectangles; you will sew the cloth face covering as if it was a single piece of fabric.



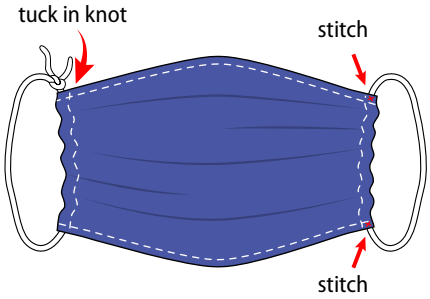
2. Fold over the long sides ¼ inch and hem. Then fold the double layer of fabric over ½ inch along the short sides and stitch down.



3. Run a 6-inch length of 1/8-inch wide elastic through the wider hem on each side of the cloth face covering. These will be the ear loops. Use a large needle or a bobby pin to thread it through. Tie the ends tight.
Don’t have elastic? Use hair ties or elastic head bands. If you only have string, you can make the ties longer and tie the cloth face covering behind your head.



4. Gently pull on the elastic so that the knots are tucked inside the hem.
Gather the sides of the cloth face covering on the elastic and adjust so the cloth face covering fits your face. Then securely stitch the elastic in place to keep it from slipping.



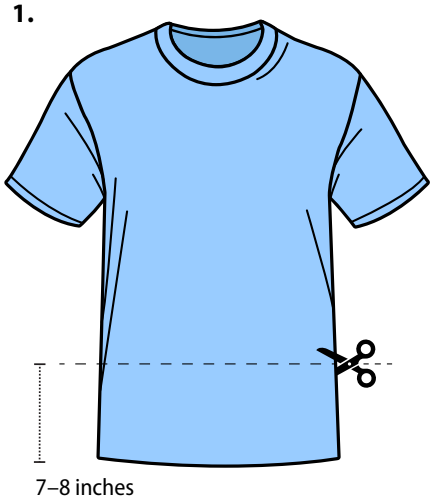
Quick Cut T-shirt Cloth Face Covering (no sew method)

Materials

- T-shirt
- Scissors

Tutorial

1.



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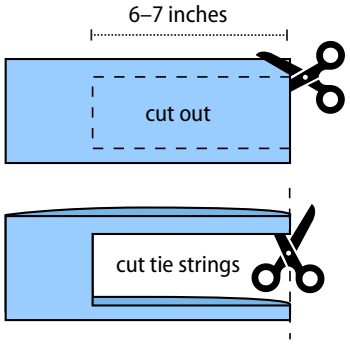

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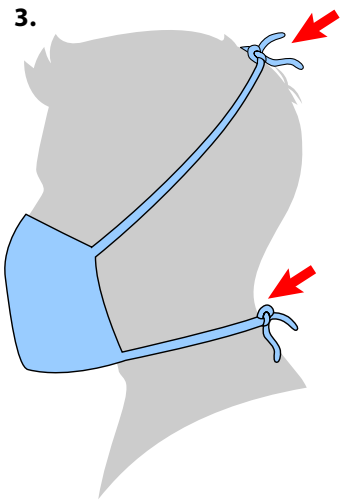


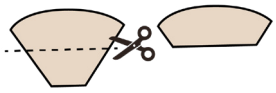
Bandana Cloth Face Covering (no sew method)

Materials

- Bandana (or square cotton cloth approximately 20”x20”)
- Coffee filter
- Rubber bands (or hair ties)
- Scissors (if you are cutting your own cloth)

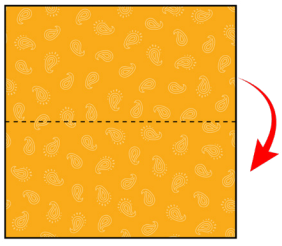
Tutorial

1.



cut coffee filter

2.



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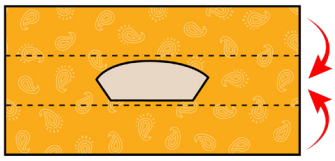


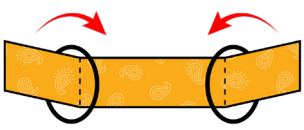
Fold filter in center of folded bandana.

Fold top down. Fold bottom up.

4.

Place rubber bands or hair ties about 6 inches apart.

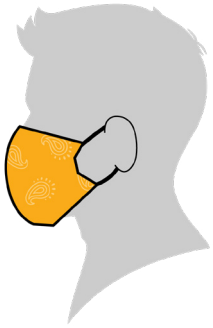
5.



Fold side to the middle and tuck.

6.

7.



APPENDIX B

QUALITY ASSURANCE PROJECT PLAN

243 SHERIDAN STREET

NEW CASSEL, NEW YORK

SECTION 11, BLOCK 44, LOT 74

Quality Assurance Project Plan

EPA Petroleum No. BF-9649919
AKRF Project Number: 200225

Prepared for:
United States Environmental Protection, Region 2
290 Broadway, 25th Floor
New York, NY 10007

On Behalf Of:
The Town of North Hempstead
210 Plandome Road
Manhasset, NY 11030

Prepared by:

AKRF, Inc.
440 Park Avenue South, 7th Floor
New York, NY 10016

JUNE 2023

TABLE OF CONTENTS

FIGURES

APPENDICES

Brownfields QAPP Template #1 - TITLE AND APPROVAL PAGE

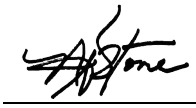
Title: Phase II Environmental Site Investigation (ESI) - 243 Sheridan Street Quality Assurance Project Plan (QAPP)
Project Name/Property Name: Phase II Environmental Site Investigation / 243 Sheridan Street
Property/Site Location: 243 Sheridan Street, New Cassel, New York 11590
Revision Number: 4
Date: June 27, 2023
Brownfields Cooperative Agreement Number: BF-9649919

The Town of North Hempstead
Brownfields Recipient

Deborah Shapiro, QEP, Sr. Vice President
AKRF, Inc.
440 Park Avenue South, 7th Floor, New York, NY 10016
646-388-9544, dshapiro@akrf.com
Preparer's Name and Organizational Affiliation
Preparer's Address, Telephone Number, and E-mail Address

June 27, 2023
Preparation Date (Day/Month/Year)

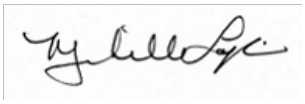
Brownfields Recipient Program Manager:



Signature

Neal Stone / Town of North Hempstead
516-869-7809, stonen@northhempsteadny.gov
Printed Name/Organization/Date

Environmental Consultant Quality Assurance Officer (QAO)



Signature

Michelle Lapin, P.E. / AKRF
Printed Name/Organization/Date

EPA Region 2 Quality Assurance Officer (QAO)

ADLY MICHAEL Digitally signed by ADLY MICHAEL Date: 2023.07.28 15:27:45 -04'00'

Signature

Adly Michael / USEPA Region 2
Printed Name/Organization/Date

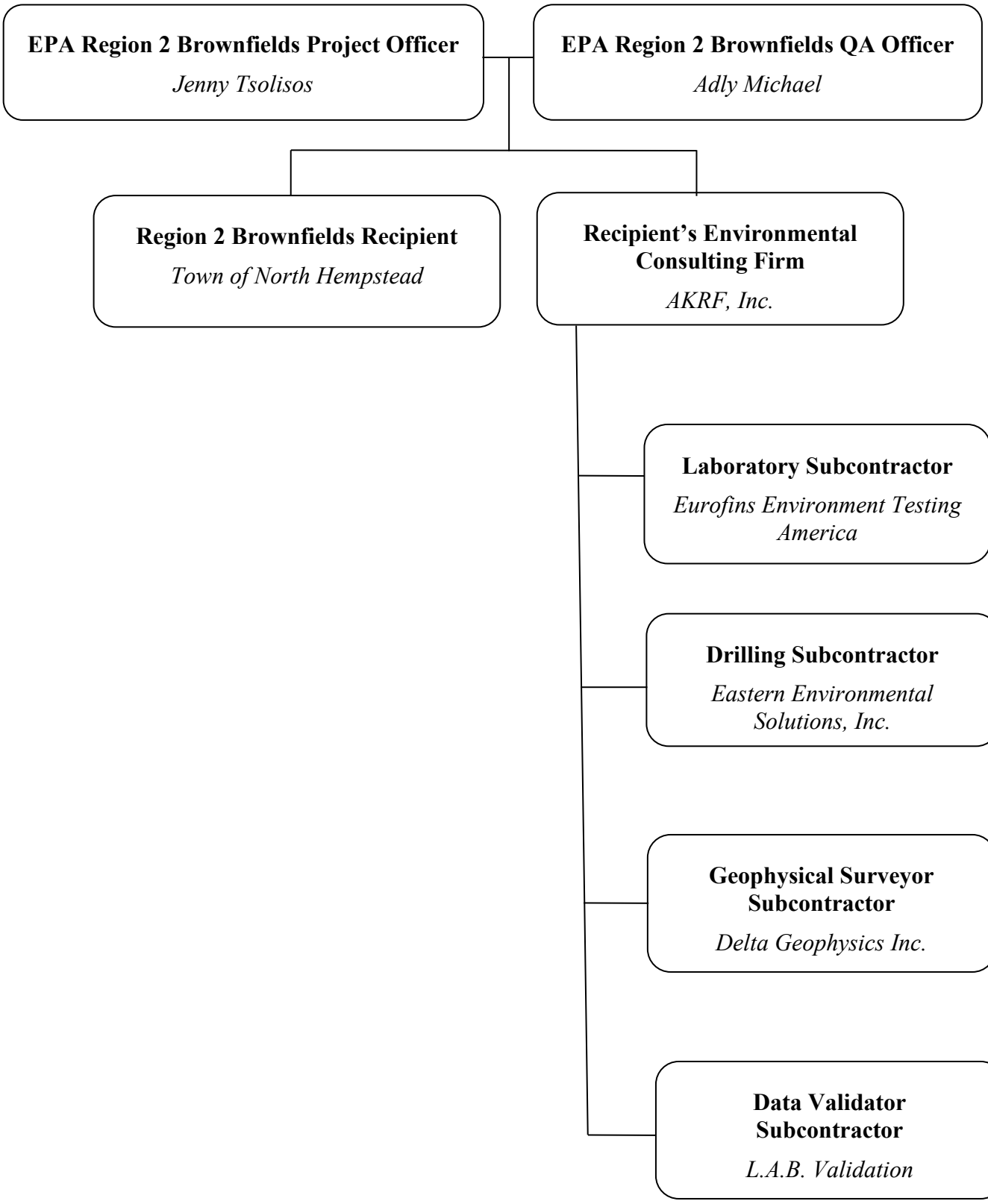
EPA Region 2 Brownfields Project Officer:

TSOLISOS.JENNY.1228755849 Digitally signed by TSOLISOS.JENNY.1228755849 Date: 2023.08.03 10:45:44 -04'00'

Signature

Jenny Tsolisos / USEPA Region 2
Printed Name/Organization/Date

1.0 BROWNFIELDS QAPP TEMPLATE #2A - PROJECT ORGANIZATION CHART



2.0 BROWNFIELDS QAPP TEMPLATE #2B - PERSONNEL RESPONSIBILITIES

Name	Title	Organization	Telephone Number	Responsibilities
Jenny Tsolisos	EPA Brownfields Project Officer (BPO)	EPA Region 2	212-637-4349	Assures overall programmatic compliance
Adly Michael	EPA Brownfields Quality Assurance Officer (QAO)	EPA Region 2	732-906-6161	Assures analytical and QA program compliance
Neal Stone, AICP MCIP	Brownfields Recipient Program Manager	Town of North Hempstead	516-869-7809	Overall program oversight
Deborah Shapiro, QEP	Project Director	AKRF, Inc.	646-388-9544	General oversight of all aspects of the project. Communicate regularly with all members of the project team to ensure a smooth flow of information between involved parties.
Tim McClintock	Project Manager	AKRF, Inc.	914-922-2374	Coordinate the implementation of all aspects of the technical scope. Interpretation of laboratory results and preparation of report.
Michelle Lapin, P.E.	Project QA/QC Officer	AKRF, Inc.	646-388-9520	Review sampling procedures with all personnel prior to commencing any fieldwork and conduct periodic site visits to assess implementation of the procedures. Review analytical data reports and associated QA/QC sampling data.
Carl Armbruster	Laboratory Quality Assurance Manager	Eurofins Environment Testing America of Edison, NJ	732-593-2519	Responsible for quality control procedures and checks in the laboratory and ensuring adherence to laboratory protocols for Eurofins Environment Testing America of Edison, NJ laboratory. Conduct a final check on the analytical calculations and sign off on the laboratory reports.
Michael Bates	Field Team Leader	AKRF, Inc.	914-355-0693	Oversee field implementation of the Work Plan and assist with preparation of the report.
Lori Beyer	Third-Party Data Validator	L.A.B. Validation	516-523-7891	Review analytical data reports and associated QA/QC sampling data, and prepare a Data Usability Summary Report (DUSR).

3.0 BROWNFIELDS QAPP TEMPLATE #3A - PROBLEM DEFINITION/PROJECT DESCRIPTION

3.1 Site Location and Description

The Site is located at 243 Sheridan Street in the Hamlet of New Cassel, NY, and is defined on the Nassau County Tax Map as Section 11, Block 44, Lot 74. The approximately 6,000-square foot (sf) Site contains a one-story, single-family residence with a cellar, a detached garage, and exterior paved and landscaped areas. The Site is bounded to the north, south, and west by single-family residences, and to the east by Sheridan Street, followed by single-family residences.

The Site location is shown on Figure 1 and the Site boundaries are shown on Figure 2.

3.2 Site History

AKRF completed a Phase I Environmental Site Assessment (ESA) of the Site in conformance with American Society for Testing and Materials (ASTM) Standard E1527-13, *Standard Practice for Environmental Site Assessments: Phase I Environmental Site Assessment Practice* in September 2022. At the time of the Phase I ESA, the Site contained a vacant, one-story, single-family residence with a cellar, a detached garage, and exterior paved and landscaped areas.

Historical documentation indicated that 243 Sheridan Street was developed with a one-story residence with a cellar and a detached garage by 1929 and an addition was constructed in the southwestern portion of the residence prior to 1941; no evidence of historical agricultural, commercial, industrial, or manufacturing operations identified at the Site. The footprints of the Site buildings remained generally consistent with the footprints observed during AKRF's Phase I ESA reconnaissance since at least 1941. Suspected fill and vent piping potentially associated with a current or former petroleum storage tank were observed protruding from the foundation wall in the northwestern portion of the residence, suggesting potential former use/storage of petroleum.

It should be noted that suspect ACM, LBP/LCP, PCB-containing material, and/or mercury-containing material may be present in fill material, buried structures, and/or buried demolition debris, which will be addressed prior to redevelopment of the Site. These hazardous contaminants are not being addressed under Petroleum Grant No. BF-9649919 and are therefore not included as part of the ESI.

3.3 Problem Definition

The environmental question being asked is: Are there contaminants in the soil and/or groundwater that exceed New York State Department of Environmental Conservation (NYSDEC) criteria and may impact human health and/or the environment?

3.4 Project Decision Statements

To assess the contaminant pathway for potential contaminants of concern, it is necessary to submit soil and groundwater samples for analysis for the full list of compounds included on the Target Compound List (TCL) and in New York State Department of Environmental Protection (NYSDEC) Part 375, Table 375-6.8(a) for volatile organic compounds (VOCs), semivolatile organic compounds (SVOCs), metals, polychlorinated biphenyls (PCBs),

pesticides, and herbicides. The primary concern would be to identify any potential impacts to the environment that may affect human health. Analytical results will be evaluated against the NYSDEC criteria listed in Part 375 for soil, and Part 703 or Division of Water Technical and Operational Guidance Series (TOGS) 1.1.1 for groundwater. Based on these assessments, it may be necessary to develop a mitigating plan for any contaminated media.

4.0 BROWNFIELDS QAPP TEMPLATE #3B - PROJECT QUALITY OBJECTIVES

The overall project objective for the ESI includes:

Determine if soil and/or groundwater at the Site poses a risk to human health, safety, and the environment by exposure to hazardous substances.

In order to meet the objective, soil samples and any groundwater samples will be analyzed for regulated hazardous substances including TCL/Part 375 VOCs, SVOCs, metals, PCBs, pesticides, and herbicides.

Who will use the data?

The collected data will be used by the Town of North Hempstead.

What types of data are needed?

Soil and groundwater samples will be collected and analyzed at an off-site laboratory for TCL/Part 375 VOCs, SVOCs, metals, PCBs, pesticides, and herbicides.

How "good" do the data need to be in order to support the environmental decision?

Data requirements and usability will be evaluated as presented in Section 24.

How much data are needed?

At least six soil samples (one from each soil boring) will be collected and analyzed for TCL/Part 375 VOCs, SVOCs, metals, PCBs, pesticides, and herbicides. One groundwater sample will be collected from each temporary groundwater well. A maximum of three groundwater samples will be collected. See Figure 2 for proposed soil sample locations. Groundwater sample locations will be determined based on field observations.

Where, when, and how should the data be collected/generated?

Continuous soil samples will be recovered from the soil borings. One sample will be submitted for laboratory analysis from the 2-foot interval exhibiting the greatest field evidence of contamination or, in the absence of contamination, from the top 2-foot interval below the surface cover (asphalt, concrete, grass/roots, etc.).

Who will collect and generate the data?

AKRF and its subcontractors will collect and generate the data. Delta Geophysics Inc. (Delta) will be responsible for collected geophysical data. Eastern Environmental Solutions, Inc. (Eastern) will be responsible for advancing the soil borings. All samples will be analyzed by Eurofins Environment Testing America of Edison, NJ, a New York State Department of Health (NYSDOH)-certified laboratory. AKRF will be responsible for collecting, reviewing, assessing, and disseminating validated data. L.A.B. Validation, a third-party data validator, will review the data and prepare a Data Usability Summary Report (DUSR). If three groundwater wells are required to be installed, a groundwater well survey will be performed by Fehringer Surveying, P.C.

How will the data be reported?

The data will be reported in a Phase II ESI Report.

How will the data be archived?

All data will be electronic, maintained for the entirety of the project, and eventually archived on the AKRF corporate server for storage in perpetuity.

5.0 BROWNFIELDS QAPP TEMPLATE #4 - PROJECT SCHEDULE/TIMELINE

Activities	Organization	Dates (MM/DD/YY)		Deliverable	Deliverable Due Date
		Anticipated Date(s) of Initiation	Anticipated Date of Completion		
Preparation of QAPP	AKRF, Inc.	11/01/22	11/21/22	Draft QAPP	11/28/22
Revision of QAPP	AKRF, Inc.	06/20/23	06/23/23	Revised Draft QAPP	06/27/23
Review of QAPP	EPA Region 2	11/28/22	07/07/23	Approved QAPP	NA
Preparation of Health and Safety Plan	AKRF, Inc.	11/01/22	11/21/22	Approved HASP	12/30/22
Field Reconnaissance/ Access	AKRF, Inc.	07/24/23	07/24/23	N/A	N/A
Procurement of Equipment	AKRF, Inc.	07/31/23	07/31/23	N/A	NA
Laboratory Request	AKRF, Inc.	08/01/23	08/01/23	N/A	NA
Collection of Field Samples	AKRF, Inc. & Eastern Environmental Solutions, Inc.	08/07/23	08/07/23	N/A	N/A
Laboratory Package Received	AKRF, Inc.	08/23/23	08/23/23	Unvalidated data package	NA
Validation of Laboratory Results	L.A.B. Validation	08/28/23	09/01/23	Validated data Packages	NA
Data Evaluation/ Preparation of Final Report	AKRF, Inc.	08/28/23	09/12/23	Final Report	NA

6.0 BROWNFIELDS QAPP TEMPLATE #5A - SAMPLING METHODS AND LOCATIONS

Samples will be collected from soil borings across the Site to provide information on general conditions. The following table summarizes the sampling locations, analytical groups, number of samples, section reference for sampling standard operating procedure (SOP) and rationale for sampling locations.

Matrix	Sampling Location(s)	Analytical Group	No. of Samples	Sampling SOP Reference	Rationale for Sampling Location
Soil	SB-01	TCL/Part 375 VOCs, SVOCs, metals, PCBs, pesticides, and herbicides	1	Section 25.1	To access soil quality in the southeastern portion of the Site, and to assess subsurface conditions adjacent to the suspected cesspool (if located during the geophysical survey)
Soil	SB-02	TCL/Part 375 VOCs, SVOCs, metals, PCBs, pesticides, and herbicides	1	Section 25.1	To access soil quality in the vicinity of the mound of potential fill material, and to assess subsurface conditions adjacent to the suspected cesspool (if located during the geophysical survey)
Soil	SB-03	TCL/Part 375 VOCs, SVOCs, metals, PCBs, pesticides, and herbicides	1	Section 25.1	To access soil quality in the vicinity of the garage, and to assess subsurface conditions adjacent to the suspected cesspool (if located during the geophysical survey)
Soil	SB-04	TCL/Part 375 VOCs, SVOCs, metals, PCBs, pesticides, and herbicides	1	Section 25.1	To access soil quality in the vicinity of the suspected petroleum storage tank fill and vent pipes, and to assess subsurface conditions adjacent to the suspected cesspool (if located during the geophysical survey)
Soil	SB-05	TCL/Part 375 VOCs, SVOCs, metals, PCBs, pesticides, and herbicides	1	Section 25.1	To assess soil quality in the vicinity of the suspected petroleum storage tank or where evidence of a current/former petroleum storage tank are identified
Soil	SB-06	TCL/Part 375 VOCs, SVOCs, metals, PCBs, pesticides, and herbicides	1	Section 25.1	To assess soil quality in the vicinity of the suspected petroleum storage tank or where evidence of a current/former petroleum storage tank are identified
Groundwater	TBD	TCL/Part 375 VOCs, SVOCs, metals, PCBs, pesticides, and herbicides	Up to 3	Section 25.3	To assess groundwater quality at locations where soil contamination extends near or to the groundwater interface

Notes:

(1) One blind duplicate will be collected from soil boring SB-02 and one matrix spike/matrix spike duplicate (MS/MSD) will be collected from soil boring SB-03. The duplicate, MS/MSD, and field blank samples will be collected and analyzed for TCL/Part 375 VOCs, SVOCs, PCBs, metals, pesticides, and herbicides. A trip blank will be submitted for analysis of TCL/Part 375 VOCs with any collected groundwater samples.

(2) The sampling interval for the soil samples submitted for laboratory analysis will be based on field observations. If field evidence of contamination is observed, the 2-foot sampling interval will include the interval displaying the greatest contamination. If contamination is not observed, the sample will be collected from the 2-foot interval below the surface cover (asphalt, concrete, grass/roots, etc.).

6.1 Geophysical Investigation, Utility Clearance, Soil Boring Advancement, and Temporary Groundwater Monitoring Well Installation

Six soil borings are proposed at the locations shown on Figure 2. To clear the proposed boring locations for subsurface utilities and to markout the location(s) of potential cesspools, underground storage tanks, etc., a geophysical survey will be conducted across accessible portions of the Site prior to any intrusive work. The geophysical survey will include both electromagnetic (EM) and ground penetrating radar (GPR) methods. Any anomalies and utlities will be marked in the field using spray paint.

Utility mark-outs are required by law and the drilling contractor is required to call Dig Safely New York at least three days prior to intrusive work. If there are any questions regarding locations of utilities in the sidewalk, the respective utility(s) will be contacted to clarify any concerns and/or the sampling location would be adjusted following consultation with USEPA.

Soil samples collected from borings advanced using the DPP will be collected in 3- or 5-foot long, 2-inch diameter, stainless steel macrocore piston rod samplers fitted with internal, dedicated acetate liners. Any soil sample collected via hand tools will be collected with a decontaminated stainless steel hand auger. All sampling equipment (e.g., drilling rods/casing, macrocore samplers, and probe rods) will be either dedicated or decontaminated between sampling locations. Soil samples will be collected continuously from surface grade to boring termination depths and soil will be inspected for evidence of contamination (e.g., odors, staining, product, etc.), screened for the presence of volatile organics with a photoionization detector (PID) equipped with a calibrated 10.6 electron volt (eV) lamp, and logged using the modified Burmister soil classification system. Soil samples will be examined by AKRF personnel, who will complete a soil boring log for each location. Each soil boring location will be surveyed using a Global Positioning System (GPS) handheld device to determine their accurate location.

Based on United States Geological Survey (USGS) mapping, groundwater beneath the Site is expected to be encountered between 45 to 55 feet below grade. If contamination is identified in any of the soil borings within 10 feet of the groundwater interface, a temporary groundwater well will be installed within the boring. A maximum of three wells will be installed. The temporary wells will be constructed with 2-inch diameter polyvinyl chloride (PVC) casing with 10-foot long, 0.020-inch slotted well screen straddling the water table. A sand pack will be installed around the well annulus from the bottom of the well to approximately 5 feet above the screened interval. Following installation, each well will be developed to remove any accumulated fines and establish a hydraulic connection with the surrounding aquifer (Section 25.2). If three or more wells are installed, the wells will be surveyed by a New York State licensed surveyor to determine their accurate location and elevation (Section 25.4).

6.2 Soil Sampling

Soil is being sampled as part of the ESI to: (1) determine the nature and extent of potentially impacted media and (2) determine if potentially impacted soil may require special handling requirements relating to construction worker health and safety and disposal. Standard operating procedures for soil sampling are outlined in Section 25.1.

The soil samples will be analyzed for the full list of TCL/Part 375 compounds for the following parameters:

- VOCs by USEPA Method 8260
- SVOCs by USEPA Method 8270
- Metals by USEPA Method 6000/7000 series
- PCBs by USEPA Method 8082
- Pesticides by USEPA Method 8081
- Herbicides by USEPA Method 8151

The sampling interval for the soil samples submitted for laboratory analysis will be one sample from the 2-foot interval displaying the greatest evidence of field contamination. If contamination is not observed, the sample will be collected from the 2-foot interval immediately below the surface cover (asphalt, concrete, grass/roots, etc.). All samples will be analyzed by a NYSDOH-certified laboratory with Category B deliverables.

For every 20 samples per media, one field (aqueous equipment rinsate) blank, one blind duplicate, and one matrix spike/matrix spike duplicate will be collected for quality control/quality assurance (QA/QC) purposes for analysis of all of the above parameters.

6.3 Groundwater Sampling

Groundwater is being sampled as part of this ESI to: (1) determine the nature and extent of potentially impacted media; (2) determine if potentially impacted groundwater requires special handling requirements relating to construction worker health and safety and discharge; and (3) determine the potential for a groundwater to soil vapor to indoor air pathway. Standard operating procedures for groundwater sampling are outlined in Section 25.3.

One groundwater sample will be collected from each well in accordance with USEPA low flow sampling techniques. The expected targeted purge rate will be around 500 milliliters per minute (mL/min) and water quality parameters will be monitored during purging.

The groundwater samples will be analyzed for the full list of TCL/Part 375 compounds for the following parameters:

- VOCs by USEPA Method 8260
- SVOCs by USEPA Method 8270
- Metals by USEPA Method 6000/7000 series
- PCBs by USEPA Method 8082
- Pesticides by USEPA Method 8081
- Herbicides by USEPA Method 8151

If the groundwater formation cannot produce enough sample volume for analysis, then a bailer will be used to collect the sample. One field blank, one blind duplicate, and one matrix spike/matrix spike duplicate will be collected for QA/QC purposes for analysis of all of the parameters. One trip blank will also be submitted for analysis of TCL/Part 375 VOCs only. All samples will be analyzed by a NYSDOH-certified laboratory with Category B deliverables.

7.0 BROWNFIELDS QAPP TEMPLATE #5B - ANALYTICAL METHODS AND REQUIREMENTS

Eurofins Environment Testing America will perform analyses on soil and groundwater.

Laboratory Location	Matrix	Analytical Group	Conc. Level	Analytical and Preparation Method	Min. Sample Volume	Containers	Preservation Requirement	Max. Hold Time
Edison, NJ	Soil	VOCs	Low	8260D / 5035A *	5 g	3, 5-g encores or terracore	Cool to 4°C +/- 2°C DI & MeOH (terracores)	2 days to lab prep / 14 days
Edison, NJ	Soil	SVOCs	Low	8270E / 3546	50 g	1, 4- or 8- oz Clear Glass	Cool to 4°C +/- 2°C	14 days to extract/40 days from extraction to analysis
Edison, NJ	Soil	PCBs	Low	8082A / 3546	100 g	1, 4- or 8- oz Clear Glass	Cool to 4°C +/- 2°C	14 days to extract/40 days from extraction to analysis
Edison, NJ	Soil	Metals	Low	6020B and 7471B / 3050B and 7471B	10 g	1, 4- or 8- oz Clear Glass	Cool to 4°C +/- 2°C	28 days Hg/180 days all other metals
Edison, NJ	Soil	Pesticides	Low	8081B/3546	100g	1, 4- or 8- oz Clear Glass	Cool to 4°C +/- 2°C	14 days to extract/40 days from extraction to analysis
Edison, NJ	Soil	Herbicides	Low	8151A/3546	100g	1, 4- or 8- oz Clear Glass	Cool to 4°C +/- 2°C	14 days to extract/40 days from extraction to analysis
Edison, NJ	Soil	Hexavalent Chromium	Low	7196A / 3060A	2.5 g	1, 4- or 8- oz Clear Glass	Cool to 4°C +/- 2°C	30 days
Edison, NJ	Groundwater	VOCs	Low	8260D / 5030C	40 mL	3, 40-mL Vials	HCl, pH < 2; Cool to 4°C +/- 2°C	14 days
Edison, NJ	Groundwater	SVOCs	Low	8270E / 3510C	250 mL	2, 250-mL Amber Glass	Cool to 4°C +/- 2°C	7 days to extract/40 days from extraction to analysis
Edison, NJ	Groundwater	PCBs	Low	8082A / 3510C	250 mL	2, 250-mL Amber Glass	Cool to 4°C +/- 2°C	7 days to extract/40 days from extraction to analysis
Edison, NJ	Groundwater	Metals	Low	6020B and 7470A / 3005A and 7470A	250 mL	1, 250-mL Plastic	HNO3, pH < 2, Cool to 4°C +/- 2°C	28 days Hg/180 days all other metals
Edison, NJ	Groundwater	Hexavalent Chromium	Low	7196A / no prep method	250 mL	1, 250-mL Plastic	Cool to 4°C +/- 2°C	24 hours
Edison, NJ	Groundwater	Pesticides	Low	8081B/3510C	250 mL	2, 250-mL Amber Glass	Cool to 4°C +/- 2°C	7 days to extract/40 days from extraction to analysis
Edison, NJ	Groundwater	Herbicides	Low	8151A/8151A	250 mL	2, 250-mL Amber Glass	Cool to 4°C +/- 2°C	7 days to extract/40 days from extraction to analysis
Edison, NJ	Groundwater	SVOC SIM MS w/ Isotope Dilution	Low	8270E SIM/3510C	250 mL	2, 250-mL Amber Glass	Cool to 4°C +/- 2°C	7 days to extract/40 days from extraction to analysis

Notes:

* Method 5035A is an appropriate preparation method as it addresses each of the various collection/preservation options associated with the use of encores and terracores. When encores are used in the field, the samples are not preserved until they are received by the laboratory. Once at the laboratory, the samples are transferred from the encores into 40mL vials (terracores) consisting of two DI water vials (for low-level analysis) and one MeOH vial (for medium-level analysis) per sample. Alternatively, if terracores are used in the field, the sample is immediately placed into two DI water vials (for low-level analysis) and one MeOH vial (for medium-level analysis). Under both scenarios, the vials are frozen until they are extracted for screening and analysis; the initial sample screening will determine whether the low-level or medium-level analysis is required.

8.0 BROWNFIELDS QAPP TEMPLATE #5C - REFERENCE LIMITS AND EVALUATION TABLES

8.1 Soil Sampling

Matrix: Soil
Analytical Group: TCL/Part 375 VOCs
Concentration Level: Low
Analytical Method: 8260D

Analyte	CAS Number	State Regulatory Standard * mg/kg		Laboratory	
		Part 375 Groundwater Protection	Part 375 Restricted Residential	Method Detection Limit (MDL) mg/kg	Reporting Limit mg/kg
1,1,1-Trichloroethane	71-55-6	0.68	100	0.000233	0.00100
1,1,2,2-Tetrachloroethane	79-34-5	NA	NA	0.000214	0.00100
1,1,2-Trichloro-1,2,2-trifluoroethane	76-13-1	NA	NA	0.000301	0.00100
1,1,2-Trichloroethane	79-00-5	NA	NA	0.000178	0.00100
1,1-Dichloroethane	75-34-3	0.27	26	0.000206	0.00100
1,1-Dichloroethene	75-35-4	0.33	100	0.000225	0.00100
1,2,3-Trichlorobenzene	87-61-6	NA	NA	0.000181	0.00100
1,2,4-Trichlorobenzene	120-82-1	NA	NA	0.000358	0.00100
1,2,4-Trimethylbenzene	95-63-6	3.6	52	0.000246	0.00100
1,2-Dibromo-3-Chloropropane	96-12-8	NA	NA	0.000460	0.00100
1,2-Dichlorobenzene	95-50-1	1.1	100	0.000361	0.00100
1,2-Dichloroethane	107-06-2	0.02	3.1	0.000296	0.00100
1,2-Dichloropropane	78-87-5	NA	NA	0.000423	0.00100
1,3,5-Trimethylbenzene	108-67-8	8.4	52	0.000314	0.00100
1,3-Dichlorobenzene	541-73-1	2.4	4.9	0.000365	0.00100
1,4-Dichlorobenzene	106-46-7	1.8	13	0.000225	0.00100
2-Butanone (MEK)	78-93-3	0.12	100	0.000368	0.00500
2-Hexanone	591-78-6	NA	NA	0.00171	0.00500
4-Methyl-2-pentanone (MIBK)	108-10-1	NA	NA	0.00156	0.00500
Acetone	67-64-1	0.05	100	0.00572	0.00600
Benzene	71-43-2	0.06	4.8	0.000258	0.00100
Bromoform	75-25-2	NA	NA	0.000425	0.00100
Bromomethane	74-83-9	NA	NA	0.00100	0.00200
Carbon disulfide	75-15-0	NA	NA	0.000266	0.00100
Carbon tetrachloride	56-23-5	0.76	2.4	0.000387	0.00100
Chlorobenzene	108-90-7	1.1	100	0.000177	0.00100

Analyte	CAS Number	State Regulatory Standard * mg/kg		Laboratory	
		Part 375 Groundwater Protection	Part 375 Restricted Residential	Method Detection Limit (MDL) mg/kg	Reporting Limit mg/kg
Chlorobromomethane	74-97-5	NA	NA	0.000281	0.00100
Chlorodibromomethane	124-48-1	NA	NA	0.000194	0.00100
Chloroethane	75-00-3	NA	NA	0.000522	0.00100
Chloroform	67-66-3	0.37	49	0.000971	0.00100
Chloromethane	74-87-3	NA	NA	0.000435	0.00100
cis-1,2-Dichloroethene	156-59-2	0.25	100	0.000358	0.00100
cis-1,3-Dichloropropene	10061-01-5	NA	NA	0.000273	0.00100
Cyclohexane	110-82-7	NA	NA	0.000221	0.00100
Dichlorobromomethane	75-27-4	NA	NA	0.000257	0.00100
Dichlorodifluoromethane	75-71-8	NA	NA	0.000338	0.00100
Ethylbenzene	100-41-4	1	41	0.000199	0.00100
Ethylene Dibromide	106-93-4	NA	NA	0.000180	0.00100
Isopropylbenzene	98-82-8	NA	NA	0.000285	0.00100
Methyl acetate	79-20-9	NA	NA	0.00430	0.00500
Methyl tert-butyl ether	1634-04-4	0.93	100	0.000512	0.00100
Methylcyclohexane	108-87-2	NA	NA	0.000499	0.00100
Methylene Chloride	75-09-2	0.05	100	0.00115	0.00200
m-Xylene & p-Xylene	179601-23-1	NA	NA	0.000174	0.00100
n-Butylbenzene	104-51-8	12	100	0.000294	0.00100
N-Propylbenzene	103-65-1	3.9	100	0.000175	0.00100
o-Xylene	95-47-6	NA	NA	0.000194	0.00100
sec-Butylbenzene	135-98-8	11	100	0.000288	0.00100
Styrene	100-42-5	NA	NA	0.000278	0.00100
tert-Butylbenzene	98-06-6	5.9	100	0.000276	0.00100
Tetrachloroethene	127-18-4	1.3	19	0.000305	0.00100
Toluene	108-88-3	0.7	100	0.000234	0.00100
trans-1,2-Dichloroethene	156-60-5	0.19	100	0.000246	0.00100
trans-1,3-Dichloropropene	10061-02-6	NA	NA	0.000266	0.00100
Trichloroethene	79-01-6	0.47	21	0.000321	0.00100
Trichlorofluoromethane	75-69-4	NA	NA	0.000406	0.00100
Vinyl chloride	75-01-4	0.02	0.9	0.000546	0.00100
Xylenes, Total	1330-20-7	1.6 (TS)	100 (TS)	0.000174	0.00200

Notes:

* State Regulatory Standard - NYSDEC Part 375 Soil Cleanup Objectives (SCOs) for Restricted Residential and for Protection of Groundwater

NA – A standard has not been established.

TS – Total Standard

Matrix: Soil
Analytical Group: TCL/Part 375 SVOCs
Concentration Level: Low
Analytical Method: 8270E

Analyte	CAS Number	State Regulatory Standard * mg/kg		Laboratory	
		Part 375 Groundwater Protection	Part 375 Restricted Residential	Method Detection Limit (MDL) mg/kg	Reporting Limit mg/kg
1,1'-Biphenyl	92-52-4	NA	NA	0.0115	0.330
1,2,4,5-Tetrachlorobenzene	95-94-3	NA	NA	0.0103	0.330
1,4-Dioxane	123-91-1	0.1	13	0.0289	0.0330
2,2'-oxybis[1-chloropropane]	108-60-1	NA	NA	0.0198	0.330
2,3,4,6-Tetrachlorophenol	58-90-2	NA	NA	0.0224	0.330
2,4,5-Trichlorophenol	95-95-4	NA	NA	0.0337	0.330
2,4,6-Trichlorophenol	88-06-2	NA	NA	0.0425	0.133
2,4-Dichlorophenol	120-83-2	NA	NA	0.0212	0.133
2,4-Dimethylphenol	105-67-9	NA	NA	0.0395	0.330
2,4-Dinitrophenol	51-28-5	NA	NA	0.163	0.266
2,4-Dinitrotoluene	121-14-2	NA	NA	0.0356	0.0670
2,6-Dinitrotoluene	606-20-2	NA	NA	0.0239	0.0670
2-Chloronaphthalene	91-58-7	NA	NA	0.0153	0.330
2-Chlorophenol	95-57-8	NA	NA	0.0118	0.330
2-Methylnaphthalene	91-57-6	NA	NA	0.00925	0.330
2-Methylphenol	95-48-7	0.33	100	0.0124	0.330
2-Nitroaniline	88-74-4	NA	NA	0.0252	0.330
2-Nitrophenol	88-75-5	NA	NA	0.0331	0.330
3 & 4 Methylphenol	15831-10-4	NA	NA	0.0206	0.330
3,3'-Dichlorobenzidine	91-94-1	NA	NA	0.0500	0.133
3-Nitroaniline	99-09-2	NA	NA	0.0785	0.330
4,6-Dinitro-2-methylphenol	534-52-1	NA	NA	0.135	0.266
4-Bromophenyl phenyl ether	101-55-3	NA	NA	0.0131	0.330
4-Chloro-3-methylphenol	59-50-7	NA	NA	0.0186	0.330
4-Chloroaniline	106-47-8	NA	NA	0.0587	0.330
4-Chlorophenyl phenyl ether	7005-72-3	NA	NA	0.0117	0.330
4-Methylphenol	106-44-5	0.33	100	0.0207	0.330
4-Nitroaniline	100-01-6	NA	NA	0.0380	0.330
4-Nitrophenol	100-02-7	NA	NA	0.0539	0.670
Acenaphthene	83-32-9	98	100	0.00943	0.330
Acenaphthylene	208-96-8	107	100	0.00946	0.330

Analyte	CAS Number	State Regulatory Standard * mg/kg		Laboratory	
		Part 375 Groundwater Protection	Part 375 Restricted Residential	Method Detection Limit (MDL) mg/kg	Reporting Limit mg/kg
Acetophenone	98-86-2	NA	NA	0.0162	0.330
Anthracene	120-12-7	1,000	100	0.0101	0.330
Atrazine	1912-24-9	NA	NA	0.0195	0.133
Benzaldehyde	100-52-7	NA	NA	0.0547	0.330
Benzo[a]anthracene	56-55-3	1	1	0.0249	0.0330
Benzo[a]pyrene	50-32-8	22	1	0.00881	0.0330
Benzo[b]fluoranthene	205-99-2	1.7	1	0.00856	0.0330
Benzo[g,h,i]perylene	191-24-2	1,000	100	0.00976	0.330
Benzo[k]fluoranthene	207-08-9	1.7	3.9	0.00649	0.0330
Bis(2-chloroethoxy)methane	111-91-1	NA	NA	0.0258	0.330
Bis(2-chloroethyl)ether	111-44-4	NA	NA	0.0115	0.0330
Bis(2-ethylhexyl) phthalate	117-81-7	NA	NA	0.0175	0.330
Butyl benzyl phthalate	85-68-7	NA	NA	0.0155	0.330
Caprolactam	105-60-2	NA	NA	0.0515	0.330
Carbazole	86-74-8	NA	NA	0.0126	0.330
Chrysene	218-01-9	1	3.9	0.0139	0.330
Dibenz(a,h)anthracene	53-70-3	1,000	0.33	0.0143	0.0330
Dibenzofuran	132-64-9	210	59	0.0110	0.330
Diethyl phthalate	84-66-2	NA	NA	0.0107	0.330
Dimethyl phthalate	131-11-3	NA	NA	0.0752	0.330
Di-n-butyl phthalate	84-74-2	NA	NA	0.0125	0.330
Di-n-octyl phthalate	117-84-0	NA	NA	0.0175	0.330
Fluoranthene	206-44-0	1,000	100	0.0116	0.330
Fluorene	86-73-7	386	100	0.00968	0.330
Hexachlorobenzene	118-74-1	3.2	1.2	0.0157	0.0330
Hexachlorobutadiene	87-68-3	NA	NA	0.00704	0.0670
Hexachlorocyclopentadiene	77-47-4	NA	NA	0.0290	0.330
Hexachloroethane	67-72-1	NA	NA	0.0114	0.0330
Indeno[1,2,3-cd]pyrene	193-39-5	8.2	0.5	0.0129	0.0330
Isophorone	78-59-1	NA	NA	0.0956	0.133
Naphthalene	91-20-3	12	100	0.00572	0.330
Nitrobenzene	98-95-3	NA	NA	0.0183	0.0330
N-Nitrosodi-n-propylamine	621-64-7	NA	NA	0.0240	0.0330
N-Nitrosodiphenylamine	86-30-6	NA	NA	0.0272	0.330
Pentachlorophenol	87-86-5	0.8	6.7	0.0678	0.266

Analyte	CAS Number	State Regulatory Standard * mg/kg		Laboratory	
		Part 375 Groundwater Protection	Part 375 Restricted Residential	Method Detection Limit (MDL) mg/kg	Reporting Limit mg/kg
Phenanthrene	85-01-8	1,000	100	0.0135	0.330
Phenol	108-95-2	0.33	100	0.0122	0.330
Pyrene	129-00-0	1,000	100	0.00823	0.330

Note:
* State Regulatory Standard - NYSDEC Part 375 Soil Cleanup Objectives (SCOs) for Restricted Residential and for Protection of Groundwater

Matrix: Soil
Analytical Group: TCL/Part 375 Metals
Concentration Level: Low
Analytical Method: 6020B / 7471B / 7196A

Analyte	CAS Number	State Regulatory Standard * mg/kg		Laboratory	
		Part 375 Groundwater Protection	Part 375 Restricted Residential	Method Detection Limit (MDL) mg/kg	Reporting Limit mg/kg
Aluminum	7429-90-5	NS	NS	5.49	20.0
Antimony	7429-90-5	NS	NS	0.146	1.00
Arsenic	7440-38-2	16	16	0.103	1.00
Barium	7440-39-3	820	400	0.145	2.00
Beryllium	7440-41-7	47	72	0.0570	0.400
Cadmium	7440-43-9	7.5	4.3	0.113	1.00
Calcium	7440-70-2	NS	NS	40.7	100
Chromium, Total	7440-47-3	NA	NA	0.908	2.00
Chromium, Hexavalent	18540-29-9	19	110	0.848	2.00
Cobalt	7440-48-4	NS	NS	0.148	2.00
Copper	7440-50-8	1,720	270	0.368	2.00
Iron	7439-89-6	NS	NS	20.2	60.0
Lead	7439-92-1	450	400	0.200	0.600
Magnesium	7439-95-4	NS	NS	10.2	100
Manganese	7439-96-5	2,000	2,000	0.403	4.00
Mercury, Total	7439-97-6	0.73	0.81	0.008	0.017
Nickel	7440-02-0	130	310	0.470	2.00

Analyte	CAS Number	State Regulatory Standard * mg/kg		Laboratory	
		Part 375 Groundwater Protection	Part 375 Restricted Residential	Method Detection Limit (MDL) mg/kg	Reporting Limit mg/kg
Potassium	7440-09-7	NS	NS	16.2	100
Selenium	7782-49-2	4	180	0.128	1.25
Silver	7440-22-4	8.3	180	0.0890	0.400
Sodium	7440-23-5	NS	NS	45.7	100
Thallium	7440-28-0	NS	NS	0.0410	0.400
Vanadium	7440-62-2	NS	NS	0.206	2.00
Zinc	7440-66-6	2,480	10,000	3.05	8.00

Notes:
* State Regulatory Standard - NYSDEC Part 375 Soil Cleanup Objectives (SCOs) for Restricted Residential and for Protection of Groundwater
NA – A standard has not been established.

Matrix: Soil
Analytical Group: PCBs
Concentration Level: Low
Analytical Method: 8082A

Analyte	CAS Number	State Regulatory Standard * mg/kg		Laboratory	
		Part 375 Groundwater Protection	Part 375 Restricted Residential	Method Detection Limit (MDL) mg/kg	Reporting Limit mg/kg
Polychlorinated Biphenyls, Total	1336-36-3	3.2	1	0.0178	0.0670

Note:
* State Regulatory Standard - NYSDEC Part 375 Soil Cleanup Objectives (SCOs) for Restricted Residential and for Protection of Groundwater

Matrix: Soil
Analytical Group: TCL/Part 375 Pesticides
Concentration Level: Low
Analytical Method: 8081B

Analyte	CAS Number	State Regulatory Standard * mg/kg		Laboratory	
		Part 375 Groundwater Protection	Part 375 Restricted Residential	Method Detection Limit (MDL) mg/kg	Reporting Limit mg/kg
4,4'-DDD	72-54-8	14	13	0.00114	0.00670
4,4'-DDE	72-55-9	17	8.9	0.000790	0.00670
4,4'-DDT	50-29-3	136	7.9	0.00123	0.00670
Aldrin	309-00-2	0.19	0.097	0.00101	0.00670
alpha-BHC	319-84-6	0.02	0.48	0.000680	0.00200
beta-BHC	319-85-7	0.09	0.36	0.000750	0.00200
Chlordane (technical)	12789-03-6	NA	NA	0.0162	0.0670
cis-Chlordane	5103-71-9	2.9	4.2	0.00106	0.00670
delta-BHC	319-86-8	0.25	100	0.000410	0.00200
Dieldrin	60-57-1	0.1	0.2	0.000870	0.00200
Endosulfan I	959-98-8	102	24	0.00102	0.00670
Endosulfan II	33213-65-9	102	24	0.00172	0.00670
Endosulfan sulfate	1031-07-8	1,000	24	0.000840	0.00670
Endrin	72-20-8	0.06	11	0.000960	0.00670
Endrin aldehyde	7421-93-4	NA	NA	0.00158	0.00670
Endrin ketone	53494-70-5	NA	NA	0.00130	0.00670
gamma-BHC (Lindane)	58-89-9	0.1	1.3	0.000620	0.00200
Heptachlor	76-44-8	0.38	2.1	0.000790	0.00670
Heptachlor epoxide	1024-57-3	NA	NA	0.00100	0.00670
Methoxychlor	72-43-5	NA	NA	0.00153	0.00670
Toxaphene	8001-35-2	NA	NA	0.0242	0.0670
trans-Chlordane	5103-74-2	NA	NA	0.00118	0.00670

Note:

* State Regulatory Standard - NYSDEC Part 375 Soil Cleanup Objectives (SCOs) for Restricted Residential and for Protection of Groundwater

NA – A standard has not been established.

Matrix: Soil
Analytical Group: TCL/Part 375 Herbicides
Concentration Level: Low
Analytical Method: 8051A

Analyte	CAS Number	State Regulatory Standard * mg/kg		Laboratory	
		Part 375 Groundwater Protection	Part 375 Restricted Residential	Method Detection Limit (MDL) mg/kg	Reporting Limit mg/kg
2,4,5-T	93-76-5	NA	NA	0.00708	0.0333
2,4-D	94-75-7	NA	NA	0.0121	0.0333
2,4,5-TP (Silvex)	93-72-1	3,800	100,000	0.00347	0.0333

Note:
* State Regulatory Standard - NYSDEC Part 375 Soil Cleanup Objectives (SCOs) for Restricted Residential and for Protection of Groundwater
NA – A standard has not been established.

8.2 Groundwater Sampling

Matrix: Aqueous
Analytical Group: TCL/Part 375 VOCs
Concentration Level: Low
Analytical Method: 8260D

Analyte	CAS Number	State Regulatory Standard * µg/L	Laboratory **	
			Method Detection Limit (MDL) µg/L	Reporting Limit ug/L
1,1,1-Trichloroethane	71-55-6	5	0.238	1.00
1,1,2,2-Tetrachloroethane	79-34-5	5	0.367	1.00
1,1,2-Trichloro-1,2,2-trifluoroethane	76-13-1	5	0.311	1.00
1,1,2-Trichloroethane	79-00-5	1	0.204	1.00
1,1-Dichloroethane	75-34-3	5	0.264	1.00
1,1-Dichloroethene	75-35-4	5	0.264	1.00
1,2,3-Trichlorobenzene	87-61-6	5	0.357	1.00
1,2,4-Trichlorobenzene	120-82-1	5	0.365	1.00
1,2,4-Trimethylbenzene	95-63-6	5	0.374	1.00
1,2-Dibromo-3-Chloropropane	96-12-8	0.04	0.376	1.00
1,2-Dichlorobenzene	95-50-1	3	0.212	1.00
1,2-Dichloroethane	107-06-2	0.6	0.430	1.00
1,2-Dichloropropane	78-87-5	1	0.353	1.00
1,3,5-Trimethylbenzene	108-67-8	5	0.326	1.00

Analyte	CAS Number	State Regulatory Standard * µg/L	Laboratory **	
			Method Detection Limit (MDL) µg/L	Reporting Limit ug/L
1,3-Dichlorobenzene	541-73-1	3	0.342	1.00
1,4-Dichlorobenzene	106-46-7	3	0.334	1.00
2-Butanone (MEK)	78-93-3	50	1.85	5.00
2-Hexanone	591-78-6	50	1.14	5.00
4-Methyl-2-pentanone (MIBK)	108-10-1	NA	1.30	5.00
Acetone	67-64-1	50	4.42	5.00
Benzene	71-43-2	1	0.203	1.00
Bromoform	75-25-2	50	0.536	1.00
Bromomethane	74-83-9	5	0.550	1.00
Carbon disulfide	75-15-0	60	0.821	1.00
Carbon tetrachloride	56-23-5	5	0.208	1.00
Chlorobenzene	108-90-7	5	0.377	1.00
Chlorobromomethane	74-97-5	5	0.412	1.00
Chlorodibromomethane	124-48-1	50	0.281	1.00
Chloroethane	75-00-3	5	0.320	1.00
Chloroform	67-66-3	7	0.326	1.00
Chloromethane	74-87-3	5	0.402	1.00
cis-1,2-Dichloroethene	156-59-2	5	0.219	1.00
cis-1,3-Dichloropropene	10061-01-5	0.4 (TS)	0.222	1.00
Cyclohexane	110-82-7	NA	0.321	1.00
Dichlorobromomethane	75-27-4	50	0.343	1.00
Dichlorodifluoromethane	75-71-8	5	0.311	1.00
Ethylbenzene	100-41-4	5	0.298	1.00
Ethylene Dibromide	106-93-4	0.0006	0.498	1.00
Isopropylbenzene	98-82-8	5	0.336	1.00
Methyl acetate	79-20-9	NA	0.785	5.00
Methyl tert-butyl ether	1634-04-4	10	0.216	1.00
Methylcyclohexane	108-87-2	NA	0.707	1.00
Methylene Chloride	75-09-2	5	0.315	1.00
m-Xylene & p-Xylene	179601-23-1	5	0.296	1.00
n-Butylbenzene	104-51-8	5	0.324	1.00
N-Propylbenzene	103-65-1	5	0.322	1.00
o-Xylene	95-47-6	5	0.361	1.00
sec-Butylbenzene	135-98-8	5	0.367	1.00
Styrene	100-42-5	5	0.415	1.00
tert-Butylbenzene	98-06-6	5	0.335	1.00
Tetrachloroethene	127-18-4	5	0.249	1.00

Analyte	CAS Number	State Regulatory Standard * µg/L	Laboratory **	
			Method Detection Limit (MDL) µg/L	Reporting Limit ug/L
Toluene	108-88-3	5	0.379	1.00
trans-1,2-Dichloroethene	156-60-5	5	0.235	1.00
trans-1,3-Dichloropropene	10061-02-6	NA	0.223	1.00
Trichloroethene	79-01-6	5	0.314	1.00
Trichlorofluoromethane	75-69-4	5	0.320	1.00
Vinyl chloride	75-01-4	2	0.171	1.00
Xylenes, Total	1330-20-7	NA	0.654	2.00

Notes:

* State Regulatory Standard – NYSDEC Class GA Ambient Water Quality Standards and Guidance Values listed in TOGS 1.1.1 and Part 703.5: "Water Quality Standards Surface Water and Groundwater"

** Some of the target analytes have a lower State Regulatory Standard than the corresponding Method Detection Limit and/or Reporting Limit. The specified Method Detection Limit and Reporting Limit are the lowest concentrations that the laboratory can achieve given the constraints of the analytical method and associated equipment.

NA – A standard has not been established.

TS – The standard is the sum total.

Matrix: Aqueous
Analytical Group: TCL/Part 375 SVOCs
Concentration Level: Low
Analytical Method: 8270E

Analyte	CAS Number	State Regulatory Standard * ug/L	Laboratory **	
			Method Detection Limit (MDL) ug/L	Reporting Limit ug/L
1,1'-Biphenyl	92-52-4	5	1.19	10.0
1,2,4,5-Tetrachlorobenzene	95-94-3	5	1.24	10.0
2,2'-oxybis[1-chloropropane]	108-60-1	NA	0.629	10.0
2,3,4,6-Tetrachlorophenol	58-90-2	NA	0.746	10.0
2,4,5-Trichlorophenol	95-95-4	NA	0.880	10.0
2,4,6-Trichlorophenol	88-06-2	NA	0.857	10.0
2,4-Dichlorophenol	120-83-2	5	1.07	10.0
2,4-Dimethylphenol	105-67-9	50	0.619	10.0
2,4-Dinitrophenol	51-28-5	510	2.63	40.0
2,4-Dinitrotoluene	121-14-2	5	0.997	10.0
2,6-Dinitrotoluene	606-20-2	5	0.826	2.00
2-Chloronaphthalene	91-58-7	10	1.18	10.0
2-Chlorophenol	95-57-8	NA	0.377	10.0

Analyte	CAS Number	State Regulatory Standard * ug/L	Laboratory **	
			Method Detection Limit (MDL) ug/L	Reporting Limit ug/L
2-Methylnaphthalene	91-57-6	NA	0.527	10.0
2-Methylphenol	95-48-7	NA	0.671	10.0
2-Nitroaniline	88-74-4	5	0.474	10.0
2-Nitrophenol	88-75-5	NA	0.747	10.0
3 & 4 Methylphenol	15831-10-4	NA	0.644	10.0
3,3'-Dichlorobenzidine	91-94-1	5	1.43	10.0
3-Nitroaniline	99-09-2	5	1.94	10.0
4,6-Dinitro-2-methylphenol	534-52-1	NA	2.99	20.0
4-Bromophenyl phenyl ether	101-55-3	NA	0.745	10.0
4-Chloro-3-methylphenol	59-50-7	NA	0.575	10.0
4-Chloroaniline	106-47-8	5	1.88	10.0
4-Chlorophenyl phenyl ether	7005-72-3	NA	1.28	10.0
4-Methylphenol	106-44-5	NA	0.651	10.0
4-Nitroaniline	100-01-6	5	1.22	10.0
4-Nitrophenol	100-02-7	NA	3.98	20.0
Acenaphthene	83-32-9	20	1.08	10.0
Acenaphthylene	208-96-8	NA	0.823	10.0
Acetophenone	98-86-2	NA	2.33	10.0
Anthracene	120-12-7	50	1.30	10.0
Atrazine	1912-24-9	7.5	1.35	2.00
Benzaldehyde	100-52-7	NA	2.10	10.0
Benzo[a]anthracene	56-55-3	0.002	0.592	1.00
Benzo[a]pyrene	50-32-8	ND	0.405	1.00
Benzo[b]fluoranthene	205-99-2	0.002	0.676	2.00
Benzo[g,h,i]perylene	191-24-2	NA	0.702	10.0
Benzo[k]fluoranthene	207-08-9	0.002	0.674	1.00
Bis(2-chloroethoxy)methane	111-91-1	5	0.589	10.0
Bis(2-chloroethyl)ether	111-44-4	1	0.633	1.00
Bis(2-ethylhexyl) phthalate	117-81-7	5	0.804	2.00
Butyl benzyl phthalate	85-68-7	50	0.854	10.0
Caprolactam	105-60-2	NA	2.24	10.0
Carbazole	86-74-8	NA	0.679	10.0
Chrysene	218-01-9	0.002	0.907	2.00
Dibenz(a,h)anthracene	53-70-3	NA	0.720	1.00
Dibenzofuran	132-64-9	NA	1.10	10.0
Diethyl phthalate	84-66-2	50	0.976	10.0
Dimethyl phthalate	131-11-3	50	0.766	10.0
Di-n-butyl phthalate	84-74-2	50	0.840	10.0

Analyte	CAS Number	State Regulatory Standard * ug/L	Laboratory **	
			Method Detection Limit (MDL) ug/L	Reporting Limit ug/L
Di-n-octyl phthalate	117-84-0	50	0.749	10.0
Fluoranthene	206-44-0	50	0.842	10.0
Fluorene	86-73-7	50	0.912	10.0
Hexachlorobenzene	118-74-1	0.04	0.396	1.00
Hexachlorobutadiene	87-68-3	0.5	0.780	1.00
Hexachlorocyclopentadiene	77-47-4	5	3.64	10.0
Hexachloroethane	67-72-1	5	0.803	2.00
Indeno[1,2,3-cd]pyrene	193-39-5	0.002	0.939	2.00
Isophorone	78-59-1	50	0.798	10.0
Naphthalene	91-20-3	10	0.541	2.00
Nitrobenzene	98-95-3	0.4	0.567	1.00
N-Nitrosodi-n-propylamine	621-64-7	NA	0.430	1.00
N-Nitrosodiphenylamine	86-30-6	50	0.891	10.0
Pentachlorophenol	87-86-5	NA	1.45	20.0
Phenanthrene	85-01-8	50	1.28	10.0
Phenol	108-95-2	1	0.292	10.0
Pyrene	129-00-0	50	1.64	10.0

Notes:

* State Regulatory Standard – NYSDEC Class GA Ambient Water Quality Standards and Guidance Values listed in TOGS 1.1.1 and Part 703.5: "Water Quality Standards Surface Water and Groundwater"

** Some of the target analytes have a lower State Regulatory Standard than the corresponding Method Detection Limit and/or Reporting Limit. The specified Method Detection Limit and Reporting Limit are the lowest concentrations that the laboratory can achieve given the constraints of the analytical method and associated equipment.

NA – A standard has not been established.

ND – The standard is a non-detectable concentration.

Matrix: Aqueous
Analytical Group: TCL/Part 375 Metals
Concentration Level: Low
Analytical Method: 6020B / 7470A / 7196A

Analyte	CAS Number	State Regulatory Standard * µg/L	Laboratory **	
			Method Detection Limit (MDL) µg/L	Reporting Limit µg/L
Aluminum	7429-90-5	NS	19.5	40.0
Antimony	7440-36-0	3	0.757	2.00
Arsenic	7440-38-2	25	0.887	2.00
Barium	7440-39-3	1,000	0.913	4.00
Beryllium	7440-39-3	1,000	0.130	0.800
Cadmium	7440-43-9	5	0.386	2.00
Calcium	7440-70-2	NS	53.6	500
Chromium, Total	7440-47-3	50	2.50	4.00
Chromium, Hexavalent	18540-29-9	50	8.14	10.0
Cobalt	7440-48-4	NS	0.707	4.00
Copper	7440-50-8	200	2.45	4.00
Iron	7439-89-6	300	58.2	120
Lead	7439-92-1	25	0.844	1.20
Magnesium	7439-95-4	35,000	46.9	200
Manganese	7439-96-5	300	1.47	8.00
Mercury	7439-97-6	0.7	0.091	0.200
Nickel	7440-02-0	100	0.905	4.00
Potassium	7440-09-7	NS	112	200
Selenium	7782-49-2	10	0.589	2.50
Silver	7440-22-4	50	0.290	2.00
Sodium	7440-23-5	20,000	219	500
Thallium	7440-28-0	0.5	0.208	0.800
Vanadium	7440-62-2	NA	0.681	4.00
Zinc	7440-66-6	2,000	6.52	16.0

Notes:

* State Regulatory Standard – NYSDEC Class GA Ambient Water Quality Standards and Guidance Values listed in TOGS 1.1.1 and Part 703.5: "Water Quality Standards Surface Water and Groundwater"

** Thallium has a lower State Regulatory Standard than the corresponding laboratory Reporting Limit. The specified Reporting Limit is the lowest concentration that the laboratory can achieve given the constraints of the analytical method and associated equipment.

Matrix: Aqueous
Analytical Group: PCBs
Concentration Level: Low
Analytical Method: 8082A

Analyte	CAS Number	State Regulatory Standard * µg/L	Laboratory **	
			Method Detection Limit (MDL) µg/L	Reporting Limit µg/L
Polychlorinated Biphenyls, Total	1336-36-3	0.09	0.119	0.400

Notes:

* State Regulatory Standard – NYSDEC Class GA Ambient Water Quality Standards and Guidance Values listed in TOGS 1.1.1 and Part 703.5: "Water Quality Standards Surface Water and Groundwater"

** Total PCBs has a lower State Regulatory Standard than the corresponding laboratory Method Detection Limit and Reporting Limit. The specified Method Detection Limit and Reporting Limit are the lowest concentrations that the laboratory can achieve given the constraints of the analytical method and associated equipment.

Matrix: Aqueous
Analytical Group: TCL/Part 375 Pesticides
Concentration Level: Low
Analytical Method: 8081B

Analyte	CAS Number	State Regulatory Standard * µg/L	Laboratory **	
			Method Detection Limit (MDL) µg/L	Reporting Limit µg/L
4,4'-DDD	72-54-8	0.3	0.00600	0.0200
4,4'-DDE	72-55-9	0.2	0.00200	0.0200
4,4'-DDT	50-29-3	0.2	0.00400	0.0200
Aldrin	309-00-2	ND	0.00300	0.0200
alpha-BHC	319-84-6	0.01	0.00700	0.0200
beta-BHC	319-85-7	0.04	0.0150	0.0200
Chlordane (technical)	12789-03-6	NA	0.0550	0.500
cis-Chlordane	5103-71-9	NA	0.00200	0.0200
delta-BHC	319-86-8	0.04	0.00500	0.0200
Dieldrin	60-57-1	0.004	0.00300	0.0200
Endosulfan I	959-98-8	NA	0.00200	0.0200
Endosulfan II	33213-65-9	NA	0.00400	0.0200
Endosulfan sulfate	1031-07-8	NA	0.00600	0.0200
Endrin	72-20-8	ND	0.00400	0.0200

Analyte	CAS Number	State Regulatory Standard * µg/L	Laboratory **	
			Method Detection Limit (MDL) µg/L	Reporting Limit µg/L
Endrin aldehyde	7421-93-4	5	0.00800	0.0200
Endrin ketone	53494-70-5	5	0.00800	0.0200
gamma-BHC (Lindane)	58-89-9	0.05	0.0120	0.0200
Heptachlor	76-44-8	0.04	0.00300	0.0200
Heptachlor epoxide	1024-57-3	0.03	0.00500	0.0200
Methoxychlor	72-43-5	35	0.00400	0.0200
Toxaphene	8001-35-2	0.06	0.110	0.500
trans-Chlordane	5103-74-2	NA	0.00300	0.0200

Notes:

* State Regulatory Standard – NYSDEC Class GA Ambient Water Quality Standards and Guidance Values listed in TOGS 1.1.1 and Part 703.5: "Water Quality Standards Surface Water and Groundwater"

** Some of the target analytes have a lower State Regulatory Standard than the corresponding laboratory Method Detection Limit and/or Reporting Limit. The specified Method Detection Limit and Reporting Limit are the lowest concentrations that the laboratory can achieve given the constraints of the analytical method and associated equipment.

NA – A standard has not been established.

ND – The standard is a non-detectable concentration.

Matrix: Aqueous
Analytical Group: TCL/Part 375 Herbicides
Concentration Level: Low
Analytical Method: 8151A

Analyte	CAS Number	State Regulatory Standard * µg/L	Laboratory **	
			Method Detection Limit (MDL) µg/L	Reporting Limit µg/L
2,4,5-T	93-76-5	35	0.120	1.20
2,4-D	94-75-7	50	0.130	1.20
2,4,5-TP (Silvex)	93-72-1	0.26	0.110	1.20

Notes:

* State Regulatory Standard – NYSDEC Class GA Ambient Water Quality Standards and Guidance Values listed in TOGS 1.1.1 and Part 703.5: "Water Quality Standards Surface Water and Groundwater"

** 2,4,5-TP (Silvex) has a lower State Regulatory Standard than the corresponding laboratory Reporting Limit. The specified Reporting Limit is the lowest concentration that the laboratory can achieve given the constraints of the analytical method and associated equipment.

9.0 BROWNFIELDS QAPP TEMPLATE #5D - ANALYTICAL LABORATORY SENSITIVITY AND PROJECT CRITERIA

Laboratory performance criteria are provided in this Section.

Matrix: Soil and Aqueous
Analytical Group: TCL/Part 375 VOCs
Concentration Level: Low

Analytical Method/SOP	Data Quality Indicators (DQIs)	Measurement Performance Criteria	QC Sample and/or Activity Used to Assess Measurement Performance	QC Sample Assesses Error for Sampling (S), Analytical (A) or Both (S&A)
8260D [(1)]	Precision – Lab	RPD ≤ 30% for soils RPD ≤ 20% for aqueous	MS/MSD	A
	Accuracy	Recovery within lab statistical QC limits provided in Sections 8.1 and 8.2 and 26.0 to 29.0	LCS	A
	Accuracy	Recovery within lab statistical QC limits provided in Sections 8.1 and 8.2 and 26.0 to 29.0	MS/MSD	A
	Accuracy	Per lab limits provided in Sections 8.1 and 8.2 and 26.0 to 29.0	Surrogate spike	A
	Sensitivity	< QL	Low point calibration standard	A
	Accuracy	Target analytes must be < RL except for common laboratory contaminates (acetone, methylene chloride and MEK) which must be < 5x RL, surrogates in criteria	Method Blank	A
	Accuracy	Minimum 5-standards; must contain all targets and lowest standard ≤ RL; Full Scan: %RSD ≤ 20% for all compounds and minimum RF found in Sections 8.1 and 8.2 and 26.0 to 29.0or "r" ≥ 0.99; SIM %RSD ≤ 20% and minimum RF found in Table 4 or "r" ≥ 0.99 for all compounds	Initial Calibration (ICAL)	A
	Accuracy	Concentration level near mid-point of ICAL curve containing all target compounds; Full Scan and SIM: min RRF criteria met; %D or % Drift ≤ 20% for all compounds	Continuing Calibration Verification (CCV)	A

Note:
(1) The Initial Calibration Blank (ICB), Continual Calibration Blank (CCB), and Instrument Blank (IB) are not EPA Method SW846 specific QC types. Method blanks are analyzed and evaluated as per the Eurofins Environment Testing America SOPs for 8260D, which indicates that method blanks are analyzed every 12 hours immediately after calibration verification.

Matrix: Soil and Aqueous
Analytical Group: TCL/Part 375 SVOCs
Concentration Level: Low

Analytical Method/SOP	Data Quality Indicators (DQIs)	Measurement Performance Criteria	QC Sample and/or Activity Used to Assess Measurement Performance	QC Sample Assesses Error for Sampling (S), Analytical (A) or Both (S&A)
8270E [(1)]	Precision – Lab	RPD ≤ 20% for aqueous and ≤ 30% for soil	MS/MSD	A
	Accuracy	Recovery within lab statistical QC limits provided in Sections 8.1 and 8.2	LCS	A
	Accuracy	Recovery within lab statistical QC limits provided in Sections 8.1 and 8.2	MS/MSD	A
	Accuracy	Per lab limits provided in Sections 8.1 and 8.2 and 26.0 to 29.0; for water matrices 30-130% for BN surrogates and 15- 110% for acid surrogates	Surrogate spike	A
	Sensitivity	< QL	Low point calibration standard	A
	Contamination/ Cross-Contamination	Must be matrix matched; Targets < RL, surrogates in criteria	Method Blank	A
	Accuracy	Minimum 5-standards; must contain all targets and lowest ≤ RL; Full Scan: RF see limits provided in Sections 8.1 and 8.2 and 26.0 to 29.0, %RSD ≤ 20% for all compounds or "r" ≥ 0.99: SIM: %RSD ≤ 20% or "r" ≥ 0.99 for all compounds	Initial Calibration (ICAL)	A
	Accuracy	Concentration level near mid-point of ICAL curve containing all target compounds; Full Scan: %D or %Drift ≤ 20% for CCCs and ≤ 30% for all other compounds; SIM: %D or %Drift ≤ 30%	Continuing Calibration Verification (CCV)	A

Note:
(1) The Initial Calibration Blank (ICB), Continual Calibration Blank (CCB), and Instrument Blank (IB) are not EPA Method SW846 specific QC types. Method blanks are analyzed and evaluated as per the Eurofins Environment Testing America SOPs for 8270E, which indicates that method blanks are prepared and analyzed with each batch of 20 client samples.

Matrix: Soil and Aqueous
Analytical Group: TCL/Part 375 Metals
Concentration Level: Low

Analytical Method/ SOP	Data Quality Indicators (DQIs)	Measurement Performance Criteria	QC Sample and/or Activity Used to Assess Measurement Performance	QC Sample Assesses Error for Sampling (S), Analytical (A) or Both (S&A)
6020B, 7470A, 7471B	Precision – Lab	Aqueous Results ≥ 5xRL, RPD ≤ 20%; Results < 5xRL: absolute difference between results ≤ RL. Soil Results ≥ 5xRL, RPD ≤ 35%: absolute difference between result ≤ RL	Laboratory Duplicates	A
	Accuracy	% Recovery 80-120%	LCS	A
	Accuracy	80-120% or as per lab limits provided in Sections 8.1 and 8.2	MS/MSD	A
	Accuracy	80-120% or as per lab limits provided in Sections 8.1 and 8.2	PDS	A
	Sensitivity	< QL	Low point calibration standard	A
	Accuracy	Minimum of 3 calibration levels plus blank; RL and Linear Range (LR) standards may be included in calibration levels; minimum of 3 integrations for each QC and field sample; linear curve fit r ≤ 0.998; if not including RL and LR standards then LLCV and HLCV check standards need to be analyzed (see below).	Initial Calibration (ICAL)	A
	Accuracy	Separate-source from calibration standards; Must contain all target analytes at the mid-range of the calibration curve ICV: 90-110% recovery	Initial Calibration Verification (ICV)	A
	Accuracy	Same source as initial calibration standards; Must contain all target analytes at the mid-range of the calibration curve CCV: 90-110% recovery	Continuing Calibration Verification (CCV)	A
	Contamination/ Cross-Contamination	Must be digested with samples using same preparation method and amount of acids; MB: ≤ RL	Method Blank	A
	Contamination/ Cross-Contamination	Must be matrix-matched (and same conc. of acid found in standards and samples); ICB/CCB: ≤ RL	Initial Calibration Blank (ICB)	A
	Contamination/ Cross-Contamination	Must be matrix-matched (and same conc. of acid found in standards and samples); ICB/CCB: ≤ RL	Continuing Calibration Blank (CCB)	A

Matrix: Soil and Aqueous
Analytical Group: Mercury
Concentration Level: Low

Analytical Method/ SOP	Data Quality Indicators (DQIs)	Measurement Performance Criteria	QC Sample and/or Activity Used to Assess Measurement Performance	QC Sample Assesses Error for Sampling (S), Analytical (A) or Both (S&A)
7471B	Precision – Lab	Aqueous Results ≥ 5xRL, RPD ≤ 20%; Results < 5xRL: absolute difference between results ≤ RL. Soil Results ≥ 5xRL, RPD ≤ 35%; Results < 5xRL: absolute difference between results ≤ 2xRL	MS/MSD	A
	Accuracy	% Recovery 80-120% or as per lab limits provided in Sections 8.1 and 8.2 and 26.0 to 29.0	LCS/ICV	A
	Accuracy	75-125% or as per lab limits provided in Sections 8.1 and 8.2 and 26.0 to 29.0	MS/MSD	A
	Sensitivity	< QL	Low point calibration standard	A
	Accuracy	Minimum of 5 calibration levels plus blank; low level standard at level of RL; linear regression with a correlation coefficient r > 0.995	Initial Calibration (ICAL)	A
	Accuracy	Separate-source from calibration standards; ICV: 90-110% recovery	Initial Calibration Verification (ICV)	A
	Accuracy	Same source as calibration standards; conc. near mid-point of calibration curve; CCV: 80-120% recovery	Continuing Calibration Verification (CCV)	A
	Contamination/ Cross-Contamination	Must be digested with samples using same preparation method and amount of acids; MB: < RL	Method Blank	A
	Contamination/ Cross-Contamination	Must be matrix-matched (and same conc. of acid found in standards and samples); ICB/CCB: ≤ RL	Initial Calibration Blank (ICB)	A
	Contamination/ Cross-Contamination	Must be matrix-matched (and same conc. of acid found in standards and samples); ICB/CCB: ≤ RL	Continuing Calibration Blank (CCB)	A

Matrix: Soil and Aqueous
Analytical Group: Hexavalent Chromium
Concentration Level: Low

Analytical Method/ SOP	Data Quality Indicators (DQIs)	Measurement Performance Criteria	QC Sample and/or Activity Used to Assess Measurement Performance	QC Sample Assesses Error for Sampling (S), Analytical (A) or Both (S&A)
7196A	Precision – Lab	RPD ≤ 20% for aqueous and soils	MS/MSD	A
	Accuracy	% Recovery 80-120% or as per lab limits provided in Sections 8.1 and 8.2 and 26.0 to 29.0	LCS/	A
	Accuracy	75-125% or as per lab limits provided in Sections 8.1 and 8.2 and 26.0 to 29.0	MS/MSD	A
	Accuracy	Minimum of 3 calibration levels plus blank; low-level standard at level of RL linear regression with a correlation coefficient r > 0.995	Initial Calibration (ICAL)	A
	Accuracy	Separate-source from calibration standards; ICV: 90-110% recovery	Initial Calibration Verification (ICV)	A
	Accuracy	Concentration level near midpoint of calibration curve; same source from ICV; CCV: 90-110% recovery	Continuing Calibration Verification (CCV)	A
	Contamination/ Cross-Contamination	Must be prepared/digested with samples in batch; MB: < RL	Method Blank	A
	Contamination/ Cross-Contamination	Must be matrix-matched (conc. of solution to match standards and samples); ICB/CCB: ≤ RL	Initial Calibration Blank (ICB)	A
	Contamination/ Cross-Contamination	Must be matrix-matched (conc. of solution to match standards and samples); ICB/CCB: ≤ RL	Continuing Calibration Blank (CCB)	A

Matrix: Soil and Aqueous
Analytical Group: PCBs
Concentration Level: Low

Analytical Method/SOP	Data Quality Indicators (DQIs)	Measurement Performance Criteria	QC Sample and/or Activity Used to Assess Measurement Performance	QC Sample Assesses Error for Sampling (S), Analytical (A) or Both (S&A)
8082A [(1)]	Precision – Lab	RPD ≤ 30% for soils and RPD ≤ 20% for aqueous	MS/MSD	A
	Accuracy	Recovery within lab statistical QC limits provided in Sections 8.1 and 8.2 and 26.0 to 29.0	LCS	A
	Accuracy	Recovery within lab statistical QC limits provided in Sections 8.1 and 8.2 and 26.0 to 29.0; Must contain Aroclors 1016 and 1260, performed on Site field sample	MS/MSD	A
	Accuracy	Per lab limits provided in Sections 8.1 and 8.2 and 26.0 to 29.0	Surrogate spike	A
	Sensitivity	< QL	Low point calibration standard	A
	Contamination/ Cross-Contamination	All Target compounds < RL, surrogates in criteria	Method Blank	A
	Accuracy	Minimum 5-levels for Aroclors 1016 and 1260 and single-level at midpoint concentration for other Aroclors; 3-5 peaks of each Aroclor evaluated using peak height or peak area; lowest level ≤ RL; other Aroclors may be warranted for 5 point calibration if PCB contamination is known. %RSD ≤ 20% or "r" ≥ 0.99 for Aroclors 1016 and 1260; regression analysis, if used, must not be forced through the origin	Initial Calibration (ICAL)	A
	Accuracy	Concentration level near mid-point of ICAL curve containing Aroclors 1016 and 1260; %D ≤ 20% and analytes fall within expected retention time windows; Aroclors other than 1016 and 1260 must be verified within 12 hours of being detected in a sample (unless I.S. quant technique is used)	Continuing Calibration Verification (CCV)	A

Note:
(1) The Initial Calibration Blank (ICB), Continual Calibration Blank (CCB), and Instrument Blank (IB) are not EPA Method SW846 specific QC types. Method blanks are analyzed and evaluated as per the Eurofins Environment Testing America SOPs for 8082A, which indicates that method blanks are prepared and analyzed with each batch of 20 client samples.

Matrix: Soil and Aqueous
Analytical Group: TCL/Part 375 Pesticides
Concentration Level: Low

Analytical Method/SOP	Data Quality Indicators (DQIs)	Measurement Performance Criteria	QC Sample and/or Activity Used to Assess Measurement Performance	QC Sample Assesses Error for Sampling (S), Analytical (A) or Both (S&A)
8081B [(1)]	Precision – Lab	RPD ≤ 30%	MS/MSD	A
	Accuracy	Recovery within lab statistical QC limits provided in Sections 8.1 and 8.2	LCS	A
	Accuracy	Recovery within lab statistical QC limits provided in Sections 8.1 and 8.2	MS/MSD	A
	Accuracy	Per lab limits provided in Sections 8.1 and 8.2 and 26.0 to 29.0	Surrogate spike	A
	Sensitivity	< QL	Low point calibration standard	A
	Accurancy	Retention Time Windows: The center of the RT window is updated based on the midpoint std of the ICAL or the first CCV in the daily sequence, whichever is most recent.	RT windows should be narrower than ±0.03 minutes. If RT windows wider that this are necessary, the instrument should be evaluated and maintenance performed as needed.	A
	Contamination/ Cross-Contamination	Must be matrix matched; Targets < RL, surrogates in criteria	Method Blank	A
	Accuracy	Minimum 5-standards; must contain all targets and lowest ≤ RL; %RSD ≤ 20% for all compounds or "r" ≥ 0.99	Initial Calibration (ICAL)	A
	Accuracy	Source separate from ICAL. Concentration level near mid-point of ICAL curve containing all target compounds; %D ≤ 20% for all compounds	Initial Calibration Verification (ICV)	A
	Accuracy	Concentration level near mid-point of ICAL curve containing all target compounds; %D ≤ 20% for all compounds	Continuing Calibration Verification (CCV)	A

Note:
[(1)] The Initial Calibration Blank (ICB), Continual Calibration Blank (CCB), and Instrument Blank (IB) are not EPA Method SW846 specific QC types. Method blanks are analyzed and evaluated as per the Eurofins Environment Testing America SOPs for 8081B, which indicates that method blanks are prepared and analyzed with each batch of 20 client samples.

Matrix: Soil and Aqueous
Analytical Group: TCL/Part 375 Herbicides
Concentration Level: Low

Analytical Method/SOP	Data Quality Indicators (DQIs)	Measurement Performance Criteria	QC Sample and/or Activity Used to Assess Measurement Performance	QC Sample Assesses Error for Sampling (S), Analytical (A) or Both (S&A)
8151A [(1)]	Precision – Lab	RPD ≤ 30%	MS/MSD	A
	Accuracy	Recovery within lab statistical QC limits provided in Sections 8.1 and 8.2	LCS	A
	Accuracy	Recovery within lab statistical QC limits provided in Sections 8.1 and 8.2	MS/MSD	A
	Accuracy	Per lab limits provided in Sections 8.1 and 8.2 and 26.0 to 29.0	Surrogate spike	A
	Sensitivity	< QL	Low point calibration standard	A
	Accurancy	Retention Time Windows: The center of the RT window is updated based on the midpoint std of the ICAL or the first CCV in the daily sequence, whichever is most recent.	RT windows should be narrower than ±0.03 minutes. If RT windows wider that this are necessary, the instrument should be evaluated and maintenance performed as needed.	A
	Contamination/ Cross-Contamination	Must be matrix matched; Targets < RL, surrogates in criteria	Method Blank	A
	Accuracy	Minimum 5-standards; must contain all targets and lowest ≤ RL; %RSD ≤ 20% for all compounds or "r" ≥ 0.99	Initial Calibration (ICAL)	A
	Accuracy	Source separate from ICAL. Concentration level near mid-point of ICAL curve containing all target compounds; %D ≤ 15% for all compounds	Initial Calibration Verification (ICV)	A
	Accuracy	Concentration level near mid-point of ICAL curve containing all target compounds; %D ≤ 15% for all compounds	Continuing Calibration Verification (CCV)	A

Note:
(1) The Initial Calibration Blank (ICB), Continual Calibration Blank (CCB), and Instrument Blank (IB) are not EPA Method SW846 specific QC types. Method blanks are analyzed and evaluated as per the Eurofins Environment Testing America SOPs for 8151A, which indicates that method blanks are prepared and analyzed with each batch of 20 client samples.

10.0 BROWNFIELDS QAPP TEMPLATE #5E - SECONDARY DATA CRITERIA AND LIMITATIONS

Secondary Data	Data Source (Originating Organization, Report Title, and Date)	Data Generator(s) (Originating Org., Data Types, Data Generation/ Collection Dates)	How Data Will Be Used	Limitations on Data Use
Phase I Environmental Site Assessment	Phase I Environmental Site Assessment of the Site, prepared by AKRF, Inc., June 2022	General information on Recognized Environmental Conditions	Indicated possible locations of contaminant sources; may be used to position sampling locations	Qualitative data

11.0 BROWNFIELDS QAPP TEMPLATE #6 – PROJECT-SPECIFIC METHODS AND STANDARD OPERATING PROCEDURES REFERENCE

For field sampling Standard Operating Procedures (SOPs), see Section 25 of this QAPP. Laboratory SOP references are listed in the following table and provided in Appendix A.

Department	Document Number	Rev.	Active Date	SOP Title
Metals	**ED-MT-017**	14	08/11/2022	Mercury Analysis for Water and Wastewater Samples by EPA 245.1 and SW846 Method 7470A; Mercury in Drinking Water using EPA 245.1; Leeman Mercury Analyzer (Cold Vapor Technique)
Metals	**ED-MT-034**	10	06/16/2022	SW-846 Method 6020B, Trace Metals Analysis of Water, Wastewater, Soil, Sediment and Leachate Samples by ICP-MS
Metals	**ED-MT-035**	5	08/11/2022	Mercury Analysis for Solid and Semisolid Waste Samples using the Leeman Mercury Analyzer (Cold Vapor Technique) by SW846 Method 7471B
Metals	**ED-MTP-002**	12	02/10/2022	Digestion of Water and Wastewater Samples for Analysis by ICP and ICP-MS using SW846 Method 3005A
Metals	**ED-MTP-005**	15	09/29/2021	Hot Block Digestion of Sediments, Sludges, and Soils by USEPA Method No(s). SW846 Method 3050B
Organic Preparation	**ED-ORP-002**	11	03/26/2018	Extraction of Semi-Volatile Organic Compounds in Aqueous Samples and Leachates - Separatory Funnel, SW846 Method 3510C
Organic Preparation	**ED-ORP-014**	13	06/27/2019	Extraction of Pesticides and PCBs in Water by Separatory Funnel using SW846 Method 3510C
Organic Preparation	**ED-ORP-015**	15	10/5/2021	Extraction of Organochlorine Herbicides in Water by SW846 Method 8151A
Organic Preparation	**ED-ORP-023**	12	10/8/2020	Extraction of Organochlorine Herbicides in Soil by SW846 Method 8151A
Organic Preparation	**ED-ORP-044**	11	03/12/2018	Procedure for Microwave Extraction of Solids, SW846 3546
SVOA GC	**ED-GCS-005**	12	10/17/2022	Analysis of Organochlorine Herbicides by SW846 Method 8151A
SVOA GC	**ED-GCS-016**	7	10/17/2022	SW846 Method SW8081B, Analysis of Organochlorine Pesticides by Gas Chromatography
SVOA GC	**ED-GCS-017**	7	10/21/2022	SW846 Method 8082A, Analysis of Polychlorinated Byphenyls by Gas Chromatography
SVOA GC/MS	**ED-MSS-009**	10	10/18/2022	Semivolatile Organic Compounds by Gas Chromatography/ Mass Spectrometry (GC/MS), SW846 Methods 8270E
VOA GC/MS	**ED-MSV-001**	10	02/09/2021	Purge and Trap for Aqueous Samples, SW486 Method 5030B and 3050C
VOA GC/MS	**ED-MSV-002**	10	04/11/2019	Closed System Purge and Trap and Extraction for Volatile Organics in Soil, SW846 Method 5035A
VOA GC/MS	**ED-MSV-014**	9	10/17/2022	Volatile Organic Compounds by Gas Chromatography/Mass Spectrometry (GC/MS) by SW846 Method 8260D
Wet Chemistry	**ED-WET-010**	13.1	01/08/2021	Method SW846 3060A, The Alkaline Digestion of Soil Samples for the Analysis of Hexavalent Chromium
Wet Chemistry	**ED-WET-011**	12	06/17/2020	The Analysis of Digestates for Hexavalent Chromium by EPA SW846 7196A
Wet Chemistry	**ED-WET-012**	11	10/01/2021	The Analysis of Waters for Hexavalent Chromium by EPA SW846 7196A
Wet Chemistry	**ED-WET-032**	8	05/08/2019	Percent Moisture Determination

12.0 BROWNFIELDS QAPP TEMPLATE #7 - FIELD EQUIPMENT CALIBRATION, MAINTENANCE, TESTING, AND INSPECTION

Field Equipment (Parameter)	Calibration Activity	Maintenance Activity	Testing/ Inspection Activity	Frequency	Acceptance Criteria	Corrective Action	SOP
MiniRAE PID (Organic vapor)	Instrument calibration with isobutylene	Charge battery Replace or clean sensor	Clean air reading Inspect for visual damage	Calibration – daily Maintenance as needed	As per operator's manual	Recalibrate Perform maintenance	Operation manual
Oil/water interface probe (Depth to GW and LNAPL or DNAPL)	Calibrated by Manufacturer	Check battery and decontaminate between wells	Lower into well water to check alarm Inspect for visual damage	Between wells	Proper tone produced	Replace battery and/or decontaminate	Operation manual
Horiba U-22 water quality meter (conductivity, turbidity, pH, ORP, DO, temperature)	Verify calibration with auto-calibration solution for pH, DO, conductivity, turbidity, ORP	Charge battery	NA Inspect for visual damage	Calibrate at beginning of day After maintenance as required	Calibration does not drift	Recalibrate or replace	Operation model

13.0 BROWNFIELDS QAPP TEMPLATE #8 - ANALYTICAL LABORATORY INSTRUMENTS AND EQUIPMENT

13.1 Analytical Laboratory Instruments and Equipment Maintenance, Testing and Inspection

Instrument/ Method/ SOP	Maintenance Activity	Testing Activity	Inspection Activity	Frequency	Acceptance Criteria	Corrective Action	Responsible Person
GC/MS 8260D 8270E 1A 2A 16A	Check for leaks, replace gas line filters, recondition or replace trap, replace column, clean injection port/liner, replace Electron Multiplier	Tune (BFB or DFTPP), Calibration	Monitor instrument performance via tuning mass criteria, and Calibration criteria	Prior to ICAL	See following table	Replace connections, replace gas line filters, replace trap, replace GC column, clip column, replace injection port liner, clean injection port, replace Electron Multiplier; repeat calibration or CCV	Lab chemist
GC/ECD 8081B 8082A 8151A 10A 11A	Check for leaks, replace gas line filters, recondition or replace column, clean injection port/liner	Calibration	Monitor instrument performance via calibration criteria	See following table	See following table	Replace connections, replace gas line filters, replace GC column, clip column, replace injection port liner, clean injection port; repeat calibration or Continuing Calibration – Verification (CCV)	Lab chemist
ICP –MS 6020B 5A	Perform leak test, change pump tubing, change torch and window, clean filters	Initial Calibration Verification and Initial Calibration (ICAL) Blank	Monitor instrument performance via Initial Calibration Verification and IC Blank	See following table	See following table	Replace pump tubing, replace torch and window, clean all filters; repeat calibration or CCV	Lab chemist
CVAA 7471B 6A	Perform leak test, change tubing, clean window, clean filters	Initial Calibration Verification and ICAL Blank	Monitor instrument performance via Initial Calibration Verification and IC Blank	See following table	See following table	Replace connections, replace pump tubing, clean all filters; repeat calibration or CCV	Lab chemist
UV-Visible Spectrophot-ometer 7196A	Clean ambient flow cell Precision check/alignment of flow cell Wavelength verification check	Monitor instrument performance via CCV and CCB	Monitor instrument performance via CCV and CCB	See following table	See following table	Perform maintenance, replace standards	Lab chemist

13.2 Analytical Laboratory Instrument Calibration

Instrument/ Method/SOP	Calibration Procedure	Frequency of Calibration	Acceptance Criteria	Corrective Action (CA)	Person Responsible for CA
GC/MS 8260D 1A, 2A	Tune Check	Prior to ICAL	Meet ion ratio criteria for reference compound: BFB (SW846 8260D, Table 3), or alternative documented criteria	Recalibrate as required by method; note outliers in narrative	Lab chemist
	Initial Calibration (ICAL)	Initially and when CCAL fails	Minimum 5-standards; must contain all targets and lowest standard ≤RL; Full Scan: limits provided in Sections 8.1 and 8.2 and 26.0 to 29.0 for minimum RF; %RSD ≤ 20% for all compounds or "r" ≥ 0.99; SIM: %RSD ≤ 20% or "r" ≥ 0.99 for all compounds	Recalibrate as required by method; analysis cannot proceed without a valid initial calibration	Lab chemist
	Continuing Calibration – Verification (CCV)	Once every 12 hours prior to sample analysis	Concentration level near mid-point of ICAL curve containing all target compounds; Full Scan and SIM: min RRF criteria met: %D or %Drift ≤ 20% and minimum RF provided in Sections 8.1 and 8.2 and 26.0 to 29.0 or "r" 0.99 for all compounds	Recalibrate as required by method; note outliers in narrative	Lab chemist
GC/MS 8270E 1A, 2A	Tune Check	Prior to ICAL	Meet ion ratio criteria for reference compound: DFTPP (SW846 8270E, Table 3), or alternative documented criteria; Tailing factor ≤ 2 and degradation ≤ 20%	Recalibrate as required by method; analysis cannot proceed without a valid tune check	Lab chemist
	Initial Calibration (ICAL)	Initially and when CCAL fails	Minimum 5-standards; must contain all targets and lowest standard ≤RL; Full Scan: RF limits provided in Sections 8.1 and 8.2 and 26.0 to 29.0; %RSD ≤ 20% for all compounds or "r" ≥ 0.99; SIM: %RSD ≤ 20% or "r" ≥ 0.99 for all compounds	Recalibrate as required by method; analysis cannot proceed without a valid initial calibration	Lab chemist
	Continuing Calibration – Verification (CCV)	Once every 12 hours prior to sample analysis	Concentration level near mid-point of ICAL curve containing all target compounds; Full Scan: %D or %Drift ≤ 20% for CCCs and ≤ 30% for all other compounds; SIM: %D or %Drift ≤ 30%	Recalibrate as required by method; note outliers in narrative	Lab chemist
GC/ECD 8082A 10A, 11A	Initial Calibration (ICAL)	Initially and when CCAL fails	Minimum 5-levels for Aroclors 1016 and 1260 and single-level at midpoint concentration for other Aroclors; 3-5 peaks or each Aroclor evaluated using peak height or peak area; lowest level ≤ RL; other Aroclors may be warranted for 5 point calibration if PCB contamination is known. %RSD ≤ 20% or "r" ≥ 0.99 for Aroclors 1016 and 1260; regression analysis, if used, must not be forced through the origin.	Recalibrate as required by method; analysis cannot proceed without a valid initial calibration	Lab chemist
	Continuing Calibration – Verification (CCV)	Prior to sample, every 12 hours or every 20 samples, whichever is more frequent and at the end of the analytical sequence	Concentration level near mid-point of ICAL curve containing Aroclors 1016 and 1260; %D ≤ ± 20% and analytes fall within expected retention time windows; Aroclors other than 1016 and 1260 must be verified within 12 hours of being detected in a sample (unless I.S. quant technique is used).	Recalibrate as required by method; note outliers in narrative	Lab chemist
ICP-MS 6020B 5A	Initial calibration (ICAL)	Daily following tuning prior to sample analysis	Minimum of 3 calibration levels plus blank; RL and Linear Range (LR) standards may be included in calibration levels; minimum of 3 integrations for each QC and field sample; linear curve fir r ≤ 0.998; if not including RL and LR standards then LLCV and HLCV check standards need to be analyzed.	Re-optimize instrument an recalibrate, repeat until successful	Lab chemist
	Continuing Calibration – Verification (CCV)	Every 10 samples and at end of run	Same source as initial calibration standards; Must contain all target analytes at the mid-range of the calibration curve CCV: 90-110% recovery	Reanalyze. If still out, recalibrate and reanalyze. All samples since last acceptable CCV.	Lab chemist
CVAA 7471B 7470A 6A	Initial Calibration (ICAL)	Daily prior to sample analysis	Minimum of 5 calibration levels plus blank; low level standard at level of RL; linear regression with correlation coefficient r ≥ 0.995	Reanalyze continuing calibration standard. If still outside limits, recalibrate and reanalyze all samples since last complaint calibration standard.	Lab chemist
	Continuing Calibration – Verification (CCV)	Every 10 samples and at end of run	Same source as calibration standards; concentration near mid-point of calibration curve; CCV: -80-120 % recovery	Reanalyze continuing calibration standard. If still outside limits, recalibrate and reanalyze all samples since last complaint calibration standard.	Lab chemist

Instrument/ Method/SOP	Calibration Procedure	Frequency of Calibration	Acceptance Criteria	Corrective Action (CA)	Person Responsible for CA
UV-Visible Spectrophotometer 7196A	Initial Calibration (ICAL)	Daily prior to sample analysis	Minimum of 3 calibration levels plus blank; low level standard at level of RL linear regression with correlation coefficient r ≥ 0.995	Reanalyze continuing calibration standard. If still outside limits, recalibrate and reanalyze all samples since last complaint calibration standard.	Lab chemist
	Continuing Calibration – Verification (CCV)	Every 10 samples and at end of run	Concentration level near mid-point of calibration curve; same source from ICV; CCV: 90-110% recovery.	Reanalyze continuing calibration standard. If still outside limits, recalibrate and reanalyze all samples since last complaint calibration standard.	Lab chemist
GC/ECD 8081B	Initial Calibration (ICAL)	Initially and when CCAL fails	For single response compounds: Minimum 5 levels, lowest level ≤ RL. . %RSD ≤ 20% or "r" ≥ 0.99 . Multi-component pesticides are calibrated using a single point calibration at the anticipated midpoint of the calibration range.	Recalibrate as required by method; analysis cannot proceed without a valid initial calibration	Lab chemist
	Continuing Calibration – Verification (CCV)	For single component pesticides, a mid-point CCV must be analyzed every 12-hours or 20 samples (whichever is more frequent). For multiresponse pesticides a CCV must be analyzed within 12 hours of any multiresponse pesticide detects.	Concentration level near mid-point of ICAL curve containing all target compounds; %D ≤ 20% for all compounds	Recalibrate as required by method; note outliers in narrative	Lab chemist
GC/ECD 8151A	Initial Calibration (ICAL)	Initially and when CCAL fails	Minimum 5 levels, lowest level ≤ RL. RSD for each analyte ≤ 20%; or Linear least squares regression: r ≥ 0.99;	Recalibrate as required by method; analysis cannot proceed without a valid initial calibration	Lab chemist
	Continuing Calibration – Verification (CCV)	A mid-point CCV must be analyzed every 12-hours or 20 samples (whichever is more frequent) and at the end of each analytical sequence. The CCV consists of a midpoint calibration standard.	The calculated concentration of the CCV must be within ±15%D of the expected concentration	Recalibrate as required by method; note outliers in narrative	Lab chemist

14.0 BROWNFIELDS QAPP TEMPLATE #9A - SAMPLE HANDLING SYSTEM

SAMPLE COLLECTION, PACKAGING, AND SHIPMENT
Sample Collection (Personnel/Organization): Michael Bates, AKRF
Sample Packaging (Personnel/Organization): Michael Bates, AKRF
Coordination of Shipment (Personnel/Organization): Michael Bates, AKRF
Type of Shipment/Carrier: Courier or overnight delivery service
SAMPLE RECEIPT AND ANALYSIS
Sample Receipt (Personnel/Organization): Eurofins Environment Testing America Personnel
Sample Custody and Storage (Personnel/Organization): Eurofins Environment Testing America Personnel
Sample Preparation (Personnel/Organization): Eurofins Environment Testing America Personnel
Sample Determinative Analysis (Personnel/Organization): Eurofins Environment Testing America Personnel
SAMPLE ARCHIVING
Field Sample Storage (No. of days from sample collection): Samples to be sent to Eurofins Environment Testing America either by a Eurofins Environment Testing America courier the same day as the sampling or by overnight delivery services to laboratory for delivery the following morning. 1 day
Sample Extract/Digestate Storage (No. of days from extraction/digestion): As per analytical methodology. 30 days
SAMPLE DISPOSAL
Personnel/Organization: Eurofins Environment Testing America
Number of Days from Analysis: Until analysis and QA/QC checks are completed; as per analytical methodology. 30 days

15.0 BROWNFIELDS QAPP TEMPLATE #9B - SAMPLE CUSTODY REQUIREMENTS

15.1 Sample Identification

All samples will be consistently identified in all field documentation, chain of custody (COC) documents and laboratory reports using an alpha-numeric code. All samples will be amended with the Site address number (243) at the beginning of the sample identification and the collection date at the end of the sample name in a year, month, day (YYYYMMDD) format. Blind duplicate sample nomenclature will consist of: the sample type, followed by a letter indicating the associated normal sample matrix (i.e., "S" for soil/fill) and an "X", followed by a sequential number of blind duplicates collected within the SDG and the matrix; and trip and field blanks will consist of "TB-" and "FB-", respectively, followed by a letter indicating the associated normal sample matrix (i.e., "S" for soil/fill), followed by a sequential number of the trip/field blanks collected within the SDG and the matrix. MS/MSD sample nomenclature will consist of the parent sample name only but triplicate sample volume will be collected and the COC comment section will explain that the additional volume is for running the MS/MSD. In accordance with NYSDEC Environmental Quality Information System (EQuIS™) protocols, special characters will not be used for sample nomenclature and sample IDs below 10 will be amended with a "0". Sample nomenclature examples are provided in Table 2.

The following table presents the sampling identification scheme.

Sample Description	Sample Designation
Soil sample collected from 0 to 2 feet from boring SB-01 on August 7, 2023	243-SB-01_0-2_20230807
Trip blank submitted with groundwater samples collected on August 7, 2023	243-TB-A01_20230807
Blind duplicate of soil sample 243-SB-02_0-2_20230807	243-SB-SX01_0-2_20230807
MS/MSD soil sample collected from 0 to 2 feet from boring SB-03 on August 7, 2023	243-SB-03_0-2_20230807
Field blank submitted with soil samples collected on August 7, 2023	243-FB-S01_20230807
Groundwater sample collected from temporary well TW-01 on August 7, 2023	243-TW-01_20230807

Following the labeling of each sample, a laboratory COC form will be completed and will accompany the samples. Each person having custody of the samples will document receipt and relinquishment of such samples.

15.2 Sample Labeling and Shipping

All sample containers will be provided with labels containing the following information:

- Project identification

- Sample identification
- Date and time of collection
- Analysis(es) to be performed
- Sampler's initials

Once the samples are collected and labeled, they will be placed in a chilled container and maintained in a secure environment until transported to the laboratory. The samples will be prepared for shipment by placing each sample container in a sealable plastic bag, then wrapping each container in bubble wrap to prevent breakage, adding fresh ice in sealable plastic bags, and the COC form. Samples will be transported by a laboratory courier or, if necessary, shipped via FedEx.

15.3 Sample Custody

Field personnel will be responsible for maintaining the sample containers in a secured location until sample custody is relinquished. The record of possession of samples from the time they are obtained in the field to the time they are delivered to the laboratory or shipped off-site will be documented on COC forms. The COC forms will contain the following information: project name; names of sampling personnel; sample number; date and time of collection and matrix; and signatures of individuals involved in sample transfer, and the dates and times of transfers. A blank example COC for is provided below.

Chain of Custody Record

eurofins | Environment Testing America

>> Select a Laboratory or Service Center <<
#N/A
#N/A
#N/A
##

Regulatory Program: ☐ DW ☐ NPDES ☐ RCRA ☐ Other:

Eurofins Environment Testing America

Client Contact	Project Manager:	Site Contact:	Date:	COC No:
Your Company Name here	Email:	Lab Contact:	Carrier:	_____ of _____ COCs
Address	Tel/Fax:	Analysis Turnaround Time		TALS Project #:
City/State/Zip		☐ CALENDAR DAYS ☐ WORKING DAYS		Sampler:
(xxx) xxx-xxxx Phone		TAT if different from Below _____		For Lab Use Only:
(xxx) xxx-xxxx FAX		☐ 2 weeks		Walk-in Client:
Project Name:		☐ 1 week		Lab Sampling:
Site:		☐ 2 days		Job / SDG No.:
P O #		☐ 1 day		

Sample Identification	Sample Date	Sample Time	Sample Type (C=Comp, G=Grab)	Matrix	# of Cont.	Filtered Sample (Y / N)	Perform MS / MSD (Y / N)	Sample Specific Notes:

Preservation Used: 1= Ice, 2= HCl; 3= H2SO4; 4=HNO3; 5=NaOH; 6= Other

Possible Hazard Identification:
Are any samples from a listed EPA Hazardous Waste? Please List any EPA Waste Codes for the sample in the Comments Section if the lab is to dispose of the sample.
☐ Non-Hazard ☐ Flammable ☐ Skin Irritant ☐ Poison B ☐ Unknown

Sample Disposal (A fee may be assessed if samples are retained longer than 1 month)
☐ Return to Client ☐ Disposal by Lab ☐ Archive for _____ Months

Special Instructions/QC Requirements & Comments:

Custody Seals Intact: ☐ Yes ☐ No | Custody Seal No.: | Cooler Temp. (°C): Obs'd:_____ Corr'd:_____ | Therm ID No.:_____

Relinquished by:	Company:	Date/Time:	Received by:	Company:	Date/Time:
Relinquished by:	Company:	Date/Time:	Received by:	Company:	Date/Time:
Relinquished by:	Company:	Date/Time:	Received in Laboratory by:	Company:	Date/Time:

Upon receipt at the laboratory, the condition of each sample will be checked to ensure that the sample integrity has not been compromised. Any discrepancy between the samples and the COC information, any broken or leaking sample bottles, or any other abnormal situations will be reported by the laboratory project manager to the AKRF project manager. If required, corrective action options will be discussed and implemented. Notations of the problem and resolution will be made in the laboratory analytical report.

Once the samples are in the custody of the laboratory, sample integrity will be maintained. Each sample batch will be assigned a unique project number by the laboratory and each sample will be assigned a unique laboratory identification number. When samples are required for preparation and/or analysis, the sample custodian or designee will distribute the samples to the appropriate analysts. An internal COC form will be signed by the individual to whom the samples are relinquished to track the samples internally.

16.0 BROWNFIELDS QAPP TEMPLATE #10 - FIELD QUALITY CONTROL SUMMARY

Matrix	Soil and Groundwater
Analytical Group	TCL/Part 375 VOCs
Concentration Level	Low
Sampling SOP(s)	Section 25.1 and 25.3
Analytical Method	8260D
Sampler's Name	Michael Bates
Field Sampling Organization	AKRF
Analytical Organization	Eurofins Environment Testing America
No. of Sample Locations	12 Soil, Max. 3 Groundwater

QC Sample:	Frequency/ Number	Method/SOP QC Acceptance Limits	Corrective Action	Person(s) Responsible for Corrective Action	Data Quality Indicator (DQI)	Measurement Performance Criteria
Matrix Spike	One per batch of 20 or fewer samples of similar matrix	Recovery within lab statistical QC limits provided in Sections 8.1 and 8.2 and 26.0 to 29.0	Note in narrative. If both the LCS and MS/MSD are unacceptable re-prep the samples and QC. Check standard prep. Flag data	Laboratory Supervisor / Data Validator	Accuracy	Recovery within lab statistical QC limits
Matrix Spike Duplicate	One per batch of 20 or fewer samples of similar matrix	Recovery within lab statistical QC limits provided in Sections 8.1 and 8.2 and 26.0 to 29.0; 30% RPD		Laboratory Supervisor / Data Validator	Accuracy & Precision	Recovery within lab statistical QC limits; 30% RPD (aq);
Field Duplicate	One per 20 per matrix	RPDs 30% for waters and < 50% for solids for results > 2x RL	Reanalyze, if necessary, qualify data and narrate issues of nonconformance	Data Validator	Precision	RPDs 30% for waters and < 50% for solids for results > 2x RL
Field Blank	One per 20 per matrix	Results < QL	Flag data as necessary Review potential contaminant sources	Data Validator	Contamination/Cross-Contamination	Results < QL unless target analytes in field samples are > 10x those in EQB
Trip Blank	One per batch of groundwater samples for VOCs analysis	Target analytes < RL	Flag data as necessary Review potential contaminant sources	Laboratory Supervisor / Data Validator	Contamination/Cross-Contamination	Target analytes < RL

Matrix	Soil and Groundwater
Analytical Group	TCL/Part 375 SVOCs
Concentration Level	Low
Sampling SOP(s)	Section 25.1 and 25.3
Analytical Method	8270E
Sampler's Name	Michael Bates
Field Sampling Organization	AKRF
Analytical Organization	Eurofins Environment Testing America
No. of Sample Locations	12 Soil, Max. 3 Groundwater

QC Sample:	Frequency/ Number	Method/SOP QC Acceptance Limits	Corrective Action	Person(s) Responsible for Corrective Action	Data Quality Indicator (DQI)	Measurement Performance Criteria
Matrix Spike	One per batch of 20 or fewer samples of similar matrix	Recovery within lab statistical QC limits provided in Sections 8.1 and 8.2 and 26.0 to 29.0	If recoveries are outside limits and surrogate and LCS criteria are met, note in narrative. If both the LCS and MS/MSD are unacceptable re-prepare the samples and QC. Check standard prep. Flag data	Laboratory Supervisor / Data Validator	Accuracy	Recovery within lab statistical QC limits
Matrix Spike Duplicate	One per batch of 20 or fewer samples of similar matrix	Recovery within lab statistical QC limits provided in Sections 8.1 and 8.2 and 26.0 to 29.0		Laboratory Supervisor / Data Validator	Accuracy & Precision	Recovery within lab statistical QC limits
Field Duplicate	One per 20 per matrix	RPDs 30% for waters and < 50% for solids for results > 2x RL	Evaluate batch precision, other duplicate results Note in Validation Report	Data Validator	Precision	RPDs 30% for waters and < 50% for solids for results > 2x RL
Field blank	One per 20 per matrix	Results < QL	Flag data as necessary Review potential contaminant sources	Data Validator	Contamination/ Cross-Contamination	Results < QL unless target analytes in field samples are > 10x those in EQB

Matrix	Soil and Groundwater
Analytical Group	TCL/Part 375 Metals
Concentration Level	Low
Sampling SOP(s)	Section 25.1 and 25.3
Analytical Method	6020B, 7471B, 7470A
Sampler's Name	Michael Bates
Field Sampling Organization	AKRF
Analytical Organization	Eurofins Environment Testing America
No. of Sample Locations	12 Soil, Max. 3 Groundwater

QC Sample:	Frequency/ Number	Method/SOP QC Acceptance Limits	Corrective Action	Person(s) Responsible for Corrective Action	Data Quality Indicator (DQI)	Measurement Performance Criteria
Matrix Spike Sample	One per prep batch of 20 or fewer samples of similar matrix	% Recovery 75% - 125%	Perform post-digestion spike analysis, qualify data	Laboratory Supervisor / Data Validator	Accuracy	Recovery ± 25 % of true value if sample < 4x spike value
Field Duplicates	One per 20 per matrix	Aq.: Results ≥ 5xRL: RPD ≤ 30%; Results < 5xRL: professional judgment Soil/Sediment: Results ≥ 5xRL: RPD ≤ 50%; Results < 5xRL	Evaluate batch precision, other duplicate results Note in Validation Report	Data Validator	Precision	Aq.: Results ≥ 5xRL: RPD ≤ 30%; Results < 5xRL: professional judgment Soil/Sediment: Results ≥ 5xRL: RPD ≤ 50%; Results < 5xRL
Field blank	One per 20 per matrix	Results < QL	Flag data as necessary Review potential contaminant sources	Data Validator	Contamination / Cross-Contamination	Results < QL unless target analytes in field samples are > 10x those in EQB

Matrix	Soil and Groundwater
Analytical Group	PCBs
Concentration Level	Low
Sampling SOP(s)	Section 25.1 and 25.3
Analytical Method	8082A
Sampler's Name	Michael Bates
Field Sampling Organization	AKRF
Analytical Organization	Eurofins Environment Testing America
No. of Sample Locations	12 Soil, Max. 3 Groundwater

QC Sample:	Frequency/ Number	Method/SOP QC Acceptance Limits	Corrective Action	Person(s) Responsible for Corrective Action	Data Quality Indicator (DQI)	Measurement Performance Criteria
Matrix Spike	One per batch of 20 or fewer samples of similar matrix	Recovery within lab statistical QC limits provided in Sections 8.1 and 8.2 and 26.0 to 29.0	If recoveries are outside limits and surrogate and LCS criteria are met, note in narrative. If both the LCS and MS/MSD are unacceptable re-prepare the samples and QC. Check standard prep. Flag data	Laboratory Supervisor / Data Validator	Accuracy	Recovery within lab statistical QC limits
Matrix Spike Duplicate	One per batch of 20 or fewer samples of similar matrix	Recovery within lab statistical QC limits provided in Sections 8.1 and 8.2 and 26.0 to 29.0; 30% RPD	Same as MS.	Laboratory Supervisor / Data Validator	Accuracy & Precision	Recovery within lab statistical QC limits; 30% RPD
Field blank	One per 20 per matrix	Results < QL	Flag data as necessary Review potential contaminant sources	Data Validator	Contamination / Cross-Contamination	Results < QL unless target analytes in field samples are > 10x those in EQB
Field Duplicate	One per 20 per matrix	RPD ≤ 30% for waters or RPD ≤ 50% for solids w/results > 2x RL	Evaluate batch precision, other duplicate results Note in Validation Report	Data Validator	Precision	RPD ≤ 30% for waters or RPD ≤ 50% for solids w/results > 2x RL

Matrix	Soil and Groundwater
Analytical Group	TCL/Part 375 Pesticides
Concentration Level	Low
Sampling SOP(s)	Section 25.1 and 25.3
Analytical Method	8081B
Sampler's Name	Michael Bates
Field Sampling Organization	AKRF
Analytical Organization	Eurofins Environment Testing America
No. of Sample Locations	12 Soil, Max. 3 Groundwater

QC Sample:	Frequency/ Number	Method/SOP QC Acceptance Limits	Corrective Action	Person(s) Responsible for Corrective Action	Data Quality Indicator (DQI)	Measurement Performance Criteria
Matrix Spike	One per batch of 20 or fewer samples of similar matrix	Recovery within lab statistical QC limits provided in Sections 8.1 and 8.2 and 26.0 to 29.0	If recoveries are outside limits and surrogate and LCS criteria are met, note in narrative. If both the LCS and MS/MSD are unacceptable re-prepare the samples and QC. Check standard prep. Flag data	Laboratory Supervisor / Data Validator	Accuracy	Recovery within lab statistical QC limits
Matrix Spike Duplicate	One per batch of 20 or fewer samples of similar matrix	Recovery within lab statistical QC limits provided in Sections 8.1 and 8.2 and 26.0 to 29.0; 30% RPD	Same as MS.	Laboratory Supervisor / Data Validator	Accuracy & Precision	Recovery within lab statistical QC limits; 30% RPD
Field blank	One per 20 per matrix	Results < QL	Flag data as necessary Review potential contaminant sources	Data Validator	Contamination / Cross-Contamination	Results < QL unless target analytes in field samples are > 10x those in EQB
Field Duplicate	One per 20 per matrix	RPD ≤ 30% for waters or RPD ≤ 50% for solids w/results > 2x RL	Evaluate batch precision, other duplicate results Note in Validation Report	Data Validator	Precision	RPD ≤ 30% for waters or RPD ≤ 50% for solids w/results > 2x RL

Matrix	Soil and Groundwater
Analytical Group	TCL/Part 375 Herbicides
Concentration Level	Low
Sampling SOP(s)	Section 25.1 and 25.3
Analytical Method	8151A
Sampler's Name	Michael Bates
Field Sampling Organization	AKRF
Analytical Organization	Eurofins Environment Testing America
No. of Sample Locations	12 Soil, Max. 3 Groundwater

QC Sample:	Frequency/ Number	Method/SOP QC Acceptance Limits	Corrective Action	Person(s) Responsible for Corrective Action	Data Quality Indicator (DQI)	Measurement Performance Criteria
Matrix Spike	One per batch of 20 or fewer samples of similar matrix	Recovery within lab statistical QC limits provided in Sections 8.1 and 8.2 and 26.0 to 29.0	If recoveries are outside limits and surrogate and LCS criteria are met, note in narrative. If both the LCS and MS/MSD are unacceptable re-prepare the samples and QC. Check standard prep. Flag data	Laboratory Supervisor / Data Validator	Accuracy	Recovery within lab statistical QC limits
Matrix Spike Duplicate	One per batch of 20 or fewer samples of similar matrix	Recovery within lab statistical QC limits provided in Sections 8.1 and 8.2 and 26.0 to 29.0; 30% RPD	Same as MS.	Laboratory Supervisor / Data Validator	Accuracy & Precision	Recovery within lab statistical QC limits; 30% RPD
Field blank	One per 20 per matrix	Results < QL	Flag data as necessary Review potential contaminant sources	Data Validator	Contamination / Cross-Contamination	Results < QL unless target analytes in field samples are > 10x those in EQB
Field Duplicate	One per 20 per matrix	RPD ≤ 30% for waters or RPD ≤ 50% for solids w/results > 2x RL	Evaluate batch precision, other duplicate results Note in Validation Report	Data Validator	Precision	RPD ≤ 30% for waters or RPD ≤ 50% for solids w/results > 2x RL

17.0 BROWNFIELDS QAPP TEMPLATE #11A - DATA MANAGEMENT AND DOCUMENTATION

Field Sample Collection Documents and Records	Analytical Laboratory Documents and Records	Data Assessment Documents and Records	Project File
• Field book/notes • Boring logs • Well development and sampling logs • COC • Photographs • Non-conformance reports • Corrective action reports	• Sample receipt logs • Internal and external COC • Equipment calibration logs • Sample preparation worksheets/logs • Sample analysis worksheets/run logs • Analytical data package • Electronic Data Deliverable (EDD) • Non-conformance reports • Corrective action reports	• Data validation report • Field inspection forms • Corrective action documentation • Electronic Data Deliverables (EDD)	• Project files will be stored on AKRF's server electronically until project completion. • After project completion, electronic project files will be archived on the AKRF corporate server in perpetuity

18.0 BROWNFIELDS QAPP TEMPLATE #11B - PROJECT REPORTS

Type of Report	Projected Delivery Date *	Person(s) Responsible for Report Preparation (Title and Organizational Affiliation)	Report Recipient(s) (Title and Organizational Affiliation)
Environmental Site Investigation Report	09/12/2023	Deborah Shapiro, QEP Senior Vice President AKRF, Inc.	Neal Stone, AICP/MCIP Project Planning Manager Town of North Hempstead

* Project delivery date is based on timing of work plan approval and implementation, as outlined in Section 5.0.

19.0 BROWNFIELDS QAPP TEMPLATE #12A - PLANNED PROJECT ASSESSMENTS

No project assessments are planned for this project.

20.0 BROWNFIELDS QAPP TEMPLATE #12B - ASSESSMENT FINDINGS AND CORRECTIVE ACTIONS RESPONSES

No project assessments or corrective action responses are planned for this project.

21.0 BROWNFIELDS QAPP TEMPLATE #13A - PROJECT DATA VERIFICATION (STEP I)

Step I is a completeness check. The following processes will be followed to verify project data:

Verification Input	Description	Internal/ External	Responsible for Verification (Name, Organization)
Field book	All entries complete, signed, corrections properly initialed, sample list corresponds to COC	I	Michelle Lapin, AKRF
COC	Field COC is completed with legible sample ID, dates, times, all analytical parameters correctly entered, preservatives noted, signatures	I	Michelle Lapin, AKRF
	Lab COC indicates any errors, signatures signifying acceptance of custody	E	Lab sample custodian, Eurofins Environment Testing America
Sample receiving document	Lab verified against COC	E	Lab sample custodian, Eurofins Environment Testing America
Draft lab results	All samples have results as requested, IDs match COC, all QC present and reported as per QAPP	I	Deborah Shapiro or Tim McClintock, AKRF
Analytical data package	Verify data package for completeness including the presence of Laboratory case narrative, sample receipt form, holding times record, sample results, blank results, MS/MSD summary forms, LCS summary forms, surrogate and internal summary forms (where appropriate), initial and continuing calibration summary and raw data.	E	Third Party Validator; Lori Beyer, L.A.B. Validation
Lab originated NCRs/CARS	When required, properly completed with appropriate corrective action specifies and signatues where required; properly filed.	I	Michelle Lapin, AKRF
Memo regarding QAPP modifications or deviations	When required, document all QAPP modifications.	I	Deborah Shapiro or Tim McClintock, AKRF
Analytical EDDs	Verify that all SDGs are reported in Excel format	I	Deborah Shapiro or Tim McClintock, AKRF

Acronyms: COC – Chain of Custody; MS/MSD – Matrix Spike/Matrix Spike Duplicate; LCS – Laboratory Control Sample; NCRs – Nonconformance Reports; CARs – Corrective Action Reports; EDDs – Electronic Data Deliverables; SDGs –Sample Delivery Groups

22.0 BROWNFIELDS QAPP TEMPLATE #13B - PROJECT DATA VALIDATION PROCESS (STEPS IIA AND IIB)

The following processes will be followed to validate project data under Step IIa (Compliance with Methods Procedures and Contracts) and Step IIb (Comparison with Performance Criteria in this QAPP).

Step IIa or IIb	Validation Input	Description	Responsible for Validation (Name, Organization)
IIa/IIb	Field book and field data sheets	Ensure that the sampling protocols and SOPs outlined in the QAPP were followed and that any deviations were noted/approved, appropriate QC samples collected, proper sample preservation	Michelle Lapin, AKRF
IIa/IIb	Field originated NCRs/CARS	All issues properly documented, corrective actions were implemented and effective	Deborah Shapiro or Tim McClintock, AKRF
IIa	Chain of Custody forms; sample receiving document	Examine COC forms against QAPP and laboratory requirements (analytical methods, sample, samples have data reported for requested analysis)	Third Party Validator; Lori Beyer, L.A.B. Validation
IIa	Analytical data package Lab SOPs/ Reference methods QAPP MPC	Holding times met all method criteria	Third Party Validator; Lori Beyer, L.A.B. Validation
IIa		Review of dilutions and re-analyses results against reported data; when multiple analyses appropriate run was reported, proper units are reported	Third Party Validator; Lori Beyer, L.A.B. Validation
IIa/IIb		Calibrations were analyzed at required frequency and met criteria	Third Party Validator; Lori Beyer, L.A.B. Validation
IIa/IIb		Comparison of QC sample results (surrogate, internal standards, spikes, blanks, etc) all match criteria in metod and QAPP	Third Party Validator; Lori Beyer, L.A.B. Validation
IIa/IIb		Blanks are free of contamination; if analytes present > RDL samples properly quallified if sample concentration < 10x Blank concentration	Third Party Validator; Lori Beyer, L.A.B. Validation
IIb		Detection limits, project action limits were met	Third Party Validator; Lori Beyer, L.A.B. Validation
IIa/IIb	Lab originated NCRs/CARS	When required, document all issues property and confirm corrective actions were implemented and effective	Deborah Shapiro or Tim McClintock, AKRF
IIb	Memo regarding QAPP modifications	When required, document all QAPP modifications and corrective actions	Deborah Shapiro or Tim McClintock, AKRF
IIb	Analytical EDDs	All data reported in excel format; EDD verified against hard copy lab report	Deborah Shapiro or Tim McClintock, AKRF

Acronyms: MPC – Measurement Performance Criteria; NCRs – Nonconformance Reports; CARs – Corrective Action Reports; EDDs – Electronic Data Deliverables

23.0 BROWNFIELDS QAPP TEMPLATE #13C - PROJECT MATRIX AND ANALYTICAL VALIDATION (STEPS IIA AND IIB) SUMMARY

Step IIa/IIb	Matrix	Analytical Group	Concentration Level	Validation Criteria	Data Validator
IIa	Soil	TCL/Part 375 VOCs, SVOCs, metals, PCBs, pesticides, and herbicides	Low	EPA Method criteria; Laboratory SOPs and control limits	Third Party Data Validator; Lori Beyer, L.A.B. Validation
IIb	Soil	TCL/Part 375 VOCs, SVOCs, metals, PCBs, pesticides, and herbicides	Low	QAPP Templates	Third Party Data Validator; Lori Beyer, L.A.B. Validation
IIa	Ground water	TCL/Part 375 VOCs, SVOCs, metals, PCBs, pesticides, and herbicides	Low	EPA Method criteria; Laboratory SOPs and control limits	Third Party Data Validator; Lori Beyer, L.A.B. Validation
IIb	Ground water	TCL/Part 375 VOCs, SVOCs, metals, PCBs, pesticides, and herbicides	Low	QAPP Templates	Third Party Data Validator; Lori Beyer, L.A.B. Validation

24.0 BROWNFIELDS QAPP TEMPLATE #13D - USABILITY ASSESSMENT (STEP III)

Summarize the usability assessment process and all procedures, including interim steps and any statistics, equations, and computer algorithms that will be used:

Data to be used in evaluating project technical objectives must be assessed to determine whether the data are of sufficient quality to allow for their unrestricted use. It is the joint responsibility of the AKRF project manager to ensure that the data collected meets the requirements specified in this QAPP. After sampling is complete and the laboratory has submitted the final data package, a third party data validator will validate the data and will prepare the DUSR. The DUSR will include limitations of the data and recommendations on the usability of the data for decision making. The AKRF project manager will review the report and, as needed, resolve any issues. The DUSR will be provided as an appendix to the Phase II ESI Report.

The following steps are taken in the data review/validation process. The DUSR will identify any non-conformances and explains any limitations on the use of the data.

Data Review/Validation Steps

- Verify that all collected samples were analyzed, using COC records
- Compare sample collection, extraction and analysis dates to applicable holding times
- Review all calibration records against method and project criteria (MPC)
    - Verify frequency and criteria of initial calibrations
    - GC/MS organics: verify frequency and criteria of tune performance
    - analysis
    - Verify frequency and criteria of continuing calibrations
    - Recalculate one or more data points from raw data (RF/CF/%D)
- Review MDL study and compare to reported DLs
- Verify frequency and criteria of method blanks
- Organic analyses: compare surrogate recovery results to applicable criteria
- GC/MS analyses: Review internal standard results for RT and area criteria
- Precision and accuracy results are reviewed against applicable criteria:
    - Matrix duplicate or MS/MSD results are compared to RPD criteria
    - Matrix spike results are compared to % Recovery criteria
    - Spiked blanked results (LCS) are evaluated against % Recovery criteria
    - Review field duplicate results against MPC
- Evaluate results of any trip and field blanks
- Recalculate 25% of sample results from raw data, using unedited data
- Assign Data Validation Qualifiers (DVQs) as needed

Describe the evaluative procedures used to assess overall measurement error associated with the project:

Sample data that do not meet the measurement performance criteria established in this QAPP will be evaluated to determine whether they are usable for meeting project objectives. Data will be assigned a DVQ when the performance criteria are not met and will be considered as estmated values (DVQ = J or UJ) or will be rejected (DVQ=R) based on the degree and impact of the non-conformance. Non-conformances and the DVQ deterination will be documented in the DUSR.

Guidelines from the EPA Region 2 Data Validation SOPs along with professional judgement will be used to evaluate sample data and assign DVQs.

Trends in precision and accuracy of the project duration will be assessed by calulating average % recovery of all matrix spikes along with minimum and maximum values and the number of results that fall outside control limits. The range of precision values will be reported along with the number of any outliers. Field duplicate results will be evaluated for each matrix to proivde a measure of representativeness and matrix heterogenity. All results, decisions and a discussion of the impact on projetc objectives will be included in the DUSR.

Identify the personnel responsible for performing the usability assessment:

The AKRF project manager and Third Party Data Validator will be responsible for performing the usability assessment.

Describe the documentation that will be generated during usability assessment and how usability assessment results will be presented so that they identify trends, relationships (correlations), and anomalies:

Following data review and validation, the Third Party Data Validator will prepare a DUSR. The report will include the following:

- Introduction: Summarizes the purpose of the QA review and validation process and the samples reviewed.
- Data quality indicators: discussion of the measurements and calculations applied to the assessment of the data quality indicators - precision, accuracy, sensitivity, representativeness, comparability and completeness.
- Conclusions and data usability: provides a summary of the results of the QC measurements (averages, ranges, trends) and discusses any limitations in the use of the analytical data.
- QC Results: all QC measurements evaluated as per above (data review) are presented by analytical parameter. Any non-conformances identified during the review process are discussed with respect to the impact on overall data quality and data usability. QC measurements are summarized and results provided in tabular form (average, range, applicable control limits, number of results outside limits).

25.0 STANDARD OPERATING PROCEDURES (SOPS)

25.1 Soil Sampling

Soil sampling will be conducted according to the following procedures:

- Soil samples will be collected continuously using 2-inch inside diameter macrocore, direct-push samplers equipped with dedicated, acetate sample collection sleeves; or via a decontaminated hand auger. Samples will be collected continuously from surface grade to boring termination depths. Retrieved samplers will be placed on a decontaminated examination table or other flat surface and carefully opened to minimize the soil sample disturbance.
- Soil samples will be examined by AKRF personnel and descriptions will be prepared according to the modified Burmister Classification System, including any evidence of contamination (e.g., staining, presence of ash, oily sheens, odors).
- Boring logs will be maintained to record boring number, sample depth and sample observations (evidence of contamination, PID readings, soil classification).
- Collect an aliquot of soil from each sampling location and place in labeled sealable plastic bags. The bag should be labeled with the soil boring number and the depth the sample was collected. Place the plastic bags in a chilled cooler to await selection of samples for laboratory analysis.
- After selecting which samples will be analyzed in the laboratory, the required laboratory-supplied sample jars will be filled with the soil from the selected sampling locations. Seal and label the sample jars and place in an ice-filled cooler.
- Retrieved samples will be field-screened for the presence of VOCs using a PID equipped with a 10.6 eV lamp and calibrated daily to a 100 parts per million (ppm) isobutylene standard. Samples will be collected from both shallow and deep intervals, based on the results of field screening and visual evidence of contamination. In the absence of contamination, the samples will be collected from the 2-foot interval the surface cover (asphalt, concrete, grass/roots, etc.).
- The soil samples slated for analysis will be collected into laboratory-supplied containers, sealed and labeled, and placed in an ice-filled cooler. All sampling equipment will be decontaminated in accordance with Section 25.5 of this QAPP.

25.2 Groundwater Monitoring Well Development

Following well installation, the wells will be developed according to the following procedure:

- Measure the depth to water using an oil/water interface probe and the total depth of the well using a weighted tape. Use these measurements to calculate the length of the water column. Calculate the volume of water in the well using 0.163 gallons per foot of water column as the conversion factors for a 2-inch diameter well.
- For the first 5-minutes of well development, develop the well using a check valve pump and re-circulate the water back into the well to create maximum agitation. This method

is intended to remove fines from the sand pack, the adjacent formation and from the well.

- After the first 5-minutes of well development, develop the well using a check valve pump and discharge the water to five-gallon buckets. Transfer water from the buckets to 55-gallon drums designated for well development water.
- During development, collect periodic samples and analyze for turbidity and water quality indicators (pH, temperature, dissolved oxygen, reduction-oxidation potential, and specific conductivity) with measurements collected approximately every five minutes.
- Continue developing the well until turbidity is less than 50 nephelometric turbidity units (NTUs) for three successive readings and until water quality indicators have stabilized to within 10% for pH, temperature and specific conductivity for three successive readings, or until at least three well volumes have been purged from the well.
- Document the volume of water removed and any other observations made during well development in the field book or on field data sheets.
- Decontaminate the equipment prior to and following development at each well location as described in Section 25.5 of this QAPP.

25.3 Groundwater Sampling

Groundwater samples will be collected following well development. Sampling will be conducted according to the following procedure:

- Prepare the sampling area by placing plastic sheeting over the well. Cut a hole in the sheeting to provide access to the well cover.
- Remove the locking cap and measure the vapor concentrations in the well with a PID.
- Measure the depth to water and total well depth, and check for the presence of light non-aqueous phase liquid (LNAPL) or dense non-aqueous phase liquid (DNAPL) using an oil/water interface probe. Measure the thickness of NAPL, if any, and record in field book and well log. Groundwater samples will not be collected from wells containing measurable NAPL.
- Use the water level and total well depth measurements to calculate the length of the mid-point of the water column within the screened interval. For example, for a well where the total depth is 20 feet, screened interval is 10 to 20 feet, and depth to water is 12 feet, the mid-point of the water column within the screened interval would be 16 feet.
- Connect dedicated tubing to either a submersible or bladder pump and lower the pump such that the intake of the pump is set at the mid-point of the water column within the screened interval of the well. Connect the discharge end of the tubing to the flow-through cell of a Horiba U-22 multi-parameter (or equivalent) meter. Connect tubing to the output of the cell and place the discharge end of the tubing in a five-gallon bucket.
- Activate the pump at the lowest flow rate setting of the pump.

- Measure the depth to water within the well. The pump flow rate may be increased such that the water level measurements do not change by more than 0.3 feet as compared to the initial static reading. The well-purging rate should be adjusted so as to produce a smooth, constant (laminar) flow rate and so as not to produce excessive turbulence in the well. The expected targeted purge rate will be around 500 mL/minute and will be no greater than 3.8 liters/minute (1 gallon per minute).
- During purging, collect periodic samples and analyze for water quality indicators [e.g., turbidity, pH, temperature, dissolved oxygen, reduction-oxidation potential (ORP), and specific conductivity] with measurements collected approximately every five minutes.
- Continue purging the well until turbidity is less than 50 NTUs and water quality indicators have stabilized to the extent practicable. The criteria for stabilization will be three successive readings for the following parameters and criteria:

Parameter	Stabilization Criteria
PH	+/- 0.1 pH units
Specific Conductance	+/- 3% mS/cm
ORP/Eh	+/- 10mV
Turbidity	<50 NTU
Dissolved Oxygen	+/- 0.3 mg/L

Notes: mS/cm = millisievert per centimeter
mV = millivolts
NTUs = nephthalometric turbidity units
mg/L = milligrams per liter

- If the water quality parameters do not stabilize and/or turbidity is greater than 50 NTUs within 2 hours, purging may be discontinued. Efforts to stabilize the water quality for the well must be recorded in the field book, and samples may then be collected as described herein.
- After purging, disconnect the tubing to the inlet of the flow-through cell. Collect groundwater samples directly from the discharge end of the tubing and place into the required sample containers. Label the containers and place in a chilled cooler.
- Collect one final field sample and analyze for turbidity and water quality parameters (pH, temperature, dissolved oxygen, reduction-oxidation potential, and specific conductivity).
- Once sampling is complete, remove the pump and tubing from the well. Dispose of disposable supplies such as tubing, sample filter, and personal protective equipment (PPE).
- Decontaminate the pump, oil/water interface probe, flow-through cell, and plastic filter chamber as described in Section 25.5.
- Record all measurements (depth to water, depth to NAPL, water quality parameters, turbidity), calculations (well volume) and observations in the field book or field data sheet, if applicable.

25.4 Surveying and Water Table Readings

If three groundwater wells are installed, the wells will be surveyed by a New York State-licensed surveyor. Three elevation measurements will be taken at each well location: the elevation of the ground beside the well; the elevation on the rim of the protective casing; and the elevation of the top of PVC casing.

Water table readings will be taken in the groundwater monitoring wells using an oil/water interface probe. The gate boxes will be unlocked and opened at each well location. The oil/water interface probe will be turned on and sound tested. The probe of the meter will be inserted into the PVC casing. The probe will be lowered down the casing until the meter alarm indicates the probe is at the water table. A reading of the depth from the top of the top of the PVC casing to the groundwater table will be recorded in the field notebook. The probe will then be lowered to the bottom of the well. If the meter alarm indicates separate phase product, a reading of the product thickness will be documented.

25.5 Decontamination

All sampling equipment (drilling rods and casing, macrocore samplers, probe rods, etc.) will be either dedicated or decontaminated between sampling locations. The decontamination procedure will be as follows:

- Scrub using tap water/Alconox™ or Liquinox™ mixture and bristle brush
- Rinse with tap water
- Scrub again with tap water/Alconox™ or Liquinox™ and bristle brush
- Rinse with tap water
- Rinse with distilled water
- Air dry the equipment, if possible

In the absence of contamination, the decontamination fluids will be discharged to the ground in accordance with NYSDEC DER-10. However, if evidence of contamination is noted during sampling or purging, the fluids will be containerized in Department of Transportation (DOT)-approved 55-gallon drums for off-site disposal in accordance with Section 25.6 of this QAPP.

25.6 Management of Investigation-Derived Waste (IDW)

All IDW will be used to backfill the corresponding borehole that generated them to within 24 inches of the surface, or will be disposed of or treated according to applicable local, state, and federal regulations. If additional material is needed, hydrated bentonite powder or chips will be used. The borings will be patched with the appropriate materials (e.g., asphalt, grass, or concrete patch), depending on the original finish. If gross contamination is identified, the soil cuttings will be containerized in DOT-approved 55-gallon drums. The boring will then be grouted with a cement/bentonite grout mixture and then patched at the surface.

In the absence of contamination, decontamination fluids will be discharged to the ground in accordance with NYSDEC DER-10. If needed, any contaminated decontaminated fluids will be containerized in DOT-approved 55-gallon drums.

Any IDW containerized for off-site disposal will be placed in DOT-approved 55-gallon drums, sealed at the end of each work day, and labeled with the date, the boring number(s), the type of waste (i.e., drill cuttings), and the contact information of the AKRF project manager. The drums will be picked up from the Site for proper disposal pending receipt of analytical results. All IDW will be handled according to applicable local, state, and federal regulations.

26.0 QC LIMITS TCL/PART 375 VOCS

Analyte	CAS #	Aqueous Spike Limits		Soil Spike Limits		Aqueous RPD Limits (%)	Soil RPD Limits (%)
		Low Recovery Limit (%)	High Recovery Limit (%)	Low Recovery Limit (%)	High Recovery Limit (%)		
1,1,1-Trichloroethane	71-55-6	68	128	78	120	30	30
1,1,2,2-Tetrachloroethane	79-34-5	63	139	66	123	30	30
1,1,2-Trichloro-1,2,2-trifluoroethane	76-13-1	51	142	75	141	30	30
1,1,2-Trichloroethane	79-00-5	74	125	80	120	30	30
1,1-Dichloroethane	75-34-3	73	130	77	129	30	30
1,1-Dichloroethene	75-35-4	68	133	70	132	30	30
1,2,3-Trichlorobenzene	87-61-6	56	144	44	120	30	30
1,2,4-Trichlorobenzene	120-82-1	67	132	68	150	30	30
1,2,4-Trimethylbenzene	95-63-6	75	125	79	120	30	30
1,2-Dibromo-3-Chloropropane	96-12-8	58	132	60	124	30	30
1,2-Dichlorobenzene	95-50-1	80	120	80	120	30	30
1,2-Dichloroethane	107-06-2	66	129	75	123	30	30
1,2-Dichloropropane	78-87-5	68	128	73	124	30	30
1,3,5-Trimethylbenzene	108-67-8	75	125	79	120	30	30
1,3-Dichlorobenzene	541-73-1	80	120	80	120	30	30
1,4-Dichlorobenzene	106-46-7	80	120	80	120	30	30
2-Butanone (MEK)	78-93-3	61	128	75	120	30	30
2-Hexanone	591-78-6	61	134	70	128	30	30
4-Methyl-2-pentanone (MIBK)	108-10-1	69	128	80	122	30	30
Acetone	67-64-1	61	134	63	131	30	30
Benzene	71-43-2	71	126	80	123	30	30
Bromoform	75-25-2	48	144	70	125	30	30
Bromomethane	74-83-9	32	150	64	150	30	30
Carbon disulfide	75-15-0	64	138	76	120	30	30
Carbon tetrachloride	56-23-5	61	131	77	121	30	30
Chlorobenzene	108-90-7	80	120	80	120	30	30
Chlorobromomethane	74-97-5	67	126	76	127	30	30
Chlorodibromomethane	124-48-1	62	130	80	120	30	30
Chloroethane	75-00-3	42	150	68	132	30	30
Chloroform	67-66-3	78	125	79	126	30	30
Chloromethane	74-87-3	43	150	63	130	30	30
cis-1,2-Dichloroethene	156-59-2	78	121	80	123	30	30
cis-1,3-Dichloropropene	10061-01-5	74	125	80	120	30	30
Cyclohexane	110-82-7	60	133	70	132	30	30
Dichlorobromomethane	75-27-4	76	121	73	124	30	30
Dichlorodifluoromethane	75-71-8	33	150	62	150	30	30
Ethylbenzene	100-41-4	78	120	76	120	30	30
Ethylene Dibromide	106-93-4	79	126	79	120	30	30
Isopropylbenzene	98-82-8	79	125	80	120	30	30
Methyl acetate	79-20-9	55	146	58	143	30	30
Methyl tert-butyl ether	1634-04-4	72	131	80	125	30	30
Methylcyclohexane	108-87-2	54	139	70	133	30	30
Methylene Chloride	75-09-2	74	127	76	120	30	30
m-Xylene & p-Xylene	179601-23-1	78	120	80	120	30	30
n-Butylbenzene	104-51-8	69	135	68	150	30	30
N-Propylbenzene	103-65-1	68	129	78	130	30	30
o-Xylene	95-47-6	78	120	80	120	30	30
sec-Butylbenzene	135-98-8	73	129	78	136	30	30
Styrene	100-42-5	75	127	80	120	30	30
tert-Butylbenzene	98-06-6	72	124	80	128	30	30
Tetrachloroethene	127-18-4	70	127	78	123	30	30
Toluene	108-88-3	78	120	80	120	30	30
trans-1,2-Dichloroethene	156-60-5	74	126	78	120	30	30

Analyte	CAS #	Aqueous Spike Limits		Soil Spike Limits		Aqueous RPD Limits (%)	Soil RPD Limits (%)
		Low Recovery Limit (%)	High Recovery Limit (%)	Low Recovery Limit (%)	High Recovery Limit (%)		
trans-1,3-Dichloropropene	10061-02-6	66	127	80	120	30	30
Trichloroethene	79-01-6	71	121	79	120	30	30
Trichlorofluoromethane	75-69-4	50	150	76	142	30	30
Vinyl chloride	75-01-4	55	144	72	131	30	30
Xylenes, Total	1330-20-7	78	120	80	120	30	30
1,1,1-Trichloroethane	71-55-6	68	128	78	120	30	30

27.0 QC LIMITS TCL/PART 375 SVOCS

Analyte	CAS #	Aqueous Spike Limits		Soil Spike Limits		Aqueous RPD Limits (%)	Soil RPD Limits (%)
		Low Recovery Limit (%)	High Recovery Limit (%)	Low Recovery Limit (%)	High Recovery Limit (%)		
1,1'-Biphenyl	92-52-4	53	120	59	120	30	30
1,2,4,5-Tetrachlorobenzene	95-94-3	46	117	60	120	30	30
2,2'-oxybis[1-chloropropane]	108-60-1	37	120	49	126	30	30
2,3,4,6-Tetrachlorophenol	58-90-2	54	122	54	120	30	30
2,4,5-Trichlorophenol	95-95-4	58	120	59	120	30	30
2,4,6-Trichlorophenol	88-06-2	61	120	58	120	30	30
2,4-Dichlorophenol	120-83-2	65	120	66	120	30	30
2,4-Dimethylphenol	105-67-9	62	120	67	120	30	30
2,4-Dinitrophenol	51-28-5	36	150	36	129	30	30
2,4-Dinitrotoluene	121-14-2	68	134	65	124	30	30
2,6-Dinitrotoluene	606-20-2	65	124	67	121	30	30
2-Chloronaphthalene	91-58-7	52	120	60	120	30	30
2-Chlorophenol	95-57-8	53	120	63	120	30	30
2-Methylnaphthalene	91-57-6	44	120	64	120	30	30
2-Methylphenol	95-48-7	44	120	63	120	30	30
2-Nitroaniline	88-74-4	49	120	48	120	30	30
2-Nitrophenol	88-75-5	60	125	64	120	30	30
3 & 4 Methylphenol	15831-10-4	35	120	61	120	30	30
3,3'-Dichlorobenzidine	91-94-1	37	137	13	136	30	30
3-Nitroaniline	99-09-2	40	120	39	122	30	30
4,6-Dinitro-2-methylphenol	534-52-1	59	135	50	136	30	30
4-Bromophenyl phenyl ether	101-55-3	62	125	67	120	30	30
4-Chloro-3-methylphenol	59-50-7	61	120	66	120	30	30
4-Chloroaniline	106-47-8	29	127	15	128	30	30
4-Chlorophenyl phenyl ether	7005-72-3	59	122	62	120	30	30
4-Methylphenol	106-44-5	33	120	61	120	30	30
4-Nitroaniline	100-01-6	45	120	55	120	30	30
4-Nitrophenol	100-02-7	12	120	52	123	30	30
Acenaphthene	83-32-9	49	120	49	120	30	30
Acenaphthylene	208-96-8	60	120	64	120	30	30
Acetophenone	98-86-2	62	120	61	120	30	30
Anthracene	120-12-7	65	120	67	120	30	30
Atrazine	1912-24-9	43	150	29	150	30	30
Benzaldehyde	100-52-7	41	150	34	150	30	30
Benzo[a]anthracene	56-55-3	63	120	62	120	30	30
Benzo[a]pyrene	50-32-8	60	139	73	123	30	30
Benzo[b]fluoranthene	205-99-2	66	125	70	125	30	30
Benzo[g,h,i]perylene	191-24-2	59	136	66	120	30	30
Benzo[k]fluoranthene	207-08-9	64	125	67	122	30	30
Bis(2-chloroethoxy)methane	111-91-1	64	120	62	120	30	30
Bis(2-chloroethyl)ether	111-44-4	63	120	60	120	30	30
Bis(2-ethylhexyl) phthalate	117-81-7	60	132	59	125	30	30
Butyl benzyl phthalate	85-68-7	58	132	62	127	30	30
Caprolactam	105-60-2	10	120	36	150	30	30
Carbazole	86-74-8	65	120	64	120	30	30
Chrysene	218-01-9	63	120	63	120	30	30
Dibenz(a,h)anthracene	53-70-3	62	140	66	128	30	30
Dibenzofuran	132-64-9	58	120	61	120	30	30
Diethyl phthalate	84-66-2	53	129	63	120	30	30
Dimethyl phthalate	131-11-3	60	124	65	120	30	30
Di-n-butyl phthalate	84-74-2	59	133	66	120	30	30

Analyte	CAS #	Aqueous Spike Limits		Soil Spike Limits		Aqueous RPD Limits (%)	Soil RPD Limits (%)
		Low Recovery Limit (%)	High Recovery Limit (%)	Low Recovery Limit (%)	High Recovery Limit (%)		
Di-n-octyl phthalate	117-84-0	49	135	65	133	30	30
Fluoranthene	206-44-0	65	123	61	120	30	30
Fluorene	86-73-7	58	120	60	120	30	30
Hexachlorobenzene	118-74-1	61	128	66	120	30	30
Hexachlorobutadiene	87-68-3	27	127	62	120	30	30
Hexachlorocyclopentadiene	77-47-4	24	123	38	120	30	30
Hexachloroethane	67-72-1	26	120	61	120	30	30
Indeno[1,2,3-cd]pyrene	193-39-5	59	137	62	130	30	30
Isophorone	78-59-1	68	121	67	120	30	30
Naphthalene	91-20-3	51	120	63	120	30	30
Nitrobenzene	98-95-3	64	120	63	120	30	30
N-Nitrosodi-n-propylamine	621-64-7	60	120	61	120	30	30
N-Nitrosodiphenylamine	86-30-6	63	120	63	120	30	30
Pentachlorophenol	87-86-5	24	131	37	126	30	30
Phenanthrene	85-01-8	65	120	66	120	30	30
Phenol	108-95-2	18	120	63	120	30	30
Pyrene	129-00-0	51	124	61	121	30	30

28.0 QC LIMITS TCL/PART 375 METALS

Analyte	CAS #	Aqueous Spike Limits		Soil Spike Limits		Aqueous RPD Limits (%)	Soil RPD Limits (%)
		Low Recovery Limit (%)	High Recovery Limit (%)	Low Recovery Limit (%)	High Recovery Limit (%)		
Aluminum	7429-90-5	80	120	80	120	20	20
Antimony	7429-90-5	80	120	80	120	20	20
Arsenic	7440-38-2	80	120	80	120	20	20
Barium	7440-39-3	80	120	80	120	20	20
Beryllium	7440-41-7	80	120	80	120	20	20
Cadmium	7440-43-9	80	120	80	120	20	20
Calcium	7440-70-2	80	120	80	120	20	20
Chromium	7440-47-3	80	120	80	120	20	20
Chromium, Hexavalent	18540-29-9	-	-	-	-	20	20
Cobalt	7440-48-4	80	120	80	120	20	20
Copper	7440-50-8	80	120	80	120	20	20
Iron	7439-89-6	80	120	80	120	20	20
Lead	7439-92-1	80	120	80	120	20	20
Magnesium	7439-95-4	80	120	80	120	20	20
Manganese	7439-96-5	80	120	80	120	20	20
Mercury	7439-97-6	80	120	80	120	20	20
Nickel	7440-02-0	80	120	80	120	20	20
Potassium	7440-09-7	80	120	80	120	20	20
Selenium	7782-49-2	80	120	80	120	20	20
Silver	7440-22-4	80	120	80	120	20	20
Sodium	7440-23-5	80	120	80	120	20	20
Thallium	7440-28-0	80	120	80	120	20	20
Vanadium	7440-62-2	80	120	80	120	20	20
Zinc	7440-66-6	80	120	80	120	20	20

29.0 QC LIMITS PCBS

Analyte	CAS #	Aqueous Spike Limits		Soil Spike Limits		Aqueous RPD Limits (%)	Soil RPD Limits (%)
		Low Recovery Limit (%)	High Recovery Limit (%)	Low Recovery Limit (%)	High Recovery Limit (%)		
Aroclor 1016	12674-11-2	53	141	65	133	30	30
Aroclor 1221	11104-28-2	-	-	-	-	-	-
Aroclor 1232	11141-16-5	-	-	-	-	-	-
Aroclor 1242	53469-21-9	-	-	-	-	-	-
Aroclor 1248	12672-29-6	-	-	-	-	-	-
Aroclor 1254	11097-69-1	-	-	-	-	-	-
Aroclor 1260	11096-82-5	55	142	67	150	30	30
Aroclor-1262	37324-23-5	-	-	-	-	-	-
Aroclor 1268	11100-14-4	-	-	-	-	-	-
Polychlorinated biphenyls, Total	1336-36-3	-	-	-	-	-	-

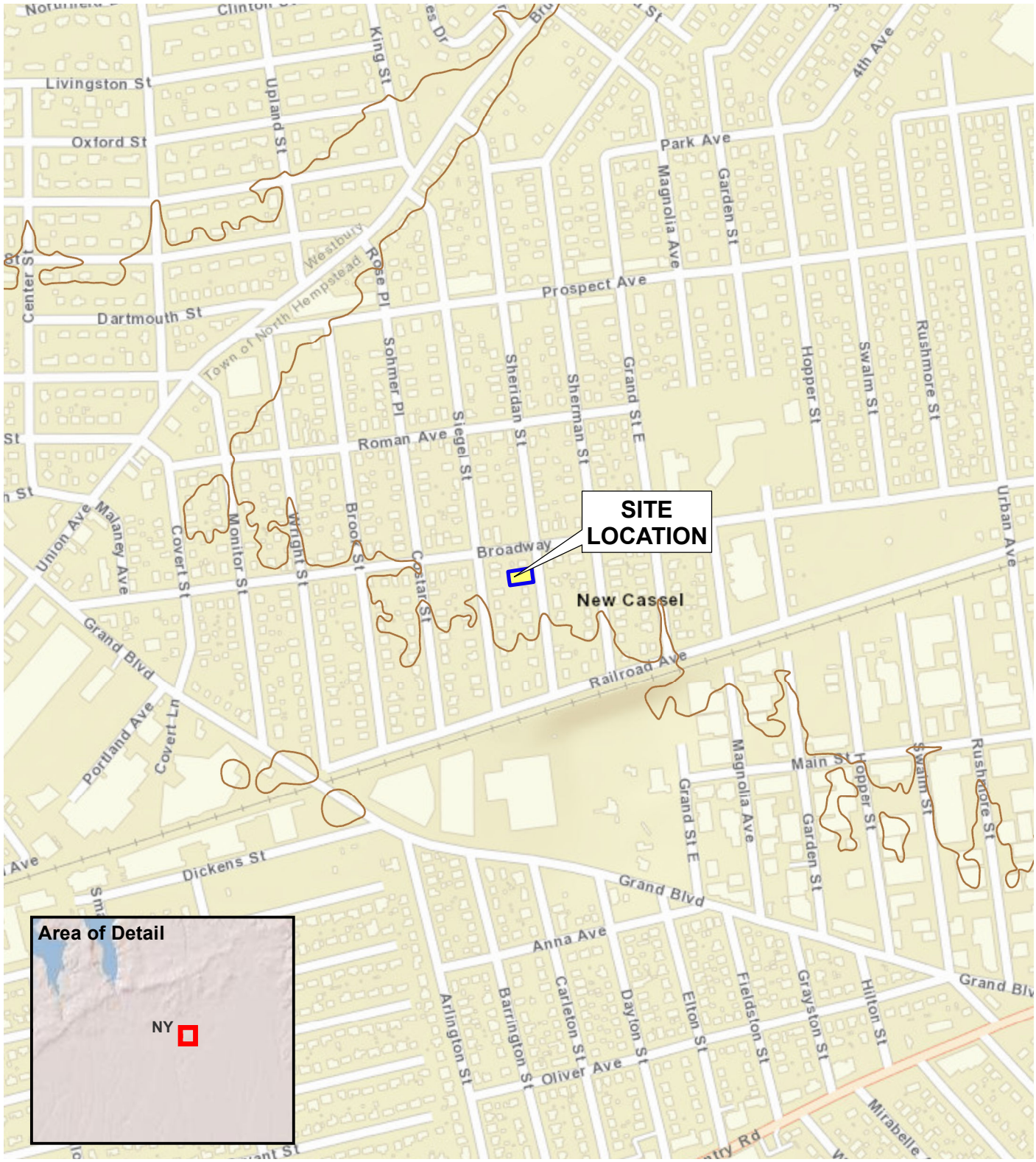
30.0 QC LIMITS TCL/PART 375 PESTICIDES

Analyte	CAS #	Aqueous Spike Limits		Soil Spike Limits		Aqueous RPD Limits (%)	Soil RPD Limits (%)
		Low Recovery Limit (%)	High Recovery Limit (%)	Low Recovery Limit (%)	High Recovery Limit (%)		
4,4'-DDD	72-54-8	59	125	64	132	30	30
4,4'-DDE	72-55-9	60	128	71	137	30	30
4,4'-DDT	50-29-3	42	136	55	138	30	30
Aldrin	309-00-2	58	125	67	130	30	30
alpha-BHC	319-84-6	65	122	72	132	30	30
beta-BHC	319-85-7	66	128	71	137	30	30
Chlordane (technical)	12789-03-6	45	120	65	122	30	30
cis-Chlordane	5103-71-9	57	123	63	136	30	30
delta-BHC	319-86-8	38	125	61	143	30	30
Dieldrin	60-57-1	57	133	66	135	30	30
Endosulfan I	959-98-8	56	124	64	135	30	30
Endosulfan II	33213-65-9	56	134	64	130	30	30
Endosulfan sulfate	1031-07-8	54	124	61	134	30	30
Endrin	72-20-8	57	135	63	136	30	30
Endrin aldehyde	7421-93-4	54	122	60	132	30	30
Endrin ketone	53494-70-5	51	132	48	150	30	30
gamma-BHC (Lindane)	58-89-9	65	123	70	134	30	30
Heptachlor	76-44-8	59	120	62	134	30	30
Heptachlor epoxide	1024-57-3	59	128	65	127	30	30
Methoxychlor	72-43-5	35	138	49	128	30	30
Toxaphene	8001-35-2	41	140	49	120	30	30
trans-Chlordane	5103-74-2	-	-	-	-	-	-

31.0 QC LIMITS TCL/PART 375 HERBICIDES

Analyte	CAS #	Aqueous Spike Limits		Soil Spike Limits		Aqueous RPD Limits (%)	Soil RPD Limits (%)
		Low Recovery Limit (%)	High Recovery Limit (%)	Low Recovery Limit (%)	High Recovery Limit (%)		
2,4,5-T	93-76-5	45	150	10	150	30	30
2,4-D	94-75-7	51	150	46	150	30	30
2,4,5-TP (Silvex)	93-72-1	50	150	41	150	30	30

FIGURES



Service Layer Credits: ESRI World Street Map, 2021.

0 600 1,200

SCALE IN FEET

AKRF
440 Park Avenue South, New York, NY 10016

243 Sheridan Street
New Cassel, New York

SITE LOCATION

DATE
11/22/2022

PROJECT NO.
200225

FIGURE
1

LEGEND

- PROJECT SITE BOUNDARY
- 74 — LOT BOUNDARY AND TAX LOT NUMBER
- **44** — BLOCK NUMBER
- ● — PROPOSED SOIL BORING LOCATION

Map Source:
http://www.nassaucountyny.gov/mynassauproperty/main.jsp.

Aerial Source:
ESRI World Imagery 2021.

0 30 60
SCALE IN FEET

AKRF
440 Park Avenue South, New York, NY 10016

243 Sheridan Street
New Cassel, New York

SITE AND PROPOSED SAMPLE LOCATION PLAN

DATE
11/22/2022

PROJECT NO.
200225

FIGURE
2

APPENDIX A

LABORATORY STANDARD OPERATING PROCEDURES

Title: Analysis of Organochlorine Herbicides by SW846 Method 8151A

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Catalina Dalangin	10/17/2022 Date	Dan Helfrich	10/17/2022 Date
GC Semivolatile Department Manager		Health & Safety Manager / Coordinator	
Carl Armbruster	10/17/2022 Date	Mark Acierno	10/17/2022 Date
Quality Assurance Manager		Laboratory Director	

Sylvanus Klusey 10/17/2022 Date
Operations Manager - Organics

1.0 Scope and Application

1.1 Analytes, Matrix(s), and Reporting Limits

This SOP describes Eurofins Edison's procedure for the analysis of select organochlorine herbicides in solid and aqueous matrices by USEPA SW846 Method 8151A using dual capillary column gas chromatography (GC) with electron capture detector (ECD). Water samples are prepared using the extraction techniques detailed in Eurofins Edison SOP No. ED-ORP-015, *Extraction of Organochlorine Herbicides in Water by SW846 Method 8151A,* current revision. Solid sample extraction procedures are detailed in Eurofins Edison SOP No. ED- ORP-023, *Extraction of Organochlorine Herbicides in Soil by SW846 Method 8151A,* current revision. Table 1 (below) lists the method target compounds as well as the analytical reporting limit for each compound by matrix.

Table 1: Analytes and Reporting Limits

Parameter	CAS No.	Soil Reporting Limits (ug/kg)	Water Reporting Limits (ug/L)	Leachate Reporting Limits (ug/L)	Waste Reporting Limits (ug/kg)
2,4-D	94-75-7	17	0.50	0.080	33
2,4,5-TP (Silvex)	93-72-1	17	0.50	0.080	33
2,4,5-T	93-76-5	17	0.50	0.080	33
Dalapon	75-99-0	17	0.50	n/a	33
Dinoseb	88-85-7	17	0.50	n/a	33
2,4-DB	94-82-6	17	0.50	n/a	33
DIcamba	1918-00-9	17	0.05	n/a	33
Dichlroprop	120-36-5	17	0.05	n/a	33
MCPA	94-74-6	1700	50.0	n/a	3300
MCPP	93-65-2	1700	50.0	n/a	3300
Pentachlorophenol	87-86-5	17	0.05	n/a	33
Picloram	1918-02-01	17	0.05	n/a	33

On occasion clients may request modifications to this SOP. These modifications are handled following the procedures outlined in Sections 9 (Review of Work) and 19 (Test Methods and Method Validation) in the Eurofins Edison Quality Assurance Manual.

2.0 Summary of Method

2.1. A measured volume or weight of sample (30 g for soil, 1000 ml for water, 15 ml for leachates) is extracted using the appropriate preparation SOP (see Section 1.1). The final volume is 10 ml in hexane (soil and water) and 5 ml in hexane (leachates).

2.2. Water samples are extracted with diethyl ether and then esterified with diazomethane. The derivatives are determined by gas chromatography with an electron capture detector (GC/ECD). The results are reported as acid equivalents.

2.3. Soil and waste samples are extracted and esterified with diazomethane. The derivatives are determined by gas chromatography with an electron capture detector (GC/ECD). The results are reported as acid equivalents.

2.4. Samples are analyzed after all the necessary calibration and QC checks have been performed as described herein. Since quantitative values for organochlorine herbicide analyses are based on the methyl ester derivatives of the free acid herbicides, the results must be adjusted for the difference in molecular weight between the free acid and the methyl ester. (See Attachment 1 for Conversion Factors)

2.5. All data undergoes two documented levels of review prior to release to clients.

3.0 Definitions

For a complete list of definitions refer to Appendix 5 in the most current revision of Eurofins Edison's Quality Assurance Manual (ED-QA-LQM).

4.0 Interferences

4.1. Method interferences may be caused by contaminants in solvents, reagents, glassware, and other sample processing hardware that lead to discrete artifacts or elevated baselines in gas chromatograms. All these materials must be routinely demonstrated to be free from interferences under the conditions of the analysis, by analyzing reagent blanks.

4.1.1. Glassware must be scrupulously cleaned. Clean each piece of glassware as soon as possible after use by rinsing it with the last solvent used in it. This should be followed by detergent washing with hot water and rinses with tap water, then with organic-free reagent water. Glassware should be solvent-rinsed with acetone and pesticide-quality hexane. After rinsing and drying, glassware should be sealed and stored in a clean environment to prevent any accumulation of dust or other contaminants. Store glassware inverted or capped with any accumulation of dust or other contaminants. Store glassware inverted or capped with aluminum foil. Immediately prior to use, glassware should be rinsed with the next solvent to be used. For complete details reference Eurofins Edison SOP No. ED-GEN-013, *Glassware Cleaning*, most current revision.

4.1.2. The use of high purity reagents and solvents helps to minimize interference problems. Purification of solvents by distillation in all-glass systems may be required.

4.2. Matrix interferences may be caused by contaminants that are coextracted from the sample. The extent of matrix interferences will vary considerably depending upon the nature and diversity of the material being analyzed.

4.3. Organic acids, especially chlorinated acids, cause the most direct interference with the determination by methylation. Phenols, including chlorophenols, may also interfere with this procedure. The determination using pentafluorobenzylation is more sensitive, and more prone to interferences from the presence of organic acids or phenols than by methylation.

4.4. Alkaline hydrolysis and subsequent extraction of the basic solution removes many chlorinated hydrocarbons and phthalate esters that might otherwise interfere with the electron capture analysis. However, hydrolysis may result in the loss of Dinoseb and the formation of aldol condensation products if any residual acetone remains from the extraction of solids. Strong organic acids react readily with alkaline substances and may be lost during analysis. Therefore, glassware must be acid-rinsed and then rinsed to constant pH with organic-free reagent water. Sodium sulfate must be acidified.

4.5. Organochlorine herbicides, being strong organic acids, react readily with alkaline substances and may be lost during analysis. Therefore, glassware must be acid-rinsed and then rinsed to constant pH with organic-free reagent water. Sodium sulfate must be acidified.

4.6. Sample extracts should be dry prior to methylation or poor recoveries may result.

5.0 Safety

Employees must abide by the policies and procedures in the Corporate Environmental Health and Safety Manual (CW-E-M-001) and this document. This procedure may involve hazardous material, operations and equipment. This SOP does not purport to address all of the safety problems associated with its use. It is the responsibility of the user of the method to follow appropriate safety, waste disposal and health practices under the assumption that all samples and reagents are potentially hazardous. Safety glasses, gloves, lab coats and closed-toe, nonabsorbent shoes are a minimum

5.1. Specific Safety Concerns or Requirements

The gas chromatograph contains zones that have elevated temperatures. The analyst needs to be aware of the locations of those zones, and must cool them to room temperature prior to working on them.

There are areas of high voltage in the gas chromatograph. Depending on the type of work involved, either turn the power to the instrument off, or disconnect it from its source of power.

5.2. Primary Materials Used

The following is a list of the materials used in this method, which have a serious or significant hazard rating. **Note: This list does not include all materials used in the method. The table contains a summary of the primary hazards listed in the MSDS for each of the materials listed in the table.** A complete list of materials used in the method can be found in the reagents and materials section. Employees must review the information in the MSDS for each material before using it for the first time or when there are major changes to the MSDS.

Material	Hazards	Exposure Limit (1)	Signs and symptoms of exposure
Acetone	Flammable	1000 ppm-TWA	Inhalation of vapors irritates the respiratory tract. May cause coughing, dizziness, dullness, and headache.
Hexane	Flammable Irritant	500 ppm-TWA	Inhalation of vapors irritates the respiratory tract. Overexposure may cause lightheadedness, nausea, headache, and blurred vision. Vapors may cause irritation to the skin and eyes.
1 – Exposure limit refers to the OSHA regulatory exposure limit.			

6.0 Equipment and Supplies

6.1. Instrumentation:

6.1.1. The system used is an Agilent Technologies (Avondale, PA) model 5890A or 6890 Gas Chromatograph (GC). It is equipped for simultaneous quantitation and confirmation columns using two separate detector channels on dual megabore capillary columns that are suitable for the analysis of organochlorine herbicides. All operations are as automated as possible with the equipment utilized.

6.1.2. Injection system: Dual tower Agilent Technologies 7673 Autosampler, or equivalent. Sample injection is accomplished by dual autoinjectors. A robot arm that shuttles samples between the sample tray and the injector turret services the auto injectors.

6.1.3. The samples are injected into a split/splitless injection port. The injection port is normally operated in splitless mode during injection with electronic pressure control (EPC) providing for constant flow during oven temperature programming.

6.1.4. Liners: The injection ports are each fitted with replaceable, heavy-walled glass liners. Each liner contains a plug of Hexane extracted, Teflon wool approximately 1 cm in length. The Teflon wool is positioned in the liner 1 cm from the bottom for the 5890 system. The Teflon wool is positioned in the middle of the goose neck of the siltek liner used in the 6890 system.

6.1.5. GC Oven: capable of integrated temperature control between 35^{O}C and 400^{O}C. Temperature programming of the gas chromatograph is employed.

6.1.6. Two dissimilar columns are used for analysis. An Agilent Technologies DB-5, 30m x 0.53mm ID column is used for sample quantitation. The secondary column is an Agilent Technologies DB-608, 30m x 0.53mm ID megabore column.

6.1.7. Detectors: The GC is equipped with dual Electron Capture Detectors (ECD), one for each column. Each detector is supplemented with make-up gas to provide sufficient detector flow for maintaining the electron

plasma. This is in addition to the gas exiting the column. The make-up gas consists of Ultra high purity (99.999%) Argon (95%)/Methane (5%) (a.k.a. P-5 Mixture) (P-5) and is fed from a supply separate from the carrier or injection port gas streams.

6.1.8. Data System:

6.1.8.1 **Chemstation:** The Chemstation is utilized for automation of runs and acquisition. The system is dedicated to a single GC and does not multitask. Therefore, data manipulation cannot be done while sample analysis is in progress. The data system acquires and stores all chromatographic data.

6.1.8.2 **Eurofins Chrom** chromatography data processing software package. Chrom is used for post acquisition GC data processing, reporting and storage and is fully integrated with Eurofins's LIMs (TALS).

6.1.9. Final processed data is uploaded to the Eurofins LIMS (TALS).

6.2. Supplies

6.2.1 4mm splitless sleeve (Restek Catalog # 11868-773 or equivalent) - This liner/glass wool combination provides many functions. The glass wool serves as a heat sink rapidly vaporizing solvent and samples resulting in higher response factors. The liner also protects the column head from accumulation of high boiling residuals and particulates.

6.2.2 0.8mm ID, Gold Inlet Seal (Restek Catalog # 21241 or 21318 or equivalent)

6.2.3 Snoop Leak Detector solution or equivalent.

6.2.4 Gas-tight syringes (various sizes)

6.2.5 Injection port septa

6.2.6 Pre-silanized glass wool (Supelco 2-0411 or equivalent)

6.2.7 Syringes, Assorted sizes 10ul - 1000ul; gas-tight

6.2.8 Bottles, 10 and 5ml amber screw cap with Teflon liner

6.2.9 Vials, 2ml amber screw cap with Teflon liner

6.2.10 Wheaton microvials 100ul or equivalent

7.0 Reagents and Standards

7. 1. Reagents

7.1.1. **Gases:** Ultra high purity (99.999%) Helium is used as the carrier and injection port purge gas. It is introduced to the GC at the injection port. Ultra high purity (99.999%) Argon (95%) / Methane (5%) (a.k.a. P-5 Mixture) is used as make-up gas. It is introduced to the GC via the make-up gas adapter at the end of the capillary column. They are supplied by M-G Industries (Valley Forge, PA). Both gases are supplied at tank pressures of 2000-2400 psig., for a 300 cft. tank. The tank pressure is regulated to an outlet pressure of 70 psig. Each tank is used until the tank pressure drops to less than 500 psig.

7.1.1.1. The gas streams are polished using three traps or filters before introduction to the GC The traps are as follows:

7.1.1.1.1. Hydrocarbon trap
7.1.1.1.2. H_2O trap
7.1.1.1.3. O_2 scrubber

7.1.2. All sample extracts are in hexane (reference the sample prep SOPs detailed in Section 2.0 above).

7.1.2.1. Hexane, Pesticide Grade, Baxter 217-4 or equivalent.

7.1.3. All solvents must be pesticide quality or equivalent. Each lot of solvent is screened for contaminants before being used for analysis as detailed in Eurofins Corporate Quality SOP No. CA-Q-S-001 (*Solvent & Acid Lot Testing & Approval*) and Eurofins Edison SOP No. ED-GEN-023 (*Bulk Solvent Testing and Approval*).

7. 2. Standards

7.2.1. Standards are purchased as concentrated solutions (see Section 7.2.2). Most stock solutions are diluted (in volumetric glassware) to working concentration using hexane as the diluent as described in Section 7.3.

7.2.2. **Standard mixes and sources:**

Table 2:
Herbicide Standard Mixes and Sources*

Standard Name ("Lab Name")	Concentration (ug/ml)	Source
Acid Herbicide Spiking Mix 2 ("Spiking Mix")	varies (in acetone)	Supelco Catalog # 861259
Underivatized Chlorinated Herbicides ("10 Compound Spiking Mix")	Varies (in methanol)	Accustandard Catalog #M-8150A)
EPA 8270 Herbicide Ester Mix ("Calibration Mix")	2000 (in hexane)	Supelco Catalog # 48474
Pentachloroanisole ("Pentachloroanisole")	100 (in methanol)	Accustandard Catalog # P-199S
Picloram methyl ester ("Picloram methyl ester")	Neat	Chem Service Catalog # F2155
Methyl Derivatives of Chlorinated Herbicides ("Calibration Mix - 10 compound")	Varies (in methanol)	AccuStandard Catalog # M-8150
Pentachlorophenol ("Pentachlorophenol spike")	1000	Spex Certiprep Catalog #

Table 2: Herbicide Standard Mixes and Sources*		
Standard Name ("Lab Name")	**Concentration (ug/ml)**	**Source**
		S-2950
Picloram (4-amino-3,5,6-trichloropicolinic acid) ("Picloram Acid Spiking Standard")	Neat	Chem Service. Catalog No. F2041
2,4-DCAA Acid Spike Mix ("Surrogate Spiking Solution")	100 (in acetone)	Supelco Catalog # 861271
2,4-DCAA methyl ester ("Surrogate Solution")	1000 (in hexane)	Supelco Catalog # 861272

* May be substituted with equivalent standards from alternate sources.

Table 3: Components of Herbicide Standard Mixes			
Parameter	**Catalog Nos.**	**Lab Standard Name**	**Concentration (ug/ml)**
2,4-dichlorophenoxyacetic acid (2,4-D)	Supelco 861259	Spiking Mix	50
2-(2,4,5-Trichlorophenoxy)propionic acid (2,4,5-TP aka Silvex)	Supelco 861259	Spiking Mix	20
2,4,5-Trichlorophenoxyacetic acid (2,4,5-T)	Supelco 861259	Spiking Mix	20
2,4-D methyl ester	Supelco 48474	Calibration Mix	2000
2,4,5-T methyl ester	Supelco 48474	Calibration Mix	2000
Silvex methyl ester	Supelco 48474	Calibration Mix	2000
2,4-D methyl ester	Accustandard M-8150	Calibration Mix – 10 compound	100
Dalapon methyl ester	Accustandard M-8150	Calibration Mix – 10 compound	100
2,4-DB methyl ester	Accustandard M-8150	Calibration Mix – 10 compound	100
Dicamba methyl ester	Accustandard M-8150	Calibration Mix – 10 compound	100
Dichloroprop methyl ester	Accustandard M-8150	Calibration Mix – 10 compound	100
Dinoseb methyl ester	Accustandard M-8150	Calibration Mix – 10 compound	100
MCPA methyl ester	Accustandard M-8150	Calibration Mix – 10 compound	10000
MCPP methyl ester	Accustandard M-8150	Calibration Mix – 10 compound	10000
2,4,5-T methyl ester	Accustandard M-8150	Calibration Mix – 10 compound	100
2,4,5-TP methyl ester	Accustandard M-8150	Calibration Mix – 10 compound	100
2,4-D	Accustandard M-8150A	Spiking Mix – 10 compound	100
2,4-DB	Accustandard M-8150A	Spiking Mix – 10 compound	100
2,4,5-T	Accustandard M-8150A	Spiking Mix – 10 compound	100
Silvex	Accustandard M-8150A	Spiking Mix – 10 compound	100

Table 3:
Components of Herbicide Standard Mixes

Parameter	Catalog Nos.	Lab Standard Name	Concentration (ug/ml)
Dalapon	Accustandard M-8150A	Spiking Mix – 10 compound	100
Dicamba	Accustandard M-8150A	Spiking Mix – 10 compound	100
Dichloroprop	Accustandard M-8150A	Spiking Mix – 10 compound	100
Dinoseb	Accustandard M-8150A	Spiking Mix – 10 compound	100
MCPA acid	Accustandard M-8150A	Spiking Mix – 10 compound	10000
MCPP acid	Accustandard M-8150A	Spiking Mix – 10 compound	100000
Pentachloroanisole	Accustandard P-199S	Pentachloroanisole	100
Pentachlorophenol	Spex Certiprep S-2960	Pentachlorophenol spike	1000
Picloram methyl ester	Chem Service Catalog # F2155	Picloram methyl ester	neat
Picloram (4-amino-3,5,6-trichloropicolinic acid)	Chem Service. Catalog No. F2041	Picloram Acid Spiking Standard	neat
2,4-Dichlorophenylacetic Acid	Supelco 861271	Surrogate Spiking Solution	100
2,4-Dichlorophenylacetic Acid methyl ester	Supelco 861272	Surrogate Solution	1000

7. 3. Standards Preparation

7.3.1. All standard stock solutions are diluted to the working concentrations with hexane or acetone (as indicated) using Class A volumetric glassware.

7.3.2. Total Herbicide Calibration Standards:

7.3.2.1 Total Herbicide Calibration Standards (10 compound analysis): The ten (10) compound/surrogate total herbicide calibration standard range is prepared when analyzing for the expanded list of herbicides. Table 4 details the preparation of the five calibration concentration levels using the 10 compound calibration mix. The standards are prepared using a methyl ester source with the final concentration adjusted to the free acid equivalent concentration using the conversion factors detailed in Attachment 1.

Table 4:
Preparation of Total Herbicide Calibration Standards (10 compound analysis)

Parameter	Level 1 Conc. (ug/ml) [1] 25.0 ul '8151-Herbicide Mix (10 compounds)' & 5ul 'Surrogate Solution" into 50ml Hexane	Level 2 Conc. (ug/ml) [1] 125 ul '8151-Herbicide Mix (10 compounds)' & 25ul 'Surrogate Solution" into 50ml Hexane	Level 3 Conc. (ug/ml) [1] 375 ul '8151-Herbicide Mix (10 compounds)' & 75ul 'Surrogate Solution" into 50ml Hexane	Level 4 Conc. (ug/ml) [1] 750 ul '8151-Herbicide Mix (10 compounds)' & 150ul 'Surrogate Solution" into 50ml Hexane	Level 5 Conc. (ug/ml) [1] 750 ul '8151-Herbicide Mix (10 compounds)' & 150ul 'Surrogate Solution" into 25ml Hexane
2,4-D methyl ester	0.047	0.235	0.705	1.41	2.82
2,4,5-T methyl ester	0.047	0.235	0.705	1.41	2.82
2,4,5-TP (Silvex) methyl ester	0.048	0.238	0.712	1.42	2.85
Dalapon methyl ester	0.046	0.228	0 .682	1.36	2.73
2,4-DB methyl ester	0.047	0.235	0.705	1.41	2.82
Dicamba methyl ester	0.047	0.235	0.705	1.41	2.82
Dichloroprop methyl ester	0.047	0.235	0.705	1.41	2.82
Dinoseb methyl ester	0.047	0.235	0.705	1.41	2.82
MCPA methyl ester	4.67	23.4	70.1	140	280
MCPP methyl ester	4.67	23.4	70.1	140	280
2,4-DCAA methyl ester (surr)	0.096	0.478	1.43	2.86	5.73

[1] The calculated final standard concentrations have been converted from the methyl ester concentration to the free acid equivalent concentration using the factors found in Attachment 1

7.3.2.2 Pentachlorophenol Calibration Standards: A pentachlorophenol /surrogate total herbicide calibration standard range is prepared when analysis is required for this compound. Table 5 details the preparation of the five calibration concentration levels. The standards are prepared using a source containing the derivatized form of the compound (pentachloroanisole) the final concentration adjusted to the free acid equivalent concentration using the conversion factors detailed in Attachment 1.

Table 5:
Preparation of Pentachlorophenol Calibration Standards

Parameter	Level 1 Conc. (ug/ml) [1] 50 ul 'Pentachloroanisole' & 10ul 'Surrogate Solution" into 100ml Hexane	Level 2 Conc. (ug/ml) [1] 25 ul 'Pentachloroanisole' & 5ul 'Surrogate Solution" into 10ml Hexane	Level 3 Conc. (ug/ml) [1] 75 ul 'Pentachloroanisole' & 15ul 'Surrogate Solution" into 10ml Hexane	Level 4 Conc. (ug/ml) [1] 150 ul 'Pentachloroanisole' & 30ul 'Surrogate Solution" into 10ml Hexane	Level 5 Conc. (ug/ml) [1] 300 ul 'Pentachloroanisole' & 60ul 'Surrogate Solution" into 10ml Hexane
Pentachlorophenol	0.048	0.237	0.712	1.42	2.85
2,4-DCAA methyl ester (surr)	0.096	0.478	1.43	2.87	5.73

[1] The calculated final standard concentrations have been converted from the deriviatized concentration to the free acid equivalent concentration using the factors found in Attachment 1

7.3.2.3 Picloram Methyl Ester Calibration Standards: A Picloram methyl ester/surrogate calibration standard range is prepared when analysis is required for this compound. Table 6 details the preparation of the five calibration concentration levels. The standards are prepared using a source containing the picloram methyl ester and the final concentration is adjusted to the free acid equivalent concentration using the conversion factors detailed in Attachment 1.

7.3.2.3.1 Prior to the preparation of the picloram calibration standards a working stock solution must be prepared using the neat material described in Table 2. This working stock solution is prepared at 9000 ug/ml by dissolving 9.00 grams of neat picloram methyl ester into 1000 ml of hexane using class A volumetric glassware. This solution is used as described in Table 6.

Table 6:
Preparation of Picloram Calibration Standards

Parameter	Level 1 Conc. (ug/ml) [1] 20 ml of Level 3 Picloram methyl ester into 100ml Hexane	Level 2 Conc. (ug/ml) [1] 33 ml of Level 3 Picloram Methyl ester into 100ml Hexane	Level 3 Conc. (ug/ml) [1] 8.5 ul Picloram Methyl ester stock (9000ug/ml) & 150 ul 'Surrogate Solution" into 100ml Hexane	Level 4 Conc. (ug/ml) [1] 17 ul Picloram Methyl ester stock (9000ug/ml) & 300 ul 'Surrogate Solution" into 100ml Hexane	Level 5 Conc. (ug/ml) [1] 34 ul Picloram Methyl ester stock (9000ug/ml) & 600 ul 'Surrogate Solution" into 100ml Hexane
Picloram	0.047	0.236	0.709	1.42	2.84
2,4-DCAA methyl ester (surr)	0.095	0.477	1.43	2.86	5.73

[1] The calculated final standard concentrations have been converted from the deriviatized concentration to the free acid equivalent concentration using the factors found in Attachment 1

7.3.3. Herbicide Spiking Standard (LCS/MS/MSD):

7.3.3.1 Herbicide QC Spiking Standard Mix (10 compound analysis): The ten (10) compound spiking standard is prepared and used when analyzing for an expanded list of herbicides. The 10 compound spiking mix is prepared using volumetric glassware as detailed in Table 7 (below) using a concentrated solution of free acid herbicides (Accustandard Catalog # M-1850A; see Tables 1 and 2):

Table 7: Preparation of Total Herbicide 10 Compound Spiking Mix	
Parameter	**Final Conc. (ug/ml)** 5.0 ml 'Spiking Mix -10 compounds)' into 10ml Acetone
2,4-D	50
2,4,5-T	50
2,4,5-TP (Silvex)	50
Dalapon	50
2,4-DB	50
Dicamba	50
Dichloroprop	50
Dinoseb	50
MCPA	5000
MCPP	5000

The concentrate is used for spiking of the following quality control sample types: MS, MSD, and LCS. For spiking instructions see the appropriate herbicide prep SOP referenced in Section 1.1.

7.3.3.2 **Pentachlorophenol QC Spiking Standard:** The pentachlorophenol spiking standard is prepared and used when analyzing for this herbicide. The pentachlorophenol spiking mix is prepared using volumetric glassware by diluting 1ml of the concentrated pentachlorophenol solution Spex # S-2950; see Tables 1 and 2) into a final volume of 10ml acetone. The concentrate is used for spiking of the following quality control sample types: MS, MSD, and LCS. For spiking instructions see the appropriate herbicide prep SOP referenced in Section 1.1.

7.3.3.3 **Picloram Acid QC Spiking Standard:** The picloram acid spiking standard is prepared and used when analyzing for this herbicide. The standard is prepared using a source containing the picloram free acid. Prior to the preparation of the picloram acid spiking standard a working stock solution must be prepared using the neat material described in Table 2. This working stock solution is prepared at 10000 ug/ml by dissolving 10.00 grams of neat picloram free acid into 1000 ml of hexane using class A volumetric glassware

7.3.3.3.1 Prepare the 5000 ug/ml picloram spiking solution by diluting 500ml of the working stock solution (see Sec. 7.3.3.4) into 1000 ml of hexane using class A volumetric glassware. For spiking instructions see the appropriate herbicide prep SOP referenced in Section 1.1

7.3.4. **Herbicide Surrogate Spiking Standard (**2,4-DCAA Acid Spike Mix**):** The herbicide surrogate spiking solution is a concentrated 10 ml solution of 2,4-DCAA at a concentration of 100 ug/ml. (Supelco Catalog # 861271, see Tables 1 and 2). The concentrate is used without further dilution prior to the spiking of samples.

7.3.5. **Initial Calibration Verification Standard:** The ICV is prepared as detailed in Section 7.3.2 (dependent upon the compound being analyzed) and must be from a separate lot or separate source from the standards used in the Initial Calibration Range (the analyst must confirm and document that a separate source or lot is used).

8.0 Sample Collection, Preservation, Shipment and Storage

Sample container, preservation techniques and holding times may vary and are dependent on sample matrix, method of choice, regulatory compliance, and/or specific contract or client requests. Listed below are the holding times and the references that include preservation requirements.

Matrix	Sample Container	Min. Sample Size	Preservation	Holding Time	Reference
Waters	Glass	1L amber	Cool 4 ± 2ºC	7 days to extraction; 40 days to analysis	USEPA SW846
Soil/Sediment	Glass	4 oz wide mouth	Cool 4 ± 2ºC	14 Days to extraction; 40 days to analysis	USEPA SW846

8.1. Extracts must be stored under refrigeration (Cool 4 ± 2ºC) in the dark and analyzed within 40 days of extraction.

9.0 Quality Control

9.1 **Sample QC** - The following quality control samples are prepared with each batch of samples.

Quality Controls	Frequency	Control Limit
Method Blank (MB)	1 in 20 or fewer samples	< Rpt. Limit
Laboratory Control Sample (LCS)[1]	1 in 20 or fewer samples	Statistical Limits [4]
Matrix Spike (MS)[2]	1 in 20 or fewer samples	Statistical Limits [4]
MS Duplicate (MSD)[2]	1 in 20 or fewer samples	Statistical Limits [4]
Surrogates	every sample[3]	Statistical Limits [4]

[1] LCS Duplicate (LCD) is performed only when insufficient sample is available for the MS/MSD or when requested by the client/project/contract.
[2] The sample selection for MS/MSD is randomly selected by the organic prep lab, unless specifically requested by a client.
[3] Analytical and QC samples (MB, LCS, MS/MSD)
[4] Statistical control limits are updated annually and are updated into TALS (LIMS).

9.1.1. Method Blanks are extracted with each sample batch on each day that samples are extracted. The analytical results for the method blank must fall below the reporting limit for each compound of interest. If a target compound is detected in the blank at a concentration higher than the reporting limit, first the extract is reanalyzed for confirmation. If results are still outside of limits the entire batch of samples extracted with the affected blank must be re-extracted and reanalyzed.

9.1.2. Laboratory Control Sample (LCS): A Laboratory Control Sample (LCS) or blank spike must be extracted and analyzed for with each batch of 20 environmental samples. The LCS data is used to ensure that the analytical system is in control. It is also used to assess performance if the MS/MSD recoveries fall outside of established limits. The recoveries of the LCS must fall within lab generated acceptance criteria. If the spiked sample recovery results fall outside the laboratory generated limits (refer to the current active TALS method limit group database), the LCS recovery is evaluated. If LCS recovery is within limits the poor sample recovery results are attributed to matrix interference. If the LCS recovery results are outside QC limits, first the extract is reanalyzed and if it is still outside the limits the entire QC batch must be re-extracted and reanalyzed.

9.1.3. Matrix Spike/Matrix Spike Duplicate (MS/MSD): A Matrix Spike/Matrix Spike Duplicate (MS/MSD) pair is extracted and analyzed with every batch of 20 environmental samples. MS/MSD recoveries are evaluated against lab generated limits (refer to refer to the current active TALS method limit group database). If the MS/MSD recovery limits fall outside of lab limits the LCS recovery is evaluated and corrective action is taken as described in 9.1.2.

9.1.4. Surrogate Standards: All samples, blanks and QC samples are spiked with surrogate standard solution containing 2,4-DCAA (see Section 7.3.4). The percent recovery of the surrogate standards is calculated and compared to lab generated limits (refer to refer to the current active TALS method limit group database). If the surrogate recovery limits are outside of acceptance limits the sample extract is reanalyzed to confirm. If the recoveries are still outside of limits the sample must be re-extracted and reanalyzed or the data flagged as “estimated concentration”.

9.2 Instrument QC

9.2.1. Initial Calibration Range and Initial Calibration Verification (ICV)

9.2.1.1. ***Initial Calibration Range*:** A five-point calibration range is analyzed for each target compound using herbicide methyl ester standard mixes. Standards are prepared following the instructions in Section 7.3.

9.2.1.2. ***Initial Calibration Verification (ICV):*** An Initial Calibration Verification (ICV) standard is analyzed immediately after the Initial Calibration Range and before any samples are analyzed.

The ICV is prepared as detailed in Section 7.3.2 (dependent upon the compound being analyzed) and must be from a separate lot or separate source from the standards used in the Initial Calibration Range (the analyst must confirm and document that a separate source or lot is used).

9.2.2. Continuing Calibration Verification (CCV): A mid-point Continuing Calibration Verification (CCV) must be analyzed every 12-hours or 20 samples (whichever is more frequent) and at the end of each analytical sequence. The CCV consists of the Level 3 calibration standard (~700 ppb) detailed in Section 7.3

9.2.3. Calibration Acceptance Summary

9.2.3.1. Retention Time Windows: Retention time windows must be established to compensate for minor shifts in absolute retention times as a result of sample loading and normal chromatographic variability. All gas chromatographs used for pesticides analysis at Eurofins Edison are equipped with electronic pressure control (EPC). The use of EPC results in little retention time variability between analyses. Accordingly, retention time variability for the purpose of retention time window determination for standards analysis is extremely small. The default retention time window option must therefore be employed as follows to accommodate the excellent precision of EPC equipped systems.

9.2.3.1.1 Obtain the retention time for all single component compounds from the analysis of the midpoint standard for the calibration curve. Establish the center of the retention time window for each analyte and surrogate by using the absolute retention time for each analyte and surrogate from the calibration verification standard at the beginning of the analytical shift. For samples run during the same shift as an initial calibration, use the retention time of the mid-point standard of the initial calibration. Apply the retention time window data in the table in Attachment 2 to its corresponding compound (ex., mid-point RT ±0.10 minute). Calculate absolute retention time windows for each analyte and surrogate on each chromatographic column and instrument.

9.2.3.1.2 New retention time windows must be established when whenever a chromatographic column is replaced or a new detector is installed .Whenever the observed retention time of each analyte and surrogate is outside of the established retention time window, the analyst is advised to determine the cause and correct the problem before continuing analyses.

9.2.3.2. Initial Calibration Range: External standard calibration is employed for this method. The response factor (defined as the

ratio of the area to the standard concentration) is calculated for each analyte at each calibration concentration. The average response factor may be used for quantitation if the % RSD across the 5 point range is <20%. Alternatively, linear regression may be used if the correlation coefficient (r1) is ≥ 0.990 (note: if the linear regression model is used the curve must NOT be forced through the origin). Calibration is checked every 12 hours or after every twenty (20) samples, whichever comes first, by injecting a calibration verification standard for all single response pesticide standards.

9.2.3.2.1 Calculate the percent Relative Standard Deviation of the response factors for each compound at each level:

% RSD = (RF Standard Deviation/RF Mean) X 100

RF = Response Factor

9.2.3.2.2 If the % RSD across the 5 point range is <20% the calibration can be assumed to be linear and the average response factor can be used to calculate concentrations of target compounds in samples.

9.2.3.2.3 If the % RSD is >20% for any given compound, a first order linear regression can be applied to the data to calculate the calibration curve and determine sample concentration. If this method is employed, the r^1 (Correlation Coefficient) value must be ≥ 0.990 for the calibration to be acceptable. Calibration is checked every 12 hours or after every twenty (20) samples, whichever comes first, by injecting a calibration verification standard for all single component pesticide standards.

9.2.3.3. Relative Standard Error: The percent relative standard error (% RSE) from the calibration curve is used to evaluate the initial calibration. This provides a measure of how much error is associated with using the calibration curve for quantitation. The average Relative Standard Error (RSD for average response factors) of the calibration points from the curve used must be ≤ 20%.

Some data systems will not measure the %RSE from a linear or quadratic fit. For the linear case, the correlation coefficient may be used as an alternative to the %RSE, and must be greater than or equal to 0.990. For the quadratic case the Coefficient of Determination may be used, and must be greater or equal to 0.990.

Note: The Relative Standard Error (RSE) is superior to the Correlation Coefficient (r) and Coefficient of Determination (r_2) for testing the fit of a set of calibration points to a line. The lower points on a curve have little effect on r. As a result, a curve may have a very good correlation coefficient (>0.990) while also having > 100% error at the low point

9.2.3.4. Initial Calibration Verification (ICV): An ICV will consist of a second source standard at or near the midpoint of the Initial Calibration Range analyzed immediately after an initial calibration range. The calculated concentration of the ICV must be within ±15%D of the expected concentration. Should the %D exceed 15% the analyst should take corrective action (check standard solution, perform instrument maintenance, etc.) and re-inject the ICV. If the %D still exceeds 15% after a single ICV reinjection, a new Initial Calibration Range must be analyzed.

9.2.3.5. Continuing Calibration Verification (CCV): A CCV will consist of an ICAL standard at or near the midpoint of the Initial Calibration Range analyzed after each 10 samples (every 12 hours at minimum). The calculated concentration of the CCV must be within ±15%D of the expected concentration. Should the %D exceed 15% the analyst should take corrective action (check standard solution, perform instrument maintenance, etc.) and re-inject the CCV. If the %D still exceeds 15% after a single CCV reinjection, a new Initial Calibration Range must be analyzed.

Step	*Standards*	*Type*	*Control Limit*	*Frequency*
Method # 8151A Instrument QC Summary				
Initial Calibration	*5 point*	*Average response factor or 1st order linear regression*	*For average RF: <20%RSD all analytes. For linear regression: r ≥0.990 or <20%RSE*	*As required when ICV or CCV do not meet requirements*
ICV	*Separate source (midpoint)*	*Average*	*± 15%D*	*After every initial calibration*
CCV	*ICAL source (midpoint)*	*Average*	*± 15%D*	*1 in 10 or fewer samples (every 12 hours minimum)*

10.0 Procedure

10.1. Gas Chromatograph Operation

10.1.1. The sequence of events for GC analysis involves many steps. First the injection system and column performance and calibration must be verified.

Maintenance operations are performed as needed. Then samples must be run on the instrument. Chromatograms and reports must be evaluated for content, integration and concentration. Re-runs and dilutions must be made based on the calibrations that were in effect at the time the sample was run. Lastly, a detailed analysis and calculations must be performed to determine the concentration of all the parameters for which the sample was analyzed.

10.1.2. Injection System: A split/splitless injection port with electronic pressure control (EPC) is used. Thirty seconds after sample injection, the purge valve is turned on to facilitate the sweeping of any remaining residual solvent/sample from the injection port. The EPC is used in constant flow mode.

10.1.2.1. For herbicide analysis the normal operating conditions of the injection port are as follows:

Injection port Temperature:	275^{o}C
Temperature Rate:	10 deg C/minute
Inlet pressure	4.0 psi
Initial Temperature:	55 deg C
EPC:	Constant flow
Detector temperature	300C

10.1.2.1.1 For pentachlorophenol analysis the following injection port settings are used:

Injection port Temperature:	250^{o}C
Temperature Rate:	4 deg C/minute
Inlet pressure	7.9 psi
Initial Temperature:	190 deg C
EPC:	Constant flow
Detector temperature	300C

10.1.2.2. In addition to the EPC, the injection port is also equipped with a siltek-coated glass double goose neck liner that contains a 1 cm glass wool plug. The plug of glass wool is located in the liner between the double goose neck.

10.1.2.3. This liner/glass wool combination provides many functions. The glass wool serves as a heat sink rapidly vaporizing solvent and samples resulting in higher response factors. The liner also protects the column head from accumulation of high boiling residuals and particulates.

10.1.2.4. Regular maintenance is performed on the injection port. When the glass wool/liner is changed, the septa also must be

changed. Injection port, oven and detector temperatures are lowered to ambient prior to "cracking" the system. This is so as to introduce a minimum of damaging oxygen molecules into the system.

10.1.2.5. After the system has cooled, the old liner is removed. The injection port should be checked for particulate residues and cleaned as needed. A flashlight is usually required for this. After a new liner has been prepared it is placed into the injection port. A graphite seal is placed around the liner. The edges of the seal must be flat, not knife-edged, and free of nicks or burrs. If any of these conditions are not met, the graphite seal must be replaced as well. The graphite seal is critical to proper operation of the injection port. If in doubt, replace it.

10.1.2.6. The locking ring on the top of the injection port should be turned, with the wrench, about 1/8 turn past finger tight. The septum nut should never be tightened more than finger tight. After the injection port is reassembled, all column nuts inside the oven should be checked for leaks using Snoop (Supelco) or another suitable leak tester.

10.1.2.7. Once the signal from both detectors has stabilized, it is time to re-heat the zones. The zones should be heated in the order of detectors, oven and then injectors. This is to ensure that volatilized contaminants do not condense on the column or detector.

10.1.3. Oven: With the megabore columns installed, temperature programming is employed to achieve higher resolution of compounds and shorter run times than could be accomplished using isothermal methods.

10.1.3.1. The standard oven program for total herbicide analysis is :

Initial Temp	Time	Rate	Final Temp	Time
55°C	3.0 min	10°/min	244°C	11.5 min

10.1.3.2. The standard oven program for TCLP herbicide analysis is :

Initial Temp	Time	Rate	Final Temp	Time
190°C	3.0 min	4.0°/min	238°C	0.00 min

10.1.4. Detectors: Detectors operate at 300°C and need to be supplied with 40-60 ml/min total flow. They are essentially maintenance free on a day-to-day basis. They are routinely baked out at 325°C to remove persistent contaminants. On occasion the detectors may be baked out at a higher temperature to remove contaminants with an extremely high boiling point (CAUTION: Do not exceed the maximum detector temperature of 380°C).

10.1.4.1. If the detectors are particularly contaminated, they must be sent to Agilent Technologies in Avondale, Pennsylvania for reconditioning. This should occur if the detector baseline is greater than 100 Hz. Detector reconditioning should be required at a maximum of biannually.

10.1.5. Chemstation: The HP Chemstation application is used for automation of runs and acquisition. The system is dedicated to a single GC and does not multitask. Therefore, data manipulation cannot be done while sample analysis is in progress. The data system acquires and stores all chromatographic data.

10.1.6. Eurofins's Chrom chromatography data processing application is utilized for the processing of ChemStation chromatography data files. Calibrations, verification standards and samples are processed using this database. All of the processed files are imported into the TALS database. Data is processed and first level reviewed by the analysts. Senior analysts or managers do the second level review and update the status of the job to 'Lab Complete' in TALS. All client reports are generated in TALS.

10.1.7. Eurofins LIMS (TALS): All data files processed in Chrom are then imported to into TALS. The data files are reprocessed in TALS after linking the appropriate reagents and standards to the batch. All samples and quality control runs are reviewed. A TALS non-conformance memo (NCM) is written for any QC outlier or situation requiring additional documentation. Samples and QC results are assigned to primary/secondary column as per reporting requirements. Files that are not reported are marked rejected. All data files that are being reported are first level reviewed in TALS by the analyst. Second level TALS review is done by a trained peer or supervisor. Once second level review is completed in TALS reports are generated and reviewed after which the job is marked complete.

10.2. Analytical Sequences

10.2.1. Calibration Ranges:

10.2.1.1 Herbicides are calibrated using a five-point calibration range. Standards are prepared as detailed in Section 7.3 and analyzed under the instrumental conditions detailed in Section 10.1 as appropriate for total or TCLP herbicide analysis.

10.2.1.2 A calibration factor, defined as the ratio of the response to the amount injected, is calculated for each analyte at each standard concentration. The average calibration factor (if the %RSD across the five-point range is <20%) or a linear calibration not through the origin (if the correlation coefficient (r1) is $\geq$ 0.99) is used for quantitation. Calibration is

checked every 12 hours or after every ten (10) samples, whichever comes first, by injection a calibration check standard.

10.2.1.3 Generating a calibration table: After the five calibration standards have been run, the data files are copied over to the Chrom data system. A new Chrom batch is created and the method file from the previous Chrom batch is copied to the new Chrom batch.

10.2.1.4 The data files representing the five levels of calibration are also copied to the new Chrom batch and processed with the method file. The integration of the five levels is checked for consistency in Chrom Review.

10.2.1.5 Linear Calibration. Check the percent relative standard deviation (% RSD) of the calibration factors for each individual analyte. If the % RSD is less than 20% over its working range, the linearity of the range is assumed. The average value of the calibration factors is used for quantitation of all the samples and continuing standards.

10.2.1.6 Linear Calibration Using Least Squares Regression. If the % RSD is >20% for any given compound, a first order linear regression can be applied to the data to calculate the calibration curve and determine sample concentration. If this method is employed, the r^1 (Correlation Coefficient) value must be $\geq$ 0.99 for the calibration to be acceptable.

10.2.1.7 Since the calibration is performed using standards made from methyl ester compounds (compounds not esterified by application of this method), then the calculation of free acid herbicide concentrations must include a correction for the molecular weight of the methyl ester versus the acid herbicide (see Attachment 1).

10.2.2. Calibration Check Standards:

10.2.2.1. **Initial Calibration Verification** (ICV) is analyzed and evaluated immediately after the initial calibration range as detailed in Sections 7.3 (standard prep) and 9.2 (instrument QC).

10.2.2.2. **Continuing Calibration Verification** (CCV): is analyzed and analyzed after each 10 sample injections as further detailed in Sections 7.3 (standard prep) and 9.2 (instrument QC). The CCV is also analyzed at the end of each analytical sequence as a closing calibration check standard.

10.2.2.3. Note that samples are always quantified against the average calibration factor from the initial calibration range (or from the initial calibration curve). Calibration check standards serve only

to confirm the validity of the initial calibration range and are never used to quantify sample concentrations.

Calibration Controls	Sequence	Control Limit
Calibration Standards	5-point (minimum) linearity	≤20% RSD
Cont. Cal. Verif. (CCV)	Prior to / after every 10 injections	Prior to/after every 10 injections
RT Windows (RTW)	Init. CCV determines midpt. of RTW	±3X SD

10.2.3. Idealized Analytical Sequence

10.2.3.1. Analysis Sequence: The automation of the GC runs is accomplished via the "SEQUENCE" macro of the Chemstation.

10.2.3.2. The Sequence File: The sequence file contains the name of Method file corresponding to the type of analysis to be performed, the range of samples to be run, and the number of injections per bottle

10.2.3.3. It is common practice to evaluate the instrument status, and then complete the Sample Table and Sequence File. If everything else is complete, the run is initiated using the START SEQUENCE soft-key of the SEQUENCE macro.

10.2.3.4. An idealized analytical sequence is presented below:

Ideal Analysis Sequence*
Instrument Blank (solvent injection)
Initial Calibration Range Standards (5)
ICV
CCV
Method Blanks and LCS
Client Samples (10)
CCV
Client Samples (10)
CCV*

*After the analysis of ten sample injections or 12 hours of analysis, a CCV must be successfully analyzed.

10.3. Documentation

10.3.1. After samples have been run, each standard and sample must be entered into the Instrument Run Log. The Instrument Run Log should contain the following information: run date, data file name, vial position, sample number, initial volume/weight, final volume, dilution factor, method, job

number, QC Batch number, extraction date, lab prep batch, Chrom batch, signature of analyst at the bottom of each page, lot numbers for standards used, and result of run (e.g., 'O.K'., 're-run at dilution', 'non-inject', etc.).

10.4. Dual Column Approach

10.4.1. The laboratory designates the rear column as the primary column and the front column as the secondary column. If the difference between the dual columns results in ≤40% RPD report the higher concentration.

10.4.2. The values are calculated from the chromatographic peaks that fall within the daily retention time windows established from the most recent preceding calibration verification.

10.4.3. If the calculated values are greater than 40% RPD of each other, report the lower concentration regardless of whether that result is from the primary or secondary column. Report the result with a flag of P.

10.4.4. If the surrogates on one column are very different (>40% RPD) compared to the other column, this may be indicative of a bad injection or column blockage. The sample should be reanalyzed. If similar results are obtained following reanalysis, report the lower of the two numbers and describe the circumstances in the job summary and report case narrative.

10.4.5. If one of the columns fails CCV criteria (but the CCV is between 15%-40% greater than expected value), the sample results shall be reported from the compliant column. If the falls outside of acceptance criteria on the low side, reanalysis shall be performed.

10.4.6. If the CCV on one of the columns is more than 40% different from the correct value, it can be assumed that there has been significant drift on that column. The sample shall be reanalyzed against an acceptable calibration.

10.4.6.1 An exception to this requirement would be if the CCV recovery on one column fails on the high side and >40% RPD but the associated samples were non-detect for all target analytes on both columns. In this case the non-detect results may be reported from the compliant column.

10.4.7 In summary, the flow chart in Attachment 3 presents a recommended rational approach to selecting the better number to report for dual column data. It shall be noted that these recommendations may be overridden by project specific requirements and that they cannot cover all eventualities. The complexity of some data set will require the final decision to be made utilizing the judgment of experienced analysts. In some cases further cleanup steps to remove interferences may be appropriate.

10.5. Data Processing

10.5.1. Process all samples, QC samples, and blanks using Chrom. Evaluate the QC samples (Method Blank, MS/MSD, and LCS) against the criteria detailed in Section 9.1. Take corrective action as detailed in that section.

10.5.2. Evaluate chromatograms for carryover and cross-contamination. Any samples suspected of having carryover must be re-prepped and re-analyzed. To make sure there is no carryover, reagent water is injected before the re-prepped sample.

10.5.3. Any compound concentration that exceeds the concentration of the calibration range must be diluted and re-analyzed.

10.5.4. The reporting limit is based on the concentration of the lowest standard in the initial calibration, adjusted for the sample wt/vol, final volume, dilution factor and %moisture (No unqualified analytical results or non detects may be reported which correspond to an extract concentration less than the lowest standard in the calibration range).

11.0. Calculations / Data Reduction

11.1. Accuracy:

$$\text{ICV / CCV, LCS \% Recovery} = \frac{\text{observed concentration}}{\text{known concentration}} \times 100$$

$$\text{MS \% Recovery} = \frac{\text{(spiked sample) - (unspiked sample)}}{\text{spiked concentration}} \times 100$$

11.2. Precision (RPD):

$$\text{Matrix Duplicate (MD)} = \frac{|\text{orig. sample value - dup. sample value}|}{[(\text{orig. sample value + dup. sample value})/2]} \times 100$$

11.3. $$\text{Concentration = mg/kg or L} = \frac{C \times V \times D}{W}$$

Where:
C = sample concentration in extract (ppm)
V = Volume of extract (mL)
D = Dilution Factor

W = Weight/Volume of sample aliquot extracted (grams or mLs)

NOTE: All dry weight corrections are made in LIMS at the time the final report is prepared

11.4. Relative Standard Error (RSE)

$$\%\,RSE = 100\% \times \sqrt{\frac{\sum_{i-1}^{N}\left[\frac{C_i - PC_i}{C_i}\right]^2}{(N-P)}}$$

Where:

N = Number of points in the curve
P = Number of parameters in the curve (= 1 for average response factor, 2 for linear, 3 for quadratic)
CI = True concentration for level i
PCi = Predicted concentration for level i

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12.0. Method Performance

12.1. Method Detection Limit Study (MDL)

The method detection limit (MDL) is the lowest concentration that can be detected for a given analytical method and sample matrix with 99% confidence that the analyte is present. The MDL is determined according to the laboratory's MDL procedure in Section 20 (Test Methods and Method Validation) of Eurofins Edison's Quality Assurance Manual (ED-QA-LQM). MDLs reflect a calculated (statistical) value determined under ideal laboratory conditions in a clean matrix, and may not be achievable in all environmental matrices. The laboratory maintains MDL studies for analyses performed; these are verified at least annually unless method requirements require a greater frequency.

12.2. Lower Limit of Quantitation (LLOQ) (aka Reporting Limit) Verification:

The LLOQ is the lowest concentration at which the laboratory has demonstrated target analytes can be reliably measured and reported with a certain degree of confidence, which must be ≥ the lowest point in the calibration curve. The lab verifies the LLOQ annually to demonstrate the capability of quantiation at lower analyte concentrations. The verification is performed by the extraction and analysis of an MDL spike at a concentration of 0.5-2 times the established LLOQ. Recovery of target analytes in the LLOQ verification should be within established in-house limits or within other such project-specific acceptance limits to demonstrate acceptable method performance at the LLOQ. Until the laboratory has sufficient data to determine acceptance limits, the LCS criteria ± 20% (i.e., lower limit minus 20% and upper limit plus 20%) will be used for the LLOQ acceptance criteria. The annual LLOQ verification is completed and documented with the required annual MDL evaluation.

12.3. Demonstration of Capabilities

For DOC procedure refer to Section 19 in the most current revision of Eurofins Edison's Quality Assurance Manual (ED-QA-LQM).

12.4. Training Requirements

Refer to Eurofins Edison SOP No. ED-GEN-022, Training, for the laboratory's training program.

13.0. Pollution Control

13.1. Pollution prevention encompasses any technique that reduces or eliminates the quantity or toxicity of waste at the point of generation. Numerous opportunities for pollution prevention exist in laboratory operation. The USEPA has established a prevention hierarchy of environmental management techniques that places pollution prevention as the management option of first choice. Whenever feasible, laboratory personnel should use pollution prevention techniques to address their waste generation. When wastes cannot be feasibly reduced at the source, the agency recommends recycling as the next best option.

13.2. The quantity of chemical purchased should be based on expected usage during its shelf life and disposal cost of unused material. Actual reagent preparation volumes should reflect anticipated usage

14.0. Waste Management

14.1. The U.S. Environmental Protection Agency requires that laboratory waste management practices conducted be consistent with all applicable rules and regulations. All waste will be disposed of in accordance with Federal, State and Local regulations. Where reasonably feasible, technological changes have been implemented to minimize the potential for pollution of the environment. Employees will abide by this method and the policies in section 13 of the Corporate Safety Manual for "Waste Management and Pollution Prevention."

14.2. The following waste streams are generated as a result of this analysis:

The following waste streams are generated when using this method:

- Auto sampler vials and expired standards: These vials are collected in satellite accumulation within the instrument laboratory. The vials are then placed into a 55 steel open top drum in the waste room. When the drums are full, the drum will be collected by the waste vendor for disposal. This waste is treated for incineration.

  Teris Profile Number: 50016652
  Onyx Profile WIP Number: 282493

- Mixed Solvent Waste: Mixed solvent waste is collected in a small beaker inside the bench top hood. This waste is then transferred into the satellite accumulation container in the Organic Prep. Lab. on a daily basis. This material is transferred into 5 gallon solvent cans as satellite accumulation. These cans are emptied every 24 hours into a steel drum in the

waste room. This drum is kept in the walk in hood until it is full. The full drum is then removed from the hood and placed on secondary containment in the waste room.

Teris Profile Number: 50016624
Onyx Profile WIP Number: 545240

15.0. References / Cross-References

15.1. United States Environmental Protection Agency, "Method 8151A: Organochlorine Herbicides by Gas Chromatography", Test Methods for Evaluating Solid Wastes, SW846 Third Edition, Volume 1B: Laboratory Manual, Physical/Chemical Methods, Revision 3, December 1996.

15.2. Eurofins Edison SOP No. ED-ORP-015, Extraction of Organochlorine Herbicides in Water by SW846 Method 8151A, current revision.

15.3. Eurofins Edison SOP No. ED- ORP-023,Extraction of Organochlorine Herbicides in Soil by SW846 Method 8151A, current revision.

15.4. Eurofins Edison Document No. ED-QA-LQM, Laboratory Quality Manual, current revision.

15.5. Eurofins Corporate Document No. CW-E-M-001, Environmental Health and Safety Manual, current revision.

15.6. Eurofins Corporate Quality SOP No. CA-Q-S-001, Solvent & Acid Lot Testing & Approval, current revision.

15.7. Eurofins Edison SOP No. ED-GEN-023, Bulk Solvent Testing and Approval, current revision.

15.8. Eurofins Edison SOP No. ED-GEN-013, Glassware Cleaning, most current revision

15.9. Eurofins Edison SOP No. ED-GEN-022, Training, current revision.

16.0. Method Modifications:

N/A

17.0. Attachments

Attachment 1: Herbicide Free Acid Conversion Chart

Attachment 2: Retention Time (RT) Windows for Primary Herbicides/Surrogate

Attachment 3: Dual Column Reporting Flowchart

18.0. Revision History

- Revision 12, dated 17 Oct 2022
    - Updated throughout to reflect Eurofins branding.
    - Section 9.2.3.3: Included details of evaluation of initial calibration for Relative Standard Error (RSE).
    - Section 11.4: added formula for calculation of RSE.
    - Section 12.2 added: details the annual LLOQ verification requirement.

- Revision 11, dated 22 March 2021
    - Added Section 10.5.4 which details the establishment of the reporting limit (RL).

- Revision 10, dated 10 December 2020
    - Updated throughout to reflect Eurofins branding.
    - Revised references to Quality Manual section numbers as required

- Revision 9, dated 22 February 2016
    - Revised throughout to correct the spelling of Picloram (4-amino-3,5,6-trichloropicolinic acid).

- Revision 8, dated 12 August 2013
    - Revised throughout to reflect the lab's implementation of the TestAmerica Chrom data system.
    - Deleted Section 7.3.2.1 (Total Herbicide Calibration Standards, 3 Compound Analysis). Renumbered remaining sections accordingly.
    - Deleted Section 7.3.1.1 (Herbicide QC Spiking Standard, 3 Compound Analysis). Renumbered remaining sections accordingly.
    - Deleted Table 4 (Preparation of Total Herbicide Calibration Standards, 3 Compound Analysis). Renumbered remaining tables accordingly.
    - Revised 2,4,5-TP (Silvex) methyl ester concentrations (all levels) and Dalapon (level 3) in Table 4 (formerly Table 5)
    - Revised Pentachlorophenol concentrations (levels 2 and 3) in Table 5 (formerly Table 6)
    - Revised Picloram concentrations (all levels) and 2,4-DCAA methyl ester (level 4) in Table 6 (formerly Table 7)

- Revision 7, dated 15 August 2011
    - Section 7.3.3.4: Corrected weight of neat Picloram from 9 grams to 10 grams
    - Attachment 3: revised to reflect current practice.
    - Revised 'Distribution' footer on Cover Page to reflect current practice.

- Revision 6, dated 28 October 2009

    - Revised SOP format in accordance with TestAmerica Corporate Quality SOP No. CW-QS-002 (Rev 0) Writing a Standard Operating Procedure (SOP).
    - Section 1.1. Added Table 1, *Analytes and Reporting Limits*
    - Section 1.1 Added reference to Quality Assurance Manual for method modifications.
    - Section 1.1: Expanded to include references to applicable prep and cleanup SOPs.
    - Section 3: revised to reference new location for definitions.
    - Section 5: Revised to include most up to date corporate health and safety references and information.
    - Section 7.2.2: Added table detailing components found in the various standards mixes.
    - Section 7.3: Updated the instructions for preparation of standards.
    - Section 7.3: Added tables with calibration standards prep details.
    - Section 8: Updated with additional details including a table outlining containers, preservation and holding times for waters and soils.
    - Section 9.1: Expanded QC sample preparation, analysis, evaluation and corrective action details.
    - Section 9.2: Expanded details of preparation, analysis, evaluation and corrective action for initial and continuing calibration and calibration verifications.
    - Section 10.2.3: added procedures for dual column evaluation.
    - Section 10.4.1: Updated Idealized Analytical Sequence table.
    - References: Expanded to include more specific SOP references
    - Section 18: Added this Revision History section
    - Throughout document: added references to TestAmerica LIMS (TALS).
    - Attachments: Added Attachment 3: Dual Column Reporting Flowchart

Attachment 1

Herbicide Free Acid Conversion Chart

Analyte	Free Acid Molecular Weight	Methyl Ester Molecular Weight
Dalapon	142.97	156.97
2, 4 - D	221.00	235.00
2, 4, 5 - TP (Silvex)	269.50	283.50
2, 4, 5 - T	255.50	269.50
Dinoseb	240.22	254.22
2, 4 - DB	249.09	263.09
Dicamba	221.04	235.04
Dichloroprop	235.05	249.05
Picloram	241.48	255.48
Pentachlorophenol	266.50	280.50
MCPA	200.63	214.63
MCPP	200.63	214.63
DCAA	297.13	311.13

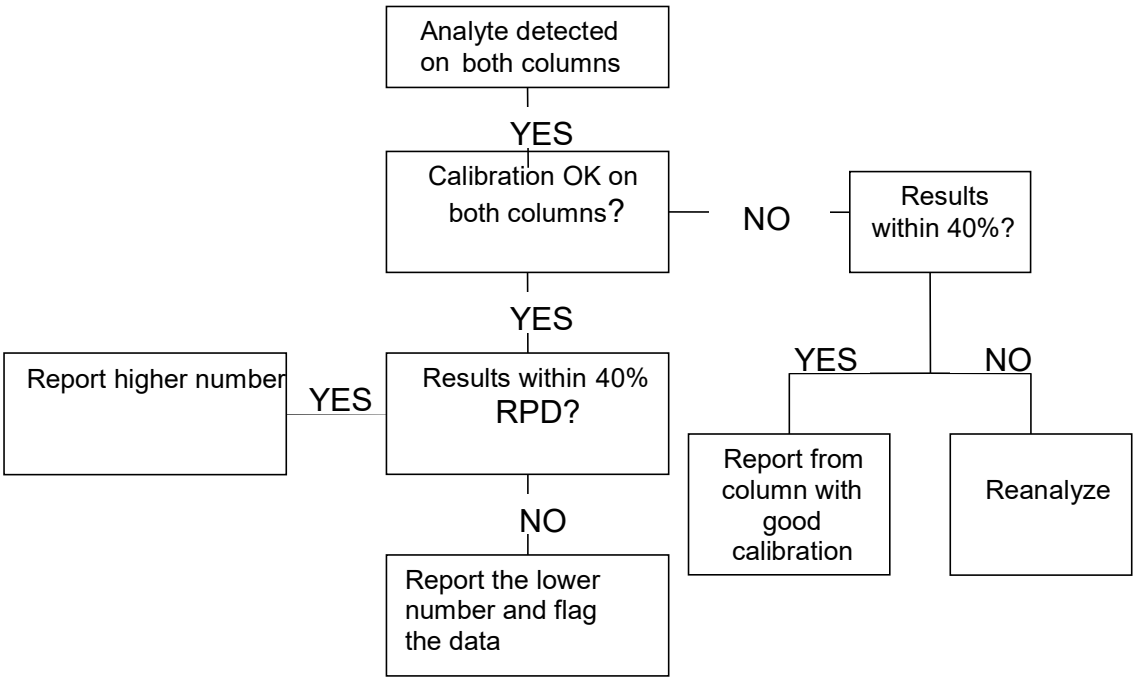
$$[\text{Free Acid}] = \frac{[\text{Methyl Ester}] \times \text{Free Acid Molecular Weight}}{\text{Methyl Ester Molecular Weight}}$$

Attachment 2

Retention Time (RT) Windows For Herbicides/Surrogate

Compound	RT Window (minutes)	
	Total Herbs	TCLP Herbs
2,4-D	*± 0.10*	*± 0.05*
2,4,5-TP (Silvex)	*± 0.10*	*± 0.05*
2,4,5-T	*± 0.10*	*± 0.05*
Dalapon	*± 0.10*	
2,4-DB	*± 0.10*	
Dicamba	*± 0.10*	
2,4-DP (Dichloroprop)	*± 0.10*	
Picloram	*± 0.10*	
Pentachlorophenol	*± 0.10*	
MCPA	*± 0.10*	
MCPP	*± 0.10*	
Picloram	*± 0.10*	
DCAA (surrogate)	*± 0.10*	*± 0.10*

Attachment 3
Dual Column Reporting Flowchart



Title: SW846 Method 8082A, Analysis of Polychlorinated Biphenyls by Gas Chromatography

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Approvals (Signature/Date):

Catalina R. Dalangin	10/21/2022	David W. Helfrich	10/21/2022
Catalina Dalangin SVOA GC Manager	Date	Dan Helfrich Health & Safety Manager	Date
	10/21/2022		10/21/2022
Carl Armbruster Quality Assurance Manager	Date	Mark Acierno Laboratory Director	Date

Sylvanus Klusey 10/21/2022
Sylvanus Klusey Date
Operations Manager - Organics

1.0 Scope and Application

This method is used to quantify specific polychlorinated biphenyls (PCBs) as Aroclors (see Table 1 below) in extracts from aqueous, soil, sludge, leachate, wipe or oil matrices by direct injection dual capillary column gas chromatography using SW846 Method 8082A. An electron capture detector (ECD) is employed for detection.

1.1 Analytes, Matrix(s), and Reporting Limits

The specific analytes determined by this method are identified in Table 1.

Table 1
Polychlorinated Biphenyls

Compound Name	CAS Registry No.
Aroclor 1016	12674-11-2
Aroclor 1221	11104-28-2
Aroclor 1232	11141-16-5
Aroclor 1242	53469-21-9
Aroclor 1248	12672-29-6
Aroclor 1254	11097-69-1
Aroclor 1260	11096-82-5
Aroclor 1262	37324-23-5
Aroclor 1268	11100-14-4

The routine Eurofins Edison reporting limits (RLs) by analyte and matrix are summarized in Table 2 (below).

Table 2
Reporting Limits by Matrix

Parameter	Soil	Soil	Water	Leachate	Oil	Wipe
	Reporting Limits (ug/kg) LOW Level	Reporting Limits (ug/kg) MED Level	Reporting Limits (ug/L)	Reporting Limits (mg/L)	Reporting Limits (ug/kg)	Reporting Limits (ug/wipe)
Aroclor-1016	67	500	0.50	0.0050	1000	0.40
Aroclor-1221	67	500	0.50	0.0050	1000	0.40
Aroclor-1232	67	500	0.50	0.0050	1000	0.40
Aroclor-1242	67	500	0.50	0.0050	1000	0.40
Aroclor-1248	67	500	0.50	0.0050	1000	0.40
Aroclor-1254	67	500	0.50	0.0050	1000	0.40
Aroclor-1260	67	500	0.50	0.0050	1000	0.40
Aroclor-1262	67	500	0.50	0.0050	1000	------
Aroclor-1268	67	500	0.50	0.0050	1000	------

The most current MDLs and RLs for this method can be found in the active Eurofins LIMS (TALS) SW846 8082A Method Limit Group (MLG) database.

1.2 On occasion clients may request modifications to this SOP. These modifications are handled following the procedures outlined in Section 7 (*Review of Work Request*) and Section 19 (*Test Methods and Method Validation)* of Eurofins Edison's Quality Assurance Manual (ED-QA-LQM).

2.0 Summary of Method

2.1. Samples undergo a preparation step prior to analysis by SW846 Method 8082A. A measured volume or weight of sample (15 g for soil, 1 g for oil, 250 ml for water and TCLP/SPLP/ASTM leachates) is extracted using the appropriate matrix-specific sample extraction technique (reference the applicable Organic Sample Prep SOPs listed below). The extract is exchanged into hexane and concentrated to a final volume between 1 and 20 ml depending upon the prep technique used.

2.1.1. Aqueous and leachate samples are extracted at a neutral pH using SW846 Method 3510C (SOP No. ED-ORP-014: *Extraction of Pesticides and PCBs in Water by Separatory Funnel, SW846 Method 3510C).*

2.1.2. Wipe samples are extracted using SW846 Method 3550B: Sonication (*SOP No. ED-ORP-018: Extraction of Pesticides/PCBs in Soil Using Low-Level Extraction, SW846 Method 3550B*)

2.1.3. Solid samples are extracted using SW846 Method 3546 (*SOP No. ED-ORP-0044: Procedure for the Microwave Extraction of Solids,SW846 Method 3546*).

2.1.4. Organic liquids are prepared using SW846 Method 3580A (*SOP No. ED-ORP-019: Waste Dilution for Pesticides and PCBs, SW846 Method 3580A*).

2.1.5. Extract cleanup steps are employed as need depending on the nature of the matrix interferences encountered. Suggested cleanups include SW846 Method 3620B (*SOP No. ED-ORP-020: Florisil Cleanup for Pesticide/PCB Sample Extracts*), SW846 Method 3660B (*SOP No. ED-ORP-021: The Removal of Elemental Sulfur from Pesticide/PCB Sample Extracts*) and SW846 Method 3665A (*SOP No. ED-ORP-022, Sulfuric Acid Cleanup for PCB Extracts, SW846 Method 3665A*) for heavy organic interferences.

2.2. After cleanup, the extract is analyzed by injecting a known volume of sample into a gas chromatograph equipped with a dual wide-bore fused silica capillary columns and dual electron capture detectors (GC/ECD). The GC is temperature programmed to separate and detect the analytes recovered during the extraction step. Quantitation is accomplished by comparing the area response of each target analyte relative to an internal standard established through a five-point initial calibration (six points for second order regression). Specific calibration and quality control steps are detailed in this SOP and meet the specification of SW846 Method 8082A.

2.3. Samples are analyzed only after all the necessary calibration and QC checks have been performed.

2.4. Acquired data from sample analysis is manually reviewed. Secondary column confirmation of target compounds and quantitation are conducted by the analyst as required.

3.0 Definitions

For a complete list of definitions refer to Appendix 2 in the most current revision of Eurofins Edison's Quality Assurance Manual (ED-QA-LQM).

4.0 Interferences

4.1. Interferences from phthalate esters introduced during sample preparation can pose major difficulties for PCB determinations.

4.1.1. Interferences from phthalate esters can be minimized by avoiding contact with any plastic materials and checking all solvents and reagents for phthalate contamination. Exhaustive cleanup of solvents, reagents and glassware may be required to eliminate background phthalate ester contamination.

4.2. The presence of elemental sulfur will result in broad peaks that interfere with the detection of early-eluting PCBs. Sulfur contamination should be expected with sediment samples. Employ SW846 Method 3660B (*SOP No. ED-ORP-021: The Removal of Elemental Sulfur from Pesticide/PCB Sample Extracts*) for removal of sulfur.

4.3. Co-eluting chlorophenols are eliminated by using SW846 Method 3620B (*SOP No. ED-ORP-020: Florisil Cleanup for Pesticide/PCB Sample Extracts*),

4.4. Interferences from other organic compounds can effectively be removed using a sulfuric acid treatment, SW846 Method 3665A (*SOP No. ED-ORP-022, Sulfuric Acid Cleanup for PCB Extracts, SW846 Method 3665A*). This destructive technique can be employed only when the sample extract is being analyzed solely for PCBs (i.e., it is not to be used prior to analysis for pesticides).

4.5. Compounds extracted from the sample matrix to which the detector will respond, such as single-component chlorinated pesticides, including the DDT analogs (DDT, DDE, and DDD) will cause interference. A standard of the DDT analogs should be injected to determine which of the PCB or Aroclor peaks may be subject to interferences on the analytical columns used. There may be substantial DDT interference with the last major Aroclor 1254 peak in some soil and sediment samples. A System Performance Check (DDT/Endrin) is analyzed daily to assist in evaluation of potential DDT interference with this Aroclor 1254 peak.

5.0 Safety

Employees must abide by the policies and procedures in the Corporate Environmental Health and Safety Manual (CW-E-M-001) and this document. This procedure may involve hazardous material, operations and equipment. This SOP does not purport to address all of the safety problems associated with its use. It is the responsibility of the user of the method to follow appropriate safety, waste disposal and health practices under the assumption that all samples and reagents are potentially hazardous. Safety glasses, gloves, lab coats and closed-toe, nonabsorbent shoes are a minimum

5.1. Specific Safety Concerns or Requirements

The gas chromatograph contains zones that have elevated temperatures. The analyst needs to be aware of the locations of those zones, and must cool them to room temperature prior to working on them.

There are areas of high voltage in the gas chromatograph. Depending on the type of work involved, either turn the power to the instrument off, or disconnect it from its source of power.

5.2. Primary Materials Used

The following is a list of the materials used in this method, which have a serious or significant hazard rating. **Note: This list does not include all materials used in the method. The table contains a summary of the primary hazards listed in the MSDS for each of the materials listed in the table.** A complete list of materials used in the method can be found in the reagents and materials section. Employees must review the information in the MSDS for each material before using it for the first time or when there are major changes to the MSDS.

Material (1)	Hazards	Exposure Limit (2)	Signs and symptoms of exposure
Acetone	Flammable	1000 ppm-TWA	Inhalation of vapors irritates the respiratory tract. May cause coughing, dizziness, dullness, and headache.
Hexane	Flammable Irritant	500 ppm-TWA	Inhalation of vapors irritates the respiratory tract. Overexposure may cause lightheadedness, nausea, headache, and blurred vision. Vapors may cause irritation to the skin and eyes.
Methylene Chloride	Carcinogen Irritant	25 ppm-TWA 125 ppm-STEL	Causes irritation to respiratory tract. Has a strong narcotic effect with symptoms of mental confusion, light-headedness, fatigue, nausea, vomiting and headache. Causes irritation, redness and pain to the skin and eyes. Prolonged contact can cause burns. Liquid degreases the skin. May be absorbed through skin.

Material (1)	Hazards	Exposure Limit (2)	Signs and symptoms of exposure
Sulfuric Acid	Corrosive Oxidizer Dehydrator Poison Carcinogen	1 Mg/M3-TWA	Inhalation produces damaging effects on the mucous membranes and upper respiratory tract. Symptoms may include irritation of the nose and throat, and labored breathing. Symptoms of redness, pain, and severe burn can occur. Contact can cause blurred vision, redness, pain and severe tissue burns. Can cause blindness.
1 – Always add acid to water to prevent violent reactions.			
2 – Exposure limit refers to the OSHA regulatory exposure limit.			

6.0 Equipment and Supplies

6.1 Gas Chromatograph:

6.1.1 Agilent Technologies (Avondale, PA) model 5890/6890 Gas Chromatograph (GC), equipped for simultaneous quantitation and confirmation columns using two separate detector channels on dual megabore capillary columns that are suitable for the analysis of organochlorine pesticides and PCB's. All operations are as automated as possible with the equipment utilized.

6.1.2 Injection system: Sample injection is accomplished by a single auto injector. The auto injector is serviced by a robot arm that shuttles a single sample between the sample tray and the injector turret.

6.1.2.1 The sample is injected into a split/splitless injection port equipped with electronic pressure control (EPC). The injection port is normally operated in splitless mode during injection. The EPC is operated in the ramp pressure mode.

6.1.3 Liners: The injection port is each fitted with a replaceable, heavy-walled glass double gooseneck liner. The liner contains a plug of silanized glass wool approximately 1 cm in length. The glass wool is positioned in the liner between the double gooseneck. The liner is replaced on a regular maintenance schedule.

6.1.4 Oven and Columns: Temperature programmable gas chromatograph ovens are required, capable of integrated temperature control between 35^{O}C and 350^{O}C.

6.1.4.1 Two dissimilar columns are used for analysis. A Restek StxCLPesticides, 30m x 0.53mm ID x 0.5um film thickness column (or equivalent) is used for sample quantitation. The secondary column is a Restek StxCLPesticides II, 30m x 0.53mm ID x 0.42um film thickness column (or equivalent).

6.1.5 Detectors: Sample detection is by electron capture. The GC is equipped with dual Electron Capture Detectors (ECD), one for each column.

6.1.5.1 Each detector is supplemented with make-up gas to provide sufficient detector flow for maintaining the electron plasma. This is in addition to the gas exiting the column. The make-up gas is fed from a supply other than the injection port.

6.2 Data System:

6.2.1 The data systems consist of Agilent Technologies GC Chemstation Revision A.08.02 and Agilent Technologies Enviroquant Chemstation G1701AA Version A.03.00 upgraded to A.03.02 which is used for acquisition and Eurofins Chrom (chromatography data processing software).

7. Reagents and Standards

7.1. Reagents

7.1.1. Gases: Ultra high purity (99.999%) Hydrogen is used as the carrier and injection port purge gas. It is introduced to the GC at the injection port. Ultra high purity (99.999%) Nitrogen is used as make-up gas. (Alternatively, ultra-high purity Helium with P-5 make-up gas may be used). Make-up gas is introduced to the GC via the make-up gas adapter at the end of the capillary column. Gases are supplied at tank pressures of 2000-2400 psig for a 300 cft tank. The tank pressure is regulated to an outlet pressure of 70 psig. Each tank is used until the tank pressure drops to less than 500 psig.

7.1.1.1. The gas streams are polished using three traps or filters before introduction to the G.C. The traps are as follows:

- Hydrocarbon trap
- H_2O (moisture) trap
- O_2 scrubber

7.1.1.2. Both the moisture trap and the Oxygen scrubber are of the indicating type. They require either replacement or reconditioning upon color change of the active agents. Refer to the instructions for the individual traps to determine if it is still active. The hydrocarbon trap is a simple activated carbon trap. With high quality gas, it should last for an extended period of time (1-yr. minimum).

7.1.2. Solvents used in the extraction, clean up procedures and dilutions include Hexane, Methylene Chloride, and Acetone that are exchanged to Hexane prior to analysis. All solvents must be pesticide quality or equivalent. Each lot of solvent is screened for contaminants before being used for analysis.

7.2. Standards

7.2.1. Standards are purchased as concentrated solutions. Standard compounds or mixtures for this analysis include an Aroclor 1016/1260 mix, Aroclor 1221, 1232, 1242, 1248, 1254, 1262, 1268 and the surrogate compound Decachlorobiphenyl (DCB) (packaged with the Tetrachloro-m-xylene (TCMX), a surrogate used in pesticide analysis).

NOTE: Two independent sources are used for quantitation standards and spiking standards

7.2.1.1. Most stock solutions are diluted (in volumetric glassware) to working concentration using hexane as the diluent.

7.2.2. Standards mixes and sources: *

Standard Name	Source	Concentration
TCMX/DCB Surrogate Calibration Mix	Restek 32000	200 ug/ml
TCMX/DCB Surrogate Spike Mix	Supelco 861275	10 ug/ml
Aroclor 1016 Calibration Standard	Supelco 48097	1000 ug/ml
Aroclor 1221 Calibration Standard	Restek 32007	1000 ug/ml
Aroclor 1232 Calibration Standard	Restek 32008	1000 ug/ml
Aroclor 1242 Calibration Standard	Restek 32009	1000 ug/ml
Aroclor 1248 Calibration Standard	Restek 32010	1000 ug/ml
Aroclor 1254 Calibration Standard	Restek 32011	1000 ug/ml
Aroclor 1260 Calibration Standard		1000 ug/ml
Aroclor 1262 Calibration Standard	Restek 32409	1000 ug/ml
Aroclor 1268 Calibration Standard	Restek 32410	1000 ug/ml
Aroclor 1660 Mix (Arolcors 1016 & 1260)	Restek 32039	1000 ug/ml
Aroclor 1016/1260 Calibration Standard (Second Source)	Restek 32039.sec	1000 ug/ml
1-Bromo-2-nitrobenzene (internal standard)	Restek 32279	1000 ug/ml

*Suppliers with equivalent standards may be used.

7.2.3. Aroclor 1016/1260 & Surrogate Calibration Standard Solution Preparation

Five levels of calibration standards are prepared using the above referenced Aroclor 1660 Mix (Restek – 23039) and TCMX/DCB Surrogate Calibration standard mix (Restek 32000). They are prepared as follows:

Final Concentration of Aroclor 1016/1260 (Concentration of DCB)	Volume (ul) of Aroclor 1660 Mix (1000 ug/ml)	Volume (ul) of TCMX/DCB Surrogate Calibration Mix (200 ug/ml)	Final Volume in hexane (ml)
50 ppb (25 ppb DCB) [1]	5	6.25	100
500 ppb (50 ppb DCB)	50	25	100
1000 ppb (100 ppb DCB)	1000	500	1000
1500 ppb (150 ppb DCB)	150	75	100

Final Concentration of Aroclor 1016/1260 (Concentration of DCB)	Volume (ul) of Aroclor 1660 Mix (1000 ug/ml)	Volume (ul) of TCMX/DCB Surrogate Calibration Mix (200 ug/ml)	Final Volume in hexane (ml)
2500 ppb (200 ppb DCB)	250	100	100

(1): The low level Aroclor 1016/1260 standard is 50 ppb and 12.5 ppb for the surrogate DCB (prepare by making a 2x dilution of the 100 ppb standard in hexane).

7.2.4. Surrogate Spiking Solution (soil and water)

A TCMX/DCB Surrogate Spike Mix is prepared by diluting 10 ml of Restek 32000 (see Table 1 above) to 200 ml of Acetone. Final solution concentration is 10 ug/ml. For reduced volume LVI preps a secondary dilution of this mix is utilized. This is prepared by diluting 20 ml of the 10 ug/ml solution to 100 ml acetone with a final solution concentration of 2 ug/ml.

7.2.5. Aroclor 1016/1260 Spiking Solution (soil, water and wipe)

An Aroclor 1660 Mix is prepared by diluting 10 ml of Restek 32039 (See Tables 1 and 2 above) to 100 ml acetone. Final solution concentration is 100 ug/ml. For reduced volume LVI preps a secondary dilution of this mix is utilized. This is prepared by diluting 20 ml of the 100 ug/ml solution to 200 ml with a final solution concentration of 20 ug/ml. used spiking soils and waters as received from Supelco without further dilution.

7.2.6. Individual Aroclor Calibration Solutions (1221, 1232, 1242, 1248, 1254, 1262 & 1268)

A 1000 ppb calibration standard is prepared for each remaining Aroclor from the stock standards detailed in Section 7.2.1. 200ul of 1000 ug/ml individual Aroclor solution and 100ul of 200 ug/ml TCMX/DCB is diluted to 200ml with Acetone. The final concentration of surrogates is 100 ppb.

7.2.7. Aroclor 1016/1260 Initial Calibration Verification (ICV) Standard Solution Preparation

A mid-point Aroclor 1016/1260 ICV standard is prepared using the second source Aroclor 1016/1260 Calibration Standard (Restek 32039.sec) detailed in Section 7.2.2. 1000 ul of 1000 ug/ml standard along with 500 ul of 200 ug/ml TCMX/DCB surrogate standard (Restek 32000) is diluted to 1000 ml with acetone for a final ICV concentration of 1 ug/ml (1000 ppb).

7.2.8. PCB Internal Standard Spike Mix (1 ug/ml)

The PCB 1 ug/ml internal standard spike mix is prepared by dilution 500ul of 1000 ug/ml of the 1-Bromo-2-Nitrobenzene standard (Restek 32279) in to 500 ml of Hexane. 20 ul of this solution is added to all standards, QC samples and field sample extracts prior to analysis.

7.2.9. System Performance Solution (3,3-DDT and Endrin at 0.25 ug/ml):

The DDT/Endrin performance solution is prepared by taking 250 ul of 500 ug/ml DDT/Endrin Mix (Supelco Catalog No 48282) and bringing it up to a volume of 500 ml with hexane.

8. Sample Collection, Preservation, Shipment and Storage

Sample container, preservation techniques and holding times may vary and are dependent on sample matrix, method of choice, regulatory compliance, and/or specific contract or client requests. Listed below are the holding times and the references that include preservation requirements.

Matrix	Sample Container	Min. Sample Size	Preservation	Holding Time	Reference
Waters	Amber glass, 250 ml	250 ml	Cool 4 ± 2°C	7 days to extraction; Analyze within 40 days of extraction	SW846
Soils	Glass, 2 or 4 oz	100 g	Cool 4 ± 2°C	14 days to extraction; Analyze within 40 days of extraction	SW846

.

8.1. Extracts must be stored under refrigeration in the dark and analyzed within 40 days of extraction.

8.2. Samples from chlorinated water sources must be treated with sodium thiosulfate (0.008% solution) at the time of collection to remove chlorine. NOTE: containers pre-preserved with sodium thiosulfate must be requested in bottle orders for samples from chlorinated water sources.

9. Quality Control

9.1. **Sample QC** - The following quality control samples are prepared with each batch of samples.

Quality Controls	Frequency	Control Limit
Method Blank (MB)	1 in 20 or fewer samples	< Rpt. Limit
Laboratory Control Sample (LCS)[1]	1 in 20 or fewer samples	Statistical Limits [4]
Matrix Spike (MS)[2]	1 in 20 or fewer samples	Statistical Limits [4]
MS Duplicate (MSD)[2]	1 in 20 or fewer samples	Statistical Limits [4]
Surrogates	every sample[3]	Statistical Limits [4]
Internal Standard	every sample[3]	Response within -50% to +100% of most recent cal standard.

[1] LCS Duplicate (LCD) is performed only when insufficient sample is available for the MS/MSD or when requested by the client/project/contract.
[2] The sample selection for MS/MSD are randomly selected, unless specifically requested by a client….predetermined by the extraction lab.
[3] Analytical and QC samples (MB, LCS, MS/MSD)
[4] Statistical control limits are updated annually and are updated into LIMS.

9.1.1. **Method Blanks** are extracted with each sample batch on each day that samples are extracted. The analytical results for the method blank must fall below the reporting limit for each compound of interest. If a target compound is detected in the blank at a concentration higher than the reporting limit, first the extract is reanalyzed for confirmation. If results are still outside of limits the entire batch of samples extracted with the affected blank must be re-extracted and reanalyzed.

9.1.2. **Laboratory Control Sample (LCS):** A Laboratory Control Sample (LCS) or blank spike must be extracted and analyzed for with each batch of 20 environmental samples. The LCS data is used to assess method performance and serves to determine whether the methodology is in control at the time of preparation and analysis. The recoveries of the LCS must fall within lab generated acceptance criteria. If the LCS recovery results are outside QC limits, the extract is reanalyzed. If upon reanalysis the recoveries remain outside of recovery limits the following evaluations are made:

- If LCS results fall outside the laboratory generated limits with low recoveries (refer to the current TALS Method Limit Group database), the LCS and all associated samples should be re-extracted and re-analyzed.

- If LCS results fall outside the laboratory generated limits with high recoveries (refer to the current TALS Method Limit Group database), the LCS and associated sample results may be reported with an Non-Conformance Memo (NCM) detailing the issue.

9.1.3. **Matrix Spike/Matrix Spike Duplicate (MS/MSD):** A Matrix Spike/Matrix Spike Duplicate (MS/MSD) pair is extracted and analyzed with every batch of 20 environmental samples. MS/MSD recoveries are evaluated against lab generated limits (refer to the current TALS Method Limit Group database). If the MS/MSD recovery limits fall outside of lab limits the LCS recovery is evaluated. If the LCS recoveries meet criteria the data is reported and a Non-Conformance Memo (NCM) is written.

9.1.4. **Surrogate Standards:** All samples, blanks and QC samples are spiked with a 2 component surrogate standard mix containing TCMX & DCB (see Section 7.2). The percent recovery of the DCB surrogate standard is calculated and compared to lab generated limits (refer to the current TALS Method Limit Group database). (Note: the surrogate must pass CCV criteria to be reportable and must be reported from the same

column as sample target analyte results). If the DCB recovery is outside of acceptance limits the sample extract is reanalyzed to confirm. If the recoveries are still outside of limits the sample must be re-extracted and reanalyzed or the data flagged as “estimated concentration”.

9.1.5. Internal Standard: The internal standard (1-bromo-2-nitrobenzne) must elute within 30 seconds of and have an area response of 50 to 100% as compared to the most recent preceding calibration standard.

9.2. Instrument QC

9.2.1. Initial Calibration Range and Initial Calibration Verification (ICV)

9.2.1.1. *Initial Calibration Range*: Aroclors 1016/1260 and the surrogate (DCB) are calibrated using a five-point calibration range using a minimum of five (5) peaks per Aroclor. The reporting limit (RL) is equal to the low point of the calibration range. The initial calibration block must include at least one level with Aroclor 1016 analyzed separately for pattern recognition purposes (note: this run does not need to be part of the actual calibration). If the 1016/1260 calibration meets the required average RF criteria all other Aroclors are then calibrated at the anticipated midpoint of the calibration range with a single point calibration using a minimum of five (5) peaks for each Arolcor (minimum of 3 peaks for Aroclor 1221). All peaks selected must be at least 25% the peak height of the largest peak in the Aroclor (except for Aroclor 1268 where the requirement is 10%). The following Aroclors can be analyzed together: 1016/1260, 1221/1254, 1232/1262 and 1242/1268. Standards are prepared following the instructions in Section 7.2.

9.2.1.2. *Initial Calibration Verification (ICV):* An Initial Calibration Verification (ICV) standard is analyzed immediately after the Initial Calibration Range and before any samples are analyzed. The ICV is prepared as detailed in Section 7.2.7 and must be from a source separate from the standards used in the Initial Calibration Range.

9.2.2. Continuing Calibration Verification (CCV): A mid-point Continuing Calibration Verification (CCV) standard (typically Aroclors 1016/1260) must be analyzed after every 20 samples at minimum. If the samples being analyzed are being specifically targeted for a different Aroclor, that Aroclor may be analyzed as the CCV instead of 1016/1260. If an Aroclor other than Aroclors 1016/1260 is detected a CCV of the identified Arolcor must be analyzed within 12 hours of the sample. If that CCV does not meet criteria the sample must be reanalyzed under an acceptable CCV using the detected Arolcor. The response factors for the CCV must be within +/- 20 % of the initial calibration RF.

9.2.3. Calibration Acceptance Summary

9.2.3.1. Retention Time Windows: Retention time (RT) windows must be determined for all analytes.

9.2.3.1.1 Initial determination of RT windows.

9.2.4.1.1.1. The center of the retention time (RT) window shall be updated based on the middle level in the initial calibration of the first CCV in the daily sequence, whichever is most recent.

9.2.3.1. Initial Calibration Range. The internal standard calibration technique is employed for this method. The response factor (defined as the ratio of the area to the standard concentration) is calculated for each characteristic peak in the Aroclor 1016/1260 standard at each calibration concentration. The percent relative standard deviation (% RSD) of the response factors for each individual peak in the Aroclor 1016/1260 mix on each column is then determined (both columns must pass calibration criteria).

9.2.3.1.1. Calculate the percent Relative Standard Deviation of the response factors for each compound at each level:

% RSD = (RF Standard Deviation/RF Mean) X 100

RF = Response Factor

9.2.3.1.2. Linear Calibration: If the % RSD is less than 20% over its working range for at least five peaks in the Aroclor 1016/1260 mix, the linearity of the range is assumed for all Aroclors over the same analytical range. Each individual peak's response factor is used for quantitation of all the samples and verification standards. The average of the value calculated for each individual peak is used to report the concentration in the samples.

9.2.3.1.3. Linear Calibration Using Least Squares Regression: If the % RSD is >20% for any given compound, a first order linear regression can be applied to the data to calculate the calibration curve and determine sample concentration. If this method is employed, the r squared value must be > 0.99 for the calibration to be acceptable

9.2.3.1.4. Relative Standard Error: The percent relative standard error (% RSE) from the calibration curve is used to evaluate the initial calibration. This provides a measure of how much error is associated with using the calibration curve for quantitation. The average Relative

Standard Error (RSD for average response factors) of the calibration points from the curve used must be ≤ 20%.

Some data systems will not measure the %RSE from a linear or quadratic fit. For the linear case, the correlation coefficient may be used as an alternative to the %RSE, and must be greater than or equal to 0.990. For the quadratic case the Coefficient of Determination may be used, and must be greater or equal to 0.990.

Note: The Relative Standard Error (RSE) is superior to the Correlation Coefficient (*r*) and Coefficient of Determination (r_2) for testing the fit of a set of calibration points to a line. The lower points on a curve have little effect on *r.* As a result, a curve may have a very good correlation coefficient (>0.990) while also having > 100% error at the low point

9.2.3.2. Initial Calibration Verification (ICV):

9.2.3.2.1. After the initial calibration range has been analyzed, an Initial Calibration Verification standard is analyzed on each column to verify the validity of the initial calibration range. The ICV standard must be from a standard lot independent of the standards used in the initial calibration range. The verification standard for PCBs is the mid-range Aroclor 1016/1260 standard at 1000ppb. (See Section 7.2.7 for details on the preparation of the ICV).

9.2.3.2.2. At least five characteristic peaks of each Aroclor 1016/1260 plus surrogates in the ICV must be checked to verify the Initial Calibration Verification. The calculated concentration of the ICV must be within ±20%D of the expected concentration. Should the %D exceed 20%, the analyst should take corrective action (check standard solution, perform instrument maintenance, etc.) and re-inject the ICV. If the %D still exceeds 20% after a single ICV reinjection, a new Initial Calibration Range must be analyzed.

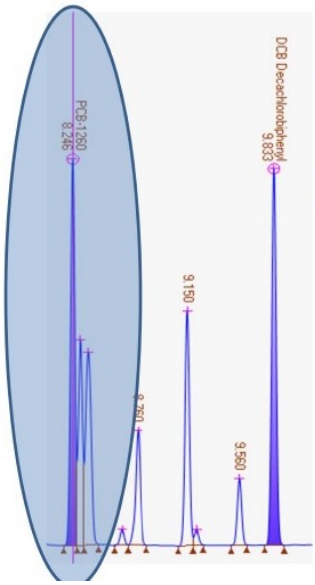
9.2.3.3. Continuing Calibration Verification (CCV) and System Performance Check:

9.2.3.3.1. On each day of analysis the System Performance Check (see Section 7.2.9) is analyzed to assist in the evaluation of possible DDT interferences with the last major peak in Aroclor 1254.

9.2.3.3.2. A mid-point Continuing Calibration Verification (CCV) standard (typically Aroclors 1016/1260) must be analyzed after every 20 samples at minimum. If the samples being analyzed are being specifically targeted for a different Aroclor, that Aroclor may be analyzed as the CCV instead of 1016/1260.

9.2.3.3.3. If there are Aroclor hits in the associated samples then the calculated concentration of the CCV must be within ±20%D of the expected concentration on both columns. If there are no Aroclor hits in the associated samples one column may exceed the ±20%D criteria on the hight side (alternatively a low level Aroclor 1016/1260 standard may be analyzed to demonstrate adequate sensitivity. Should the foregoing criteria not be met the analyst must take corrective action (check standard solution, perform instrument maintenance, etc.) and re-inject the CCV. If the %D still exceeds 20% after a single CCV reinjection, a new Initial Calibration Range must be analyzed. The surrogate (DCB) must also meet CCV criteria on both columns.

9.2.3.3.4. Resolution (Aroclor 1260): The CCV level Aroclor 1016/1260 standard must meet the following resolution critieria: Resolution (degree of overlap) for the triplet towards the end of the 1260 chromatogram must be <75% on <u>one</u> of the two columns used. The resolution requirement must be met for peak 1/2 and peak 2/3:



The circled triplet of peaks is observed towards the end of the 1260 pattern on columns such as CLP 1. Minimum resolution (degree of overlap) requirement between peak 1 / 2 and peak 2 / 3 is <75%. This chromatogram shows overlap of about 50% between peak 2 and 3, and 30% between peak 1 and 2.

Resolution (degree of overlap) is calculated as

[Height of the valley / (Sum of the two peak heights / 2)] x 100%

The acceptance criteria for the Initial Calibration Range, the ICV and the CCV are detailed in the table below.

Step	Standards	Type	Control Limit	Frequency
Method # 8082A				
Initial Calibration Range	50, 500, 1000, 1500 and 2500 ppb for Aroclor 1016/1260, 1000 ppb for all remaining Aroclors	*Average response factor or 1st order linear regression*	*For average RF: <20%RSD all analytes. For linear regression: r^2 ≥0.990*	*Initially and as required when ICV or CCV do not meet requirements*
ICV	*1000 ppb*	*Average*	*± 20%D*	*Once after each initial calibration*
CCV	*1000 ppb*	*Average*	*± 20%D (see Section 9.2.3.4.2 for exceptions); Acceptable Resolution*	*Every 20 samples*
System Performance Standard			*For evaluation of DDT intereference with Ar1254*	*Once daily*

10. Procedure

10.1. Gas Chromatograph (GC) Operation

10.1.1. The sequence of events for GC analysis involves many steps. First the injection system and column performance and calibration must be verified. Maintenance operations are performed as needed. Then samples must be run on the instrument. Chromatograms and reports must be evaluated for content, integration and concentration. Re-runs and dilutions must be made based on the calibrations that were in effect at the time the sample was run. Lastly, a detailed analysis and calculations must be performed to determine the concentration of all the parameters for which the sample was analyzed.

10.1.2. General Instrument Operating Conditions:

10.1.2.1. **Injection System**: A splitless injection port with electronic pressure control (EPC) is used. Seventy-five seconds after sample injection, the purge valve is turned on to facilitate the sweeping of any remaining residual solvent/sample from the injection port.

10.1.2.2. The EPC is used in the pressure Ramp mode. The ramp pressure program is as follows:

Initial Pressure	InitialTime	Rate	Final Pressure	Hold
25 psi	0.50 min	20psi/min	15 psi	2.00 min
		8 psi/min	12 psi	6.60 min
		10.0 min	16 psi	2.00 min

10.1.2.3. For PCB analysis the normal operating conditions of the injection port are as follows:

Injection Temperature:	250^{o}C
Injection Port Pressure: Column flow:	25ml/min 33.2 ml/minute
Split vent flow:	60.0 ml/minute
Purge vent flow:	1.2 ml/minute
EPC:	Ramp pressure mode

10.1.2.4. In addition to the EPC, the injection port is also equipped with a siltek-coated glass double goose neck liner that contains a 1 cm glass wool plug. This liner/glass wool combination provides many functions.

10.1.2.5. The glass wool serves as a heat sink rapidly vaporizing solvent and samples resulting in higher response factors. The liner also protects the column head from accumulation of high boiling residuals and particulates.

10.1.2.6. Regular maintenance is performed on the injection port. When the glass wool/liner is changed, the septa also must be changed. Injection port, oven and detector temperatures are lowered to ambient prior to "cracking" the system. This is so as to introduce a minimum of damaging oxygen molecules into the system.

10.1.2.6.1. After the system has cooled, the old liner is removed. The injection port should be checked for particulate residues and cleaned as needed. A flashlight is usually required for this. After a new liner has been prepared it is placed into the injection port. A graphite seal is placed around the liner. The edges of the seal must be flat, not knife-edged, and free of nicks or burrs. If any of these conditions are not met, the graphite seal must be replaced as well. The graphite seal is critical to proper operation of the injection port. If in doubt, replace it.

10.1.2.6.2. The locking ring on the top of the injection port should be turned, with the wrench, about 1/8 turn past finger tight. The septum nut should never be tightened more than finger tight. After the injection port is reassembled, all

column nuts inside the oven should be checked for leaks using Snoop (Supelco) or another suitable leak tester.

10.1.2.6.3. The septa should be changed each time the injection port is opened. Another routine maintenance operation to improve column performance is the removal of the first 3 cm of the column.

10.1.2.6.4. Once the signal from both detectors has stabilized, it is time to re-heat the zones. The zones should be heated in the order of detectors, oven and then injectors. This is to ensure that volatilized contaminants do not condense on the column or detector.

10.1.2.7. Oven: With the megabore columns installed, temperature programming is employed to achieve higher resolution of compounds and shorter run times than could be accomplished using isothermal methods.

10.1.2.7.1. The oven program and pressure ramping for PCB analysis is employed for all columns as follows:

Initial Temp	Hold Time 1	Rate1	Temp1
164°C	0.0min	12°/min	234°C
Hold Time2	**Rate2**	**FinalTemp**	**FinalTime**
2.4 min	40°/min	325°C	1.5min

10.1.2.8. If the detectors are particularly contaminated, they must be sent to Agilent Technologies in Avondale, Pennsylvania for reconditioning. This should occur if the detector baseline is greater than 100 Hz. Detector reconditioning should be required at a maximum of biannually.

10.1.2.9. Chemstation: The Chemstation is utilized for automation of runs and acquisition. The system is dedicated to a single GC and does not multitask. Therefore, data manipulation cannot be done while sample analysis is in progress. The data system acquires and stores all chromatographic data.

10.1.2.10. Eurofins Chrom data processing software is used for the processing of the chromatography data files. Calibrations, verification standards and samples are processed and reviewed using this database. Chrom is integral to Eurofins LIMS (TALS) which is used to generate all reports.

10.2. Analytical Sequence

10.2.1. The instrument operating conditions should be set as detailed in Section 10.1.

10.2.2. Once instruments conditions have been established, the Initial Calibration Range, calibration verifications and retention time windows must be established Section 9.2.

10.2.3. The analytical sequence is established via the "SEQUENCE" macro of the Chemstation data system. The sequence file contains the name of Method file corresponding to the type of analysis to be performed, the range of samples to be run, and the number of injections per bottle. It is common practice to run the calibration and/or calibration verification standards, evaluate the instrument status, and, finally, (if all meet criteria) complete the Sample Table and Sequence File. If everything else is complete, the run is initiated using the START SEQUENCE soft-key of the SEQUENCE macro

10.2.4. An idealized analytical sequence including an Initial Calibration Range is presented in the table below.

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Idealized Analytical Sequence with Initial Calibration Range	
Injection Number	**Identification**
1	Hexane
2	Instrument Blank
3	Aroclor-1660 Level 1 Cal Std (50 ppb)
4	Aroclor-1660 Level 2 Cal Std (500 ppb)
5	Aroclor-1660 Level 3 Cal Std (1000ppb)
6	Aroclor-1660 Level 4 Cal Std (1500 ppb)
7	Aroclor-1660 Level 5 Cal Std (2500 ppb)
8	Aroclor-1221 Level 3 Cal Std (1000 ppb)
9	Aroclor-1232 Level 3 Cal Std (1000 ppb)
10	Aroclor-1242 Level 3 Cal Std (1000 ppb)
11	Aroclor-1248 Level 3 Cal Std (1000 ppb)
12	Aroclor-1254 Level 3 Cal Std (1000 ppb)
13	Aroclor-1262 Level 3 Cal Std (1000 ppb)
14	Aroclor-1268 Level 3 Cal Std (1000 ppb)
15	Initial Calibration Verification (Aroclor 1660)
16	Hexane
17	Continuing Calibration Verification (Aroclor1660)
18 thru 37	Client samples and QC Samples (MS/MSD, LCS, Method Blank)
19	Continuing Calibration Verification (Aroclor1660) (*every 20 samples*)

10.2.5. After each 20 samples a CCV standard mix must be analyzed. If this standard fails the criteria listed in Section 9.2.3.4, all samples analyzed during the previous period must be re-analyzed with a passing CCV.

10.2.6. PCB Data Reporting: The Chrom data system calculates the concentrations of the selected Aroclor Peaks. The reporting limit is based on the concentration of the lowest standard in the initial calibration, adjusted for the sample wt/vol, final volume, dilution factor and % moisture (No unqualified analytical results or non detects may be reported which correspond to an extract concentration less than the lowest standard in the calibration range).

10.2.7.1. The quantitative values for all confirmed analytes must agree within 40% between the primary column and the confirmation column.

10.2.7.2. If the quantitative values do not agree within 40%, the discrepancy must be noted in the report with a qualification

10.3 Dual Column Approach

NOTE: Data generated under the NJDEP DKQP requires the reporting of the higher concentration in all cases unless it can be demonstrated that interfering compounds are the cause of the higher results in which case the lower value can be reported with a narrative explanation. Other programs may also require a dual column reporting approach different than the one described below in which case the lab will report as required by that program.

10.3.1. The laboratory designates the rear column as the primary column and the front column as the secondary column. Results are reported from the primary column unless the difference in concentration between the two columns results in ≥40% RPD in which case the lower concentration is reported (unless the client or program requirements dictate otherwise).

10.3.2. The values are calculated from the chromatographic peaks that fall within the daily retention time windows established from the most recent preceding calibration verification.

10.3.3. If the calculated values are greater than 40% RPD of each other, report the lower concentration regardless of whether that result is from the primary or secondary column. Report the result with a flag of P*.

10.3.4. If the surrogates on one column are very different (>40% RPD) compared to the other column, this may be indicative of a bad injection or columnar blockage. The sample should be reanalyzed. If similar results are obtained following reanalysis, report the lower of the two numbers and describe the circumstances in the job summary and report case narrative.

10.3.5. If one of the columns fails CCV criteria (but the CCV is between 15%-40% greater than expected value), the sample results shall be reported from the

compliant column. If the recovery falls outside of acceptance criteria on the low side, reanalysis shall be performed.

10.3.6. If the CCV on one of the columns is more than 40% different from the correct value, it can be assumed that there has been significant drift on that column. The sample shall be reanalyzed against an acceptable calibration.

10.3.6.1. An exception to this requirement would be if the CCV recovery on one column fails on the high side and >40% RPD but the associated samples were non-detect for all target analytes on both columns. In this case the non-detect results may be reported from the compliant column.

10.3.7. In some cases where the sample chromatography is complex and has largely varying peaks concentrations, the chromatographic separation may not be sufficient on the 0.53mm ID columns. In this case a confirmatory analysis on an instrument with 0.32 ID columns may be required. The supplemental data produced using analysis on the 0.32mm ID 'microbore' column may minimize overlapping and baseline interference difficulties, and better resolves potential positive identifications. Use of this alternative chromatographic technique shall be noted in the job summary and report case narrative.

10.3.8. In summary, the flow chart in Attachment 1 presents a recommended approach to selecting the better number to report for dual column data. It shall be noted that these recommendations may be overridden by project specific requirements and that they cannot cover all eventualities. The complexity of some data set will require the final decision to be made utilizing the judgment of experienced analysts. In some cases further cleanup steps to remove interferences may be appropriate.

10.4 Extract Cleanup

10.4.1. Cleanup methods are dictated by the original sample matrix and the parameters being determined.

10.4.2. Cleanup of all water samples, if needed, is performed using Sulfuric Acid Permanganate and/or TBA sulfite. Refer to Eurofins Edison SOP No. ED-ORP-021: *The Removal of Elemental Sulfur from Pesticide/PCB Sample Extracts, SW846 Method 3660B,* most current revision and Eurofins Edison SOP No. ED-ORP-022: *Sulfuric Acid Cleanup for PCB Extracts, SW846 Method 3665A*, *SW846 Method 3665A,* most current revision. Blanks must also undergo cleanup following the same procedures as samples..

10.4.3. Cleanup of all soil samples is conducted using TBA sulfite and Sulfuric Acid Permanganate. Blanks must also undergo cleanup following the same procedures as samples.

10.4.4. Cleanup using Sulfuric Acid Permanganate effectively destroys the majority of organic material in the sample extract and should be used

only when PCB is the only analysis to be performed on the sample extract.

10.5 Documentation

10.5.1 Before the analysis sequence is initiated the GC Performance and Repairs logbook must be filled out. It should contain the following information: date, injector temp, oven temp, detector temp, column A flow, column B flow, signal A, signal B, analysts initials, and notes for any necessary repairs.

10.5.2 After samples have been run, each standard and sample must be entered into the Instrument Run Log. The Instrument Run Log should contain the following information: run date, data file name, vial position, sample number, initial volume/weight, final volume, dilution factor method, job number, QA number, extraction date, lab prep batch, target batch signature of analyst at the bottom of each page, lot numbers for standards used, and result of run (O.K., dilution, non-inject, etc.).

11.0. Calculations / Data Reduction

11.1. Accuracy:

$$\text{ICV / CCV, LCS \% Recovery} = \frac{\text{observed concentration}}{\text{known concentration}} \times 100$$

$$\text{MS \% Recovery} = \frac{\text{(spiked sample) - (unspiked sample)}}{\text{spiked concentration}} \times 100$$

11.2. Precision (RPD):

$$\text{Matrix Duplicate (MD)} = \frac{|\text{orig. sample value - dup. sample value}|}{[(\text{orig. sample value + dup. sample value})/2]} \times 100$$

11.3. Calculation of Sample Amounts (Internal Standard Procedure)

11.3.1 Aqueous Samples

$$\text{Concentration } (\mu g/L) = \frac{(As)(Cis)(D)}{(Ais)(RF)(Vs)\,(Vi)\,(1000)}$$

Where:

As	=	Area of the target analyte peak in the sample
Cis	=	Concentration of the internal standard (ug/L)
D	=	Dilution factor, if the sample or extract was diluted prior to analysis. If no dilution is performed, D = 1.
Vi	=	Volume of the extract injected (ul)

Ais	=	Area of the internal standard peak
RF	=	Average response factor from the initial calibration.
Vs	=	Volume of sample extracted (ml)

The 1000 in the denominator represents the number of ul in 1 ml.

11.3.2 Solid Samples

$$\text{Concentration (µg/kg)} = \frac{(As)(Cis)(D)(Vt)}{(Ais)(RF)(Ws)\,(Vi)\,(1000)}$$

Where:

As	=	Area of the target analyte peak in the sample
Cis	=	Concentration of the internal standard (ug/L)
D	=	Dilution factor, if the sample or extract was diluted prior to analysis. If no dilution is performed, D = 1.
Vi	=	Volume of the extract injected (ul)
Ais	=	Area of the internal standard peak
RF	=	Average response factor from the initial calibration.
Vt	=	Volume of concentrated extract (ul)
Ws	=	Weight of sample (g)

The 1000 in the denominator represents the number of ul in 1 ml

11.3.3 Wipe Samples

$$\text{Concentration (µg/kg)} = \frac{(As)(Cis)(D)(Vt)}{(Ais)(RF)(W)\,(Vi)\,(1000)}$$

Where:

As	=	Area of the target analyte peak in the sample
Cis	=	Concentration of the internal standard (ug/L)
D	=	Dilution factor, if the sample or extract was diluted prior to analysis. If no dilution is performed, D = 1.
Vi	=	Volume of the extract injected (ul)
Ais	=	Area of the internal standard peak
RF	=	Average response factor from the initial calibration.
Vt	=	Volume of concentrated extract (ul)
W	=	Wipe

The 1000 in the denominator represents the number of ul in 1 ml

11.4 Relative Response Factors

$$RRF = \frac{A_x}{A_{is}} \times \frac{C_{is}}{C_x}$$

Where:

A_x = Area of target analyte peak
Ais = Area of internal standard peak
Cis = Concentration of internal standard
Cx = Concentration of compound in standard

11.5 Percent Relative Standard Deviation (% RSD):

$$\%\ RSD = \frac{\text{Standard Deviation of RRFs}}{\text{Mean RRF}}$$

11.6 Percent Difference (% D):

$$\overline{\%\ D} = \frac{RRF_c - \overline{RRF_i}}{\overline{RRF_i}} \times 100$$

Where: RRFc = RRF from continuing calibration

$\overline{RRF_i}$ = Mean RRF from current initial calibration

11.7 Relative Standard Error (RSE)

$$\%\ RSE = 100\% \times \sqrt{\frac{\sum_{i=1}^{N}\left[\frac{C_i - PC_i}{C_i}\right]^2}{(N-P)}}$$

Where:

N = Number of points in the curve
P = Number of parameters in the curve (= 1 for average response factor, 2 for linear, 3 for quadratic)
CI = True concentration for level i
PCi = Predicted concentration for level i

11.8 Percent Recovery (% R): Surrogates and Spikes

Concentration (or amount) found

$$\text{Recovery (\%)} = \frac{\text{Concentration (or amount) added}}{\text{Concentration (or amount) added}} \times 100$$

11.9 Dry Weight Correction: All solid samples must be corrected for dry weight using the following formula for dry weight determination

$$DW = \frac{Gd}{Gw} \times 100$$

Where:

DW	=	Percent % Dry Weight
Gd	=	Dry weight of selected sample aliquot
Gw	=	Wet weight of selected sample aliquot

Multiply the DW value times the wet weight of the sample extracted.

NOTE: All dry weight corrections are made in TALS at the time the final report is prepared.

12.0. Method Performance

12.1. Method Detection Limit Study (MDL) The method detection limit (MDL) is the lowest concentration that can be detected for a given analytical method and sample matrix with 99% confidence that the analyte is present. The MDL is determined according to the laboratory's MDL procedure in Section 19 (Test Methods and Method Validation) of Eurofins Edison's Quality Assurance Manual (ED-QA-LQM). MDLs reflect a calculated (statistical) value determined under ideal laboratory conditions in a clean matrix, and may not be achievable in all environmental matrices. The laboratory maintains MDL studies for analyses performed; these are verified at least annually unless method requirements require a greater frequency.

12.2. Lower Limit of Quantitation (LLOQ) (aka Reporting Limit) Verification: The LLOQ is the lowest concentration at which the laboratory has demonstrated target analytes can be reliably measured and reported with a certain degree of confidence, which must be ≥ the lowest point in the calibration curve. The lab verifies the LLOQ annually to demonstrate the capability of quantiation at lower analyte concentrations. The verification is performed by the extraction and analysis of an MDL spike at a concentration of 0.5-2 times the established LLOQ. Recovery of target analytes in the LLOQ verification should be within established in-house limits or within other such project-specific acceptance limits to demonstrate acceptable method performance at the LLOQ. Until the laboratory has sufficient data to determine acceptance limits, the LCS criteria ± 20% (i.e., lower limit minus 20% and upper limit plus 20%) will be used for the LLOQ acceptance criteria. The annual LLOQ verification is completed and documented with the required annual MDL evaluation.

12.3. Demonstration of Capabilities

For DOC procedure refer to Section 19 in the most current revision of Eurofins Edison's Quality Assurance Manual (ED-QA-LQM).

12.4. Training Requirements

Refer to Eurofins SOP No. ED-GEN-022, *Training*, for the laboratory's training program.

13.0. Pollution Control

13.1. Pollution prevention encompasses any technique that reduces or eliminates the quantity or toxicity of waste at the point of generation. Numerous opportunities for pollution prevention exist in laboratory operation. The USEPA has established a prevention hierarchy of environmental management techniques that places pollution prevention as the management option of first choice. Whenever feasible, laboratory personnel should use pollution prevention techniques to address their waste generation. When wastes cannot be feasibly reduced at the source, the agency recommends recycling as the next best option.

13.2. The quantity of chemical purchased should be based on expected usage during its shelf life and disposal cost of unused material. Actual reagent preparation volumes should reflect anticipated usage

14.0. Waste Management

14.1. The U.S. Environmental Protection Agency requires that laboratory waste management practices conducted be consistent with all applicable rules and regulations. All waste will be disposed of in accordance with Federal, State and Local regulations. Where reasonably feasible, technological changes have been implemented to minimize the potential for pollution of the environment. Employees will abide by this method and the policies in section 13 of the Corporate Safety Manual for "Waste Management and Pollution Prevention."

14.2. The following waste streams are generated as a result of this analysis:

- Auto sampler vials and expired standards: These vials are collected in satellite accumulation within the instrument laboratory. The vials are then placed into a 55 steel open top drum in the waste room. When the drums are full, the drum will be collected by the waste vendor for disposal. This waste is treated for incineration.
  Teris Profile Number: 50016652
  Onyx Profile WIP Number: 282493

- Mixed Solvent Waste: Mixed solvent waste is collected in a small beaker inside the bench top hood. This waste is then transferred into the satellite accumulation container in the Organic Prep. Lab. on a daily basis. This

material is transferred into 5 gallon solvent cans as satellite accumulation. These cans are emptied every 24 hours into a steel drum in the waste room. This drum is kept in the walk in hood until it is full. The full drum is then removed from the hood and placed on secondary containment in the waste room.

Teris Profile Number: 50016624
Onyx Profile WIP Number: 545240

- Soil Retain Samples - These samples if not flagged in the system for any hazardous constituents are transferred to poly-lined cubic yard boxes. These boxes when full are sent to stabilization or incineration. These materials are sent out as hazardous for lead and chromium

  Teris Profile Number (incineration): 50016710
  Onyx Profile Number: (stabilization) 402535

15.0. References / Cross-References

15.1. United States Environmental Protection Agency, "Method SW8000C: Determinative Chromatographic Separations,", Test Methods for Evaluating Solid Wastes, SW846, Revision 3, March 2003.

15.2. United States Environmental Protection Agency, "Method 8082A, Organochlorine Pesticide by Gas Chromatography", Test Methods for Evaluating Solid Wastes, SW846 Third Edition, Volume 1B: Laboratory Manual, Physical/Chemical Methods, Revision 1, February 2007.

15.3. Eurofins Edison Document No. ED-QA-LQM, *Laboratory Quality Manual*, most current revision.

15.4. Eurofins Edison SOP No. ED-ORP-014: *Extraction of Pesticides and PCBs in Water by Separatory Funnel, SW846 Method 3510C,* most current revision.

15.5. Eurofins Edison SOP No. ED-ORP-018*: Extraction of Pesticides/PCBs in Soil Using Low-Level Extraction, SW846 Method 3550B,* most current revision.

15.6. Eurofins Edison SOP ED-ORP-044, *Procedure for the Microwave Extraction of Solids, SW846 3546*

15.7. Eurofins Edison SOP No. ED-ORP-019*: Waste Dilution for Pesticides and PCBs, SW846 Method 3580A,* most current revision.

15.8. Eurofins Edison SOP No. ED-ORP-020*: Florisil Cleanup for Pesticide/PCB Sample Extracts*, SW846 Method 3620B, most current revision.

15.9. Eurofins Edison SOP No. ED-ORP-021: *The Removal of Elemental Sulfur from Pesticide/PCB Sample Extracts, SW846 Method 3660B,* most current revision.

15.10. Eurofins Edison SOP No. ED-ORP-022: *Sulfuric Acid Cleanup for PCB Extracts, SW846 Method 3665A*, *SW846 Method 3665A,* most current revision.

15.11. Eurofins Edison SOP No. ED-GEN-022, *Training,* most current revision.

15.12. Eurofins Corporate Work Instruction No. CA-T-WI-003, PCB Minimum Requirements, most current revision.

16.0. Method Modifications:

None

17.0. Attachments

Attachment 1: Dual Column Approach

18.0. Revision History

Revision 7, dated 21 Oct 2022

- Section 4.5 added which details possible DDT and analogs interference with the last major DDT peak.
- Section 7.2.9: Added System Performance Standard (DDT/Endrin) for use in evaluating potential interference of DDT with last major Aroclor 1254 peak.
- Section 9.2.3.3.1: Added requirement to analyze System Performance standard daily for evaluation of DDT interference with Aroclor 1254 quantitation.

Revision 6 dated 17 Oct 2022

- Updated to Eurofins branding, removed TestAmerica references
- Added Section 8.2 to include requirement to treat samples from chlorinated sources with sodium thiosulfate
- Section 9.2.3.1.4: Section 9.2.4.3: Included details of evaluation of initial calibration for Relative Standard Error (RSE).
- Section 11.7: added formula for calculation of RSE.
- Section 12.2 added: details the annual LLOQ verification requirement.

Revision 5 dated 01 Apr 2020

- Updated to Eurofins branding
- Section 7.2.5: added wipe matrix
- Added Section 11.3.3 – calculation for wipe samples

Revision 4 dated 15 Dec 2016

- Section 7.2.8: clarified text to more accurately describe the PCB Internal Standard Mix.

Revision 3 dated 16 May 2016

- Section 2.1: removed SW846 3550 as a prep method for solids and added it as a prep method for wipe samples.
- Section 2.1.1 and 15.0: removed reference to SW846 Method 3520C (SOP No. ED-ORP-003: *Extraction of Semi-Volatile Organic Compounds in Water by Continuous Liquid-Liquid Extraction, SW846 Method 3520C).*
- Section 2.2: expanded to include reference to internal standard calibration.
- Section 7.1.1: revised to clarify that the primary carrier:make-up gas combo is hydrogen and nitrogen.
- Section 7.2.2: added 1-Bromo-2-nitrobenzene (internal standard) to the list of standards (Restek 32279). Components and concentration added to the table
- Added Section 7.2.8 which describes the preparation of the internal standard solution.
- Section 9.1: added internal standard to 'Sample QC' table
- Section 9.1.2: completely re-written to clarify LCS evaluation and corrective action.
- Section 9.1.4: clarified that surrogates must pass CCV criteria in order to be reportable and must be reported from the same column as target analyte results.
- Added Section 9.1.5 which describes acceptance criteria for internal standards (retention time and response)
- Section 9.2.1.1: added following sentence for clarity and compliance with TestAmerica PCB Minimum Requirements document (WI No. CA-T-WI-003): "All other Aroclors are calibrated using a single point calibration up to 8 peaks at the anticipated midpoint of the calibration range if the 1016/1260 calibration meets the required average RF criteria." Added language detailing which Aroclor standards can be analyzed together.
- Section 9.2.1.1: added this sentence: "The initial calibration block must include at least one level with 1016 analyzed separately for pattern recognition purposes (note: this run does not need to be part of the actual calibration)."
- Section 9.2.1.1: clarified that a minimum of five peaks are required for quantitation of each Aroclor (except for Aroclor 1221 which requires a minimum of three peaks). Added requirement that selected peaks must be at least 25% of height of largest peak in Aroclor (except Aroclor 1268 where the requirement is 10%).
- Section 9.2.2: Revised first sentence to read: "A mid-point Continuing Calibration Verification (CCV) standard must be analyzed after every 20 samples at minimum," (i.e., removed the closing CCV requirement and 12 hour requirement).
- Section 9.2.2: clarified that the CCV typically consists of a 1016/1260 standard. Also added the following sentence: "If the samples being analyzed are being specifically targeted for a different Aroclor, that Aroclor may be analyzed as the CCV instead of 1016/1260."
- Section 9.2.2: added requirement to analyze a CCV for each detected Aroclor within 12 hours of detection.

- Table 2: updated with the expected RT acceptance criteria from Minimum Requirements document (0.03 minutes).
- Section 9.2.3.2: Revised 'External standard' to 'Internal Standard'. Emphasized that both columns must pass calibration criteria.
- Section 9.2.3.2.2: revised to require that 5 peaks must be selected (was 3) and evaluated for initial calibration (per corporate Minimum Requirements).
- Section 9.3.3.3.1: corrected section reference for preparation of ICV (was Section 7.2.9; now Section 7.2.7);
- Section 9.2.3.3.2: revised first sentence to read: "At least five characteristic peaks of each Aroclor 1016/1260 plus surrogates in the ICV must be checked to verify the Initial Calibration Verification." (it had previously required three peaks).
- Section 9.2.4.3.1: updated text reflect corporate Minimum Requirements document.
- Added Section 9.2.3.4.2: more fully describes CCV acceptance criteria and corrective actions. Updated table with calibration criteria.
- Added Section 9.2.3.4.3: details resolution requirements per the corporate Minimum Requirements document.
- Section 10.2.5: revised to remove 12 hour requirement (replaced solely with 20 sample requirement) and bracketing requirement. Corrected section reference for CCV acceptance criteria.
- Section 11: completely re-written to include internal standard result calculations and other required QC calculations.
- Section 15 (References): added following document reference: TestAmerica Corporate Work Instruction No. CA-T-WI-003, PCB Minimum Requirements, most current revision.
- Attachment 1: revised completely.

- Revision 2, dated 08 Jun 2015

  - Section 1.1: Corrected Method Limit Group (MLG) reference to 8082 (was incorrectly listed as 8081B,
  - Table 2: updated RLs for leachates from 0.0050 mg/L to 0.00050 mg/L.
  - Throughout document: updated the default initial volumes for aqueous preps (250 ml) and leachate (TCLP/SPLP/ASTM) preps (250 ml).
  - Throughout document: removed any notes referencing option for 'reduced volume' extractions since this is now standard as defined in SOP.
  - Section 2.2.2: replaced SW846 3541 (Soxtherm) prep with SW846 3546 (Microwave) prep.
  - Section 6.2.1 (and throughout document): replaced Target software references with references to TestAmerica's Chrom chromatography data processing software.
  - Section 7.2.2 and throughout document: updated source of standards from Supelco to Restek.
  - Section 7.2.3 and throughout document revised concentration of low calibration standard to 50 ppb.
  - Section 7.29: updated ICV prep instructions using Restek standards.
  - Section 8: updated sample container from 1000ml to 250ml; updated minimum sample size to 250ml.
  - Section 10.3: added note explaining NJDEP DKQP requirement to report the higher concentration in all cases.

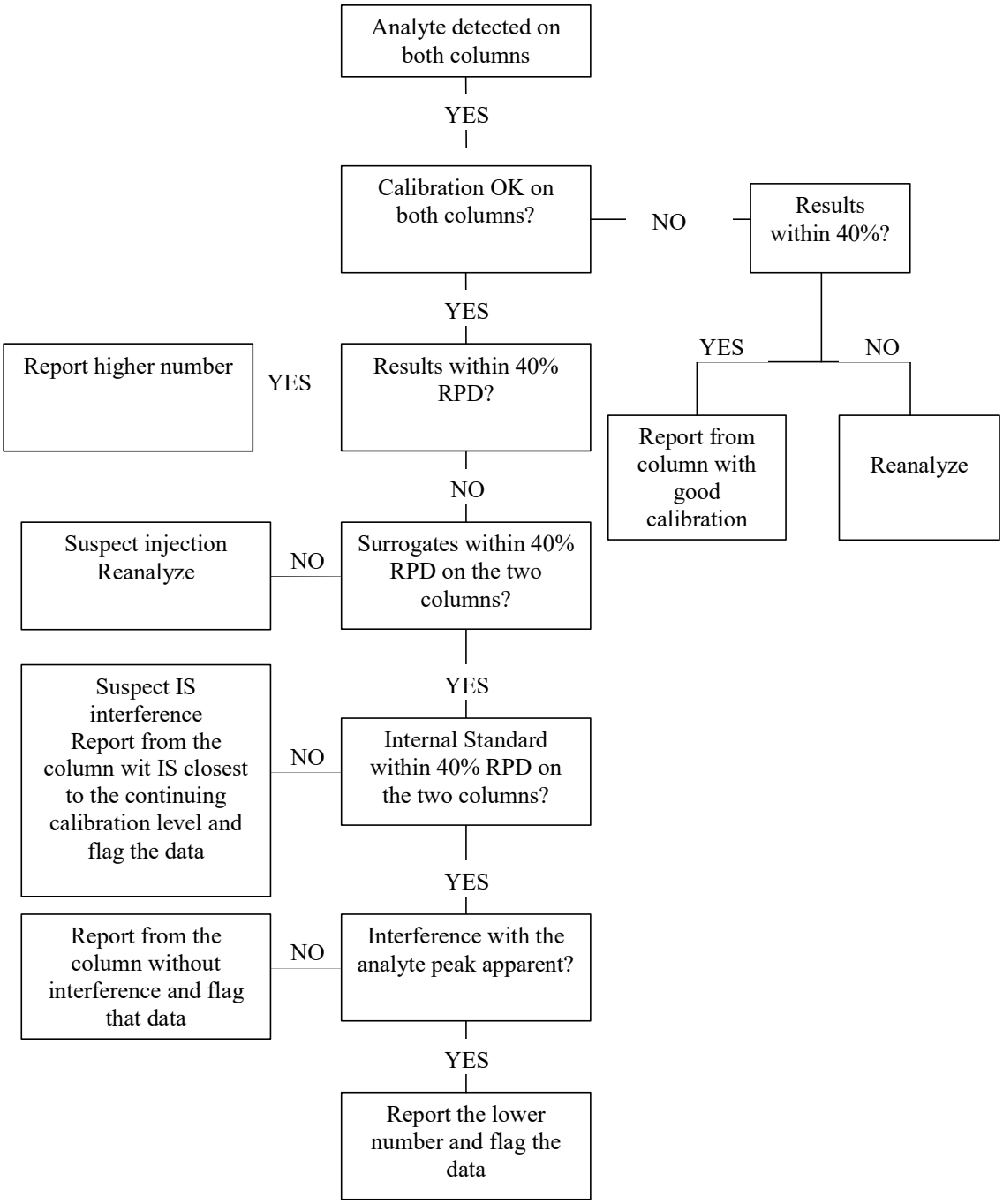
- Section 10.3 and Attachment 1: revised to reflect current TestAmerica dual column reporting rules.
- Section 15: removed outdated references.

- Revision1, dated 11 October 2012

  - Throughout document: Revised LQM section references to reflect the most current LQM revision
  - Revised Table 2 to reflect RLs for reduced initial/final volume prep option.
  - Section 2.1.1: added description of reduced initial volume (125ml)/final volume (1ml) extraction option.
  - Section 2.1.2 and Section 15.0: Added reference to TestAmerica Edison SOP ED-ORP-044, *Procedure for the Microwave Extraction of Solids, SW846 3546.*
  - Section 7.2.3: added procedure for prep and analysis of a lower level Aroclor 1016/1260 standard (50 ppb) and for the surrogate DCB (12.5 ppb) when analyzing samples prepped using the reduced initial/final volume procedure.
  - Section 9.2.3.4.1: Added reference to 50 ppb 1016/1260 ICAL standard for the reduced initial/final volume method.
  - Section 10.2.4: Added 50 ppb 1016/1260 ICAL standard to the sequence for the reduced initial/final volume method

- Revision 0, dated 02/16/2011: NEW

Attachment 1
Dual Column Approach


Analyte detected on both columns
YES
Calibration OK on both columns?
NO
Results within 40%?
YES
NO
YES
Report higher number
YES
Results within 40% RPD?
Report from column with good calibration
Reanalyze
NO
Suspect injection Reanalyze
NO
Surrogates within 40% RPD on the two columns?
YES
Suspect IS interference Report from the column wit IS closest to the continuing calibration level and flag the data
NO
Internal Standard within 40% RPD on the two columns?
YES
Report from the column without interference and flag that data
NO
Interference with the analyte peak apparent?
YES
Report the lower number and flag the data

Title: Semivolatile Organic Compounds by Gas Chromatography/Mass Spectrometry (GC/MS), SW846 Methods 8270E

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Approvals (Signature/Date):

Sylvanus Klusey Organics Operations Manager	10/18/2022 Date	Dan Helfrich Health & Safety Manager	10/18/2022 Date
Carl Armbruster Quality Assurance Manager	10/18/2022 Date	Mark Acierno Laboratory Director	10/18/2022 Date

Diaa Nimer — 10/18/2022 Date
SVOA GC/MS Manager

1.0 Scope and Application

1.1 Analytes, Matrix(s), and Reporting Limits

USEPA Method 8270E is an analytical method which employs the use of GC/MS to determine the concentration of semivolatile organic compounds in extracts prepared from many types of solid waste matrices, soils, and water samples

Eurofins Edison has the capability to analyze and report the compounds listed in Table 1 via Method 8270E.

Table 1

Compound	CAS No.	Compound	CAS No.
1,1'-Biphenyl	92-52-4	Anthracene **(1)**	120-12-7
1,2,4,5-Tetrachlorobenzene	95-94-3	Atrazine	1912-24-9
1,2,4-Trichlorobenzene	120-82-1	Benzaldehyde	100-52-7
1,2-Dichlorobenzene	95-50-1	Benzidine	92-87-5
1,2-Diphenylhydrazine	122-66-7	Benzo[a]anthracene **(1)**	56-55-3
1,3-Dichlorobenzene	541-73-1	Benzo[a]pyrene **(1)**	50-32-8
1,3-Dimethylnaphthalene	575-41-7	Benzo[b]fluoranthene **(1)**	205-99-2
1,4-Dichlorobenzene	106-46-7	Benzo[g,h,i]perylene **(1)**	191-24-2
1,4-Dichlorobenzene-d4 **(ISTD)**	3855-82-1	Benzo[k]fluoranthene **(1)**	207-08-9
1,4-Dioxane **(1) (2)**	123-91-1	Benzoic acid	65-85-0
1-Methylnaphthalene	90-12-0	Benzyl alcohol	100-51-6
1-Naphthylamine	134-32-7	Bis(2-chloroethoxy)methane	111-91-1
2,2'-oxybis[1-chloropropane]	108-60-1	Bis(2-chloroethyl)ether **(1)**	111-44-4
2,3,4,6-Tetrachlorophenol	58-90-2	Bis(2-ethylhexyl) phthalate	117-81-7
2,3,7,8-TCDD	1746-01-6	Bisphenol-A	80-05-7
2,3-Dihydroindene	496-11-7	Butyl benzyl phthalate	85-68-7
2,3-Dimethylaniline	87-59-2	Caprolactam	105-60-2
2,4,5-Trichlorophenol	95-95-4	Carbamazepine	298-46-4
2,4,5-Trimethylaniline	137-17-7	Carbazole	86-74-8
2,4,6-Tribromophenol **(Surrogate)**	118-79-6	Chrysene **(1)**	218-01-9
2,4,6-Trichlorophenol	88-06-2	Chrysene-d12 **(ISTD)**	1719-03-5
2,4-Dichlorophenol	120-83-2	Coumarin	91-64-5
2,4-Dimethylphenol	105-67-9	Dibenz(a,h)anthracene **(1)**	53-70-3
2,4-Dinitrophenol	51-28-5	Dibenzofuran	132-64-9
2,4-Dinitrotoluene	121-14-2	Diethyl phthalate	84-66-2
2,4-Xylidine	95-68-1	Dimethyl phthalate	131-11-3
2,6-Dinitrotoluene	606-20-2	Di-n-butyl phthalate	84-74-2
2-Chloronaphthalene	91-58-7	Di-n-octyl phthalate	117-84-0
2-Chlorophenol	95-57-8	Fluoranthene **(1)**	206-44-0
2-Ethylaniline	578-54-1	Fluorene **(1)**	86-73-7
2-Fluorobiphenyl **(Surrogate)**	321-60-8	Hexachlorobenzene **(1)**	118-74-1
2-Fluorophenol **(Surrogate)**	367-12-4	Hexachlorobutadiene	87-68-3
2-Methylnaphthalene	91-57-6	Hexachlorocyclopentadiene	77-47-4
2-Methylphenol	95-48-7	Hexachloroethane	67-72-1
2-Naphthylamine	91-59-8	Indeno[1,2,3-cd]pyrene **(1)**	193-39-5
2-Nitroaniline	88-74-4	Isophorone	78-59-1
2-Nitrophenol	88-75-5	n,n'-Dimethylaniline	121-69-7
2-tertbutyl-4-methylphenol	2409-55-4	Naphthalene **(1)**	91-20-3
2-Toluidine	95-53-4	Naphthalene-d8 **(ISTD)**	1146-65-2

Table 1

Compound	CAS No.	Compound	CAS No.
3 & 4 Methylphenol	15831-10-4	n-Decane	124-18-5
3,3'-Dichlorobenzidine	91-94-1	Nitrobenzene	98-95-3
3,4-Dimethylaniline	95-64-7	Nitrobenzene-d5 **(Surrogate)**	4165-60-0
3,5-di-tert-butyl-4-hydroxytol	128-37-0	N-Nitrosodimethylamine **(1)**	62-75-9
3-Nitroaniline	99-09-2	N-Nitrosodi-n-propylamine	621-64-7
4,6-Dinitro-2-methylphenol **(1)**	534-52-1	N-Nitrosodiphenylamine	86-30-6
4-Bromophenyl phenyl ether	101-55-3	n-Octadecane	593-45-3
4-chloro-2-methylaniline	95-69-2	o-Toluidine-d9 **(Surrogate)**	194423-47-7
4-Chloro-3-methylphenol	59-50-7	Pentachloronitrobenzene	82-68-8
4-Chloroaniline	106-47-8	Pentachlorophenol **(1)**	87-86-5
4-Chloroaniline–d4 **(Surrogate)**	191656-33-4	Perylene-d12 **(ISTD)**	1520-96-3
4-Chlorophenyl phenyl ether	7005-72-3	Phenanthrene **(1)**	85-01-8
4-Methylphenol	106-44-5	Phenanthrene-d10 **(ISTD**)	1517-22-2
4-Nitroaniline	100-01-6	Phenol	108-95-2
4-Nitrophenol	100-02-7	Phenol-d5 **(Surrogate)**	4165-62-2
Acenaphthene **(1)**	83-32-9	Phenyl ether	101-84-8
Acenaphthene-d10 **(ISTD)**	15067-26-2	Pyrene **(1)**	129-00-0
Acenaphthylene **(1)**	208-96-8	Pyridine	110-86-1
Acetophenone	98-86-2	Terphenyl-d14 **(Surrogate)**	1718-51-0
Aniline	62-53-3	Total Cresols	STL00160
Aniline-d5 **(Surrogate)**	4165-61-1		

(1) Compound can be analyzed by full scan or Selected Ion Monitoring (SIM).
(2) Compound can also be analyzed by Isotope Dilution/SIM.

1.2 For a listing of method detection limits (MDLs) and Reporting Limits (RLs) please refer to the currently active Method 8270 Method Limit Groups in TALS (Eurofins LIMS).

1.3 On occasion clients may request modifications to this SOP. These modifications are handled following the procedures outlined in Section 7 (*Review of Work)*, and Section 19 (*Test Methods and Method Validation)* in Eurofins Edison's Quality Assurance Manual (Eurofins Edison Document No. ED-QA-LQM).

1.4 Any variation in procedure shall be completely documented using an NCM. The NCM is approved by the supervisor and then automatically sent to the laboratory Project Manager by e-mail so that the client can be notified as appropriate. The QA department also receives NCMs by e-mail for tracking and trending purposes. The NCM process is described in more detail in SOP ED-GEN-003. The NCM shall be filed in the project file and addressed in the case narrative. Any unauthorized deviations from this procedure must also be documented as a nonconformance, with a cause and corrective action described.

2.0 Summary of Method

2.1 This method is used for the analysis of aqueous and solid matrices for semi-volatile base, neutral and acid organic compounds that are extracted from the sample matrix with an organic solvent.

2.2 An aliquot of sample containing surrogate spiking compounds is extracted with an organic solvent. The extract is concentrated on a steam bath to a suitable volume. Internal standards are added to the extract.

2.3 Sample extraction techniques are specified for each matrix in the following Eurofins Edison SOPs:

- ED-ORP-002 (*Extraction of Semivolatile Organic Compounds in Water by Separatory Funnel, SW846 Method 3510C*);
- ED-ORP-043 (*SW846 Method 3580A - Waste Dilution Prep for Analysis of BNAs by SW846 Method 8270);*
- ED-ORP-0044 (*Microwave Extraction for Solids, SW846 Method 3546*);

2.4 A small aliquot of the extract is injected into a gas chromatograph (GC) equipped with a capillary column. The GC is temperature programmed to separate the compounds which were recovered during the extraction step by boiling point. The effluent of the gas chromatograph is interfaced to a mass spectrometer (MS) which is used to detect the compounds eluting from the GC. The detected compounds are fragmented with an electron beam to produce a mass spectrum which is characteristic of the compound introduced into the MS. Identification of target analytes is accomplished by comparing their mass spectra with the electron ionization spectra of authentic standards. Quantitation is accomplished by comparing the response of a major ion (quantitation ion) relative to an internal standard established through a five-point calibration (six points for second order regression). Specific calibration and quality control steps are included in the method that must be performed and must meet the specifications of SW846 Method 8270E as applicable.

2.5 The standard preparation procedure for aqueous samples involves use of a Reduced Volume Extraction (250 ml) (RVE) followed by analysis using a Large Volume Injection (LVI). Optionally, a full volume (1000 ml nominal) may be employed. The details of the extractions are outlined in the applicable prep SOPs while the analytical details for 8270E is presented in this SOP.

2.6 These methods are also applicable to the analysis of samples by Selected Ion Monitoring (SIM) for the purpose of obtaining lower reporting limits for the following compounds:

Table 2 – SIM Analytes

SIM Analytes	CAS #
1,4-Dioxane	123-91-1
4,6-Dinitro-2-methylphenol	534-52-1
Acenaphthene	83-32-9

Table 2 – SIM Analytes	
SIM Analytes	**CAS #**
Acenaphthylene	208-96-8
Anthracene	120-12-7
Benzo[a]anthracene	56-55-3
Benzo[a]pyrene	50-32-8
Benzo[b]fluoranthene	205-99-2
Benzo[g,h,i]perylene	191-24-2
Benzo[k]fluoranthene	207-08-9
Bis(2-chloroethyl)ether	111-44-4
Chrysene	218-01-9
Dibenz(a,h)anthracene	53-70-3
Fluoranthene	206-44-0
Fluorene	86-73-7
Hexachlorobenzene	118-74-1
Indeno[1,2,3-cd]pyrene	193-39-5
Naphthalene	91-20-3
N-Nitrosodimethylamine	62-75-9
Pentachlorophenol	87-86-5
Phenanthrene	85-01-8
Pyrene	129-00-0

2.7 An isotope dilution selected ion monitoring (SIM) technique for the analysis of 1,4-dioxane in water at a reporting level of 0.2 ug/l is also described in this SOP. Using this technique 1,4-dioxane-d8 is added prior to sample extraction and is used as an internal standard to calculate the concentration of 1,4-dioxane present. Additionally, 1,4-dichorobenzene-d4 is added to the extract prior to analysis to monitor the recovery of 1,4-dioxane-d8.

3.0 Definitions

For a complete list of definitions refer to Appendix 2 in the most current revision of the Quality Assurance Manual (ED-QA-LQM).

4.0 Interferences

4.1 GC/MS data from all blanks, samples, and spikes must be evaluated for interferences. Analysts must take steps to determine the source of the interference and take corrective action to eliminate the problem.

4.1.1 Contamination by carryover can occur whenever high-concentration and low-concentration samples are sequentially analyzed. To reduce

carryover, the sample syringe is automatically rinsed with solvent between sample injections. Whenever an unusually concentrated sample is encountered, it should be followed by the analysis of a solvent blank to check for cross-contamination. Alternately, verify that the sample analyzed after the high concentration sample does not show any carryover through inspection of chromatogram and target results.

4.1.2 Contaminants from the extraction process detected in the method blank should be evaluated to determine the impact on the analysis. Interferences from any target analyte must not be present in the method blank above the reporting limit for that compound. If these types of interferences occur, corrective action is required. The source should be identified and corrective action initiated to eliminate the interference from the extraction process. Affected samples must be re-extracted and re-analyzed.

4.1.3 The analyst must take precautions to make sure that contaminants do not enter the analytical system. These precautions include systematic procedures designed to eliminate interferences.

4.2 Some compounds analyzed by this method are unstable or sensitive to extraction and/or instrument conditions:

- Benzidine is easily oxidized during extraction. Neutral extraction may enhance the recovery of this compound.
- Hexachlorocyclopentadiene breaks down photochemically and can decompose from high temperatures, particularly in the injection port of the GC. This compound can also react with acetone in solution.
- 1,2-Diphenylhydrazine is unstable even at room temperature and readily converts to azobenzene.
- Phenols are sensitive to active sites and can give a low response or exhibit poor chromatography by tailing. Therefore, it is important the GC is maintained in the best possible condition. See Section 10.1 for proper daily maintenance.
- N-Nitrosodiphenylamine decomposes in the gas chromatographic inlet and cannot be distinguished from diphenylamine.
- 3-Methylphenol cannot be separated from 4-methylphenol by the conditions specified in this method. They are reported as 3 and 4-methylphenol.
- Pyridine may perform poorly at the GC injection port temperatures listed in this SOP. Lowering the injection port temperature may reduce the amount of degradation.

5.0 Safety

Employees must abide by the policies and procedures in the Corporate Environmental Health and Safety Manual (CW-E-M-001) and this document. This procedure may involve hazardous material, operations and equipment. This SOP does not purport to address all of the safety problems associated with its use. It is the responsibility of the user of the method to follow appropriate safety, waste disposal and health practices under the assumption that all samples and reagents are potentially hazardous. Safety glasses, gloves, lab coats and closed-toe, nonabsorbent shoes are a minimum.

5.1. Specific Safety Concerns or Requirements

The gas chromatograph and mass spectrometer contain zones that have elevated temperatures. The analyst needs to be aware of the locations of those zones, and must cool them to room temperature prior to working on them.

The mass spectrometer is under deep vacuum. The mass spectrometer must be brought to atmospheric pressure prior to working on the source.

There are areas of high voltage in both the gas chromatograph and the mass spectrometer. Depending on the type of work involved, either turn the power to the instrument off, or disconnect it from its source of power.

5.2. Primary Materials Used

The following is a list of the materials used in this method, which have a serious or significant hazard rating. **Note: This list does not include all materials used in the method. The table contains a summary of the primary hazards listed in the MSDS for each of the materials listed in the table.** A complete list of materials used in the method can be found in the reagents and materials section. Employees must review the information in the MSDS for each material before using it for the first time or when there are major changes to the MSDS.

Material (1)	Hazards	Exposure Limit (2)	Signs and symptoms of exposure
Methanol	Flammable Poison Irritant	200 ppm-TWA	A slight irritant to the mucous membranes. Toxic effects exerted upon nervous system, particularly the optic nerve. Symptoms of overexposure may include headache, drowsiness and dizziness. Methyl alcohol is a defatting agent and may cause skin to become dry and cracked. Skin absorption can occur; symptoms may parallel inhalation exposure. Irritant to the eyes.
Methylene Chloride	Carcinogen Irritant	25 ppm-TWA 125 ppm-STEL	Causes irritation to respiratory tract. Has a strong narcotic effect with symptoms of mental confusion, light-headedness, fatigue, nausea, vomiting and headache. Causes irritation, redness and pain to the skin and eyes. Prolonged contact can cause burns. Liquid degreases the skin. May be absorbed through skin.
Toluene	Flammable Poison Irritant	200 ppm-TWA 300 ppm-Ceiling	Inhalation may cause irritation of the upper respiratory tract. Symptoms of overexposure may include fatigue, confusion, headache, dizziness and drowsiness. Peculiar skin sensations (e. g. pins and needles) or numbness may be produced. Causes severe eye and skin irritation with redness and pain. May be absorbed through the skin.
Dimethyl-dichloro-silane	Flammable	none	Can be corrosive to the respiratory tract causing severe irritation and tissue damage. Harmful if absorbed through the skin. May cause severe irritation and systemic damage. Severely irritating to the skin and eyes. Harmful if swallowed. Can cause abdominal discomfort, nausea, vomiting, diarrhea, and irritation to the mouth, throat and stomach.
1 – Always add acid to water to prevent violent reactions.			
2 – Exposure limit refers to the OSHA regulatory exposure limit.			

6.0 Equipment and Supplies

6.1 Gas chromatograph/mass spectrometer system

6.1.1 Gas chromatograph: An Agilent/HP 6890/7890/900 Intuvo (or equivalent) houses the capillary column. The GC provides a splitless injection port and allows the column to be directly coupled to the mass spectrometer. The oven is temperature programmable to meet the requirements of the method. An HP/Agilent 7673/7683/7963 autosampler (or equivalent) with a 10 ul syringe provides automatic injection of sample extracts while the instrument is unattended.

6.1.2 Analytical Column: 30m x 0.25mm ID, 0.25 um film thickness, Restek Rxi-5Sil MS, Catalog #13623

6.1.3 Mass spectrometer: Agilent (HP) 5972, 5973, 5975 or 5977A Mass Selective Detector (MSD) Capable of scanning from 35 to 500 amu every 1 sec or less, using 70 volts electron energy in the electron ionization mode. The mass spectrometer must be capable of producing a mass spectrum for 50 ng of decafluorotriphenylphosphine (DFTPP) which meets the criteria in Section 9.2.1 when 2 ul of the 25 ug/ml GC/MS tuning standard is injected through the GC.

6.1.4 GC/MS interface: Any GC-to-MS interface may be used that gives acceptable calibration points at 50 ng per injection for each compound of interest and achieves acceptable tuning performance criteria.

6.1.5 Data system: The data system is interfaced to the mass spectrometer and accommodates continuous acquisition and storage of GC/MS data throughout the duration of the chromatographic program. The data system consists of a Hewlett-Packard Chemstation equipped with Mustang software used for instrument control and data acquisition. This, in turn, is interfaced to Eurofins's Chrom software for data processing. Data from sample extract analysis can be accessed in real-time, while sample data reports and library searches can be performed on data files from previously run samples. The software is also capable of searching any GC/MS data file for ions of a specific mass whose abundances can be plotted versus time or scan number which allows integration of abundances for any extracted ion between specified times or scan-number limits. Library searches utilize a NIST 02.1 Mass Spectral Library.

6.2 Bottles, glass with polytetrafluoroethylene (PTFE)-lined screw caps or crimp tops.

6.3 Injection port liners, splitless

6.4 Injection port septa

6.5 Injection port graphite seals

6.6 Pre-silanized glass wool (Supelco 2-0411 or equivalent)

6.7 Syringes, Assorted sizes 10ul - 1000ul; gas-tight

6.8 Bottles, 10 and 5ml amber screw cap with Teflon liner

6.9 Vials, 2ml amber screw cap with Teflon liner

6.10 Wheaton microvials 100ul (or equivalent)

6.11 Volumetric Flasks, Class A with ground glass stoppers (2ml - 100ml)

6.12 Analytical balance, ASP Model SP-180 (or equivalent), capable of accurately weighing to 0.0001 gr.

7.0 Reagents and Standards

The following items are recommended for performing this procedure. Equivalent items should only be used when they result in an improvement in quality, efficiency, productivity, or cost. An item can be considered equivalent if with its use, the analytical and QA/QC requirements in this SOP can be met. Please refer to the MSDS prior to the use of any reagent or standard.

The preparation of standards, surrogates and spiking solutions is documented in the TALS Reagent Module. Formulary reports can be generated upon request.

7.1. Reagents:

7.1.1. Methylene Chloride: J.T.Baker Resi-Analyzed, used for Organic Residue Analysis (P/N 9266-V8 or equivalent).

7.1.2. Methanol: J.T.Baker Purge and Trap Grade (P/N 9077-02 or equivalent).

7.1.3. Sylon-CT: Supelco (P/N 33065-U or equivalent). Sylon-CT is a highly reactive silanizing reagent consisting of 95% Toluene and 5% Dimethyldichlorosilane (DMDCS).

7.1.4. Each lot of solvent is screened for contaminants before being used for analysis as detailed in Eurofins Corporate Quality SOP No. CA-Q-S-001 (*Solvent & Acid Lot Testing & Approval*) and Eurofins Edison SOP No. ED-GEN-023 (*Bulk Solvent Testing and Approval*).

7.2. Standards:

7.2.1. **Calibration Standards (Full Scan Analysis)**: Stock analytical standard solutions are purchased mainly from Restek Corporation. Other standards are prepared in the laboratory as needed using neat compounds or prepared solutions purchased from Agilent, SPEX CertiPrep, Chem Service,

Accustandard, Supelco or other suppliers. Standards prep instructions are detailed for the following full scan analyte list options:

- Full Volume Aqueous Prep; and,
- Reduced Volume Aqueous Prep and Soils

Secondary dilutions are either made from purchased stock solutions as listed below or from prepared solutions as listed in the following table:

NOTE: Second sources (from certified separate lots) are used for ICV standards.

Table 3 – Full Scan Stock Standards			
Target Analyte Standard Name	**Conc. (PPM)**	**Vendor**	**Catalog #**
1,2,3,4-TCDD	50	SPEX	SVO-TANJ-12
Agilent Mix (contains compounds listed in Table 4 below)	2000 *	Agilent	Cus 0456
8270 List 1/ Std #1 Megamix	Varied	Restek	571995
8270 List 1/ Std#9	2000	Restek	569730
8270 List 1/ Std#11	2000	Restek	569732
8270 Surrogate Standard	5000*	Restek	567685
8270 Internal Standard	2000	Restek	567684
8270 List 1/ Std#10	2000	Restek	569731
Bisphenol-A	1000	Agilent	Cus-0457

*Agilent Mix, 8270 list1/std#9 and 8270 Surrogate standard are diluted to 100ppm prior to the preparation of the 1.0ppm and 0.5ppm standards.

Table 4	
Agilent Mix Catalog No. Cus-0456	
Analyte	**Concentration (PPM)**
Pentachloronitrobenzene	2000
2 -tert-butyl-4-Methylphenol	2000
2,6-Di-tert-butyl-4-Methylphenol	2000
Coumarin	2000
Phenyl ether	2000
N,N'-Dimethylaniline	2000
N-Methylaniline	2000
Carbamazepine	2000
Benzonitrile	2000
1,3-Dimethylnaphthalene	2000

7.2.1.1. Individual calibration standards for full scan analysis are prepared in one of several ways depending upon the technique (full volume aqueous prep, soils prep, reduced volume prep with LVI) as well as the target analyte list. The following tables detail the preparation of calibration standard solutions for each of these techniques and analyte lists. Prepare by combining the indicated volumes of each stock solution using volumetric flask. Dilute to the volume marker with methylene chloride.

Table 5
Full Volume Aqueous Prep and Soils
Working Standards Preparation

Solution Name	120 PPM	80 PPM	50 PPM	20 PPM	10 PPM	5 PPM	2 PPM	1 PPM	0.5 PPM
8270 List 1/ Std #1 Megamix	3000 ul	2000 ul	2500 ul	500ul	250ul	250ul	100ul	50ul	25ul
8270 List 1/ Std #9	1500 ul	1000 ul	1250 ul	250ul	125ul	125ul	50ul	500ul*	250ul *
8270 List 1/ Std #10	1500 ul	1000 ul	1250 ul	250ul	125ul	125ul	-	-	-
Agilent custom Mix	1500 ul	1000 ul	1250 ul	250ul	125ul	125ul	50ul	500ul*	250ul *
1,2,3,4-TCDD	-	-	500ul	-	-	-	-	-	-
8270 Surrogate Standard	600ul	400ul	500ul	100ul	50ul	50ul	20ul	500ul*	250ul *
8270 Internal Standard	500ul	500ul	1000 ul	500ul	500ul	1000 ul	1000 ul	1000 ul	1000 ul
Bisphenol-A	3000 ul	2000 ul	2500 ul	500ul	250ul	250ul	100ul	-	-
8270 List 1/ Std #11	400ul	300ul	500ul	200ul	125ul	125ul	50ul	25ul	-
Final Volume (ml)	**25**	**25**	**50**	**25**	**25**	**50**	**50**	**50**	**50**

Note: The 1.0ppm and 0.5pmm standards (above) are prepared using the 100ug/ml standard for Agilent custom Mix, 8270 List1/std#9 and 8270 Surrogate Standard.

Table 6
Reduced Volume Extraction/LVI
Working Standards Preparation

Solution Name	24 PPM	16 PPM	10 PPM	4 PPM	2 PPM	1 PPM	0.4 PPM	0.2 PPM	0.1 PPM
120 ppm (see Table 5)	2.0mL								
80 ppm (see Table 5)		2.0 mL							
50 ppm (see Table 5)			2.0 mL						
20 ppm (see Table 5)				2.0 mL					

Table 6 Reduced Volume Extraction/LVI Working Standards Preparation									
Solution Name	**24 PPM**	**16 PPM**	**10 PPM**	**4 PPM**	**2 PPM**	**1 PPM**	**0.4 PPM**	**0.2 PPM**	**0.1 PPM**
10 ppm (see Table 5)					2.0 mL				
5.0 ppm (see Table 5)						2.0 mL			
2.0 ppm (see Table 5)							2.0mL		
1.0 ppm (see Table 5)								2.0 mL	
0.5 ppm (see Table 5)									2.0mL
Final Volume (ml)	**10**	**10**	**10**	**10**	**10**	**10**	**10**	**10**	**10**

7.2.1.2. **Initial Calibration Verification (full scan)**: Second source ICVs for full scan analysis are prepared in one of several ways depending upon the technique (full volume aqueous prep, soils prep, reduced volume prep with LVI) as well as the target analyte list. The following tables detail the preparation of ICVs for each of these techniques and analyte lists. Prepare by combining the indicated volumes of each stock solution using volumetric flask. Dilute to the volume marker with methylene chloride.

Table 7 8270/625 ICV Working Standards Preparation	
Solution Name	**25 PPM**
8270 List 1/ Std #1 Megamix (2nd Lot)	250ul
8270 List 1/ Std #9 (2nd Lot)	125ul
8270 List 1/ Std #10 (2nd Lot)	125ul
Agilent custom Mix (2nd Lot)	125ul
8270 Internal Standard	200ul
8270 List 1/ Std#11	125ul
Bisphenol-A (2nd Lot)	250ul
Final Volume (ml)	**10**

7.2.1.3. **Surrogate Standards (Full Scan Analysis):** A 5000ppm Surrogate Standard is purchased from Restek for use in spiking blanks, samples and associated QC prior to extraction (reference the applicable sample prep SOPs for spiking instructions).

Table 8 Full Scan Surrogate Standards Solution Restek Catalog No. 567685	
Surrogate Standard Compounds	**Concentration (PPM)**
Nitrobenzene-d5	5000
p-Terphenyl-d14	5000
2,4,6-Tribromophenol	5000
Phenol-d5	5000
2-Fluorobiphenyl	5000
2-Fluorophenol	5000

7.2.1.4. **Internal Standards** (**Full Scan Analysis**): The Internal Standards Solution at 2000ppm is purchased from Restek (Catalog # 567684). The Internal Standard solution is stored in 10ml amber screw cap bottles with Teflon liners in the dark at 4°C. The Internal standard solution is used in preparing all analytical standards. Inject 20ul of this solution (2000ppm) per ml of sample extract prior to analysis resulting in a concentration of 40ppm (ug/ml) in the extract.

Table 9 Full Scan Internal Standards Solution Restek Catalog No. 567684	
Internal Standard Compounds	**Concentration (PPM)**
1,4-Dichlorobenzene-d4	2000
Phenanthrene-d10	2000
Naphthalene-d8	2000
Chrysene-d12	2000
Acenaphthene-d10	2000
Perylene-d12	2000

7.2.2. **Calibration Standards (SIM analysis)**: The Edison lab currently analyzes only a select list of compounds by 8270E SIM (see Sections 1.0 and 2.0). Stock analytical SIM standard solutions are purchased mainly from Agilent. Working standards are prepared from these solutions as listed in the tables in Section 7.2.2.1:

Table 10 Stock SIM Standards			
Standard Name	**Concentration**	**Vendor**	**Catalog #**
Pentachlorophenol	1000ppm	AGILENT	PH-180-1
n-Nitrosodimethylamine	100ppm	AGILENT	NS-100-1
Hexachlorobenzene	100ppm*	AGILENT	CH-151-1
PAH Mix	100ppm	AGILENT	PAH-605-1
Bis(2-chloroethyl)ether	100ppm*	AGILENT	BEC-110-1
4,6-Dinitro-2-methylphenol	1000ppm**	AGILENT	PH-150
1,4-Dioxane	1000ppm**	AGILENT	NV-152-1

*Hexachlorobenzene and Bis(2-chloroethyl)ether are diluted to 10ppm prior to SIM Standards prep

** 4,6-Dinitro-2-methylphenol and 1,4-Dioxane is diluted (10x) to 100ppm prior to SIM Standards prep

NOTE: Second sources (from separate lots are used for ICV standards).

7.2.2.1 Individual calibration standards for SIM analysis are prepared in one of two ways depending upon the technique (full volume aqueous prep or reduced volume prep with LVI) as well as the target analyte list. The following tables detail the preparation of calibration standard solutions for each of these techniques and analyte lists. Prepare by combining the indicated volumes of each stock solution using volumetric flask. Dilute to the volume marker with methylene chloride.

Table 11
Full Volume Aqueous Prep – SIM
Working Standards Preparation

	0.025 PPM	0.05 PPM	0.1 PPM	0.5 PPM	1.0 PPM	5.0 PPM
Pentachlorophenol	2.5uL	2.5uL	12.5uL	10uL	20uL	50uL
n-Nitrosodimethylamine	25uL	25uL	125uL	100uL	200uL	500uL
PAH mix	6.25uL	5uL	25uL	50uL	100uL	200uL
Hexachlorobenzene	25uL	25uL	250uL	1000uL	2000uL	500uL*
Bis(2-chloroethyl)ether	25uL	25uL	250uL	1000uL	2000uL	500uL*
4,6-dinitro-2-methylphenol	50ul	50ul	250ul	200ul	400ul	1000ul
1,4-Dioxane	25ul	50ul	250ul	200ul	400ul	1000ul
ISTD	500uL	200uL	500uL	200uL	200uL	200uL
Final Volume (ml)	**25**	**10**	**25**	**10**	**10**	**10**

*For Hexachlorobenzene and Bis(2-chloroethyl)ether the 5.0 ppm level is prepared using the 100ppm standard.

Table 12
Reduced Volume Extraction/LVI – SIM
Working Standards Preparation

	0.005 PPM	0.01 PPM	0.02 PPM	0.10 PPM	0.20 PPM	1.0 PPM
0.025 PPM Std (see Table 11)	1.0 mL					
0.05 PPM Std (see Table 11)		1.0 mL				
0.1 PPM Std (see Table 11)			1.0 mL			
0.5 PPM Std (see Table 11)				1.0 mL		
1.0 PPM Std (see Table 11)					1.0 mL	
5.0 PPM Std (see Table 11)						1.0 mL
Final Volume (ml)	**5**	**5**	**5**	**5**	**5**	**5**

7.2.2.2 Initial Calibration Verification (SIM): A 0.1 ppm separate lot SIM ICV is prepared as detailed in Table 13 using the stock standards detailed in Section 7.2.2 (above)

Table 13 0.1ppm SIM ICV preparation	
Pentachlorophenol	25uL
n-Nitrosodimethylamine	25uL
PAH mix	5uL
Hexachlorobenzene	5uL
1,4-Dioxane	5ul
4,6-Dinitro-2-methylphenol	100ul
ISTD	**100uL**
Final Volume	**5 ml**

7.2.2.3 Internal Standard solution (SIM): A 50 ppm Internal Standard solution for SIM analysis is prepared by adding 125ul of the 2000ppm stock ISTD (see Section 7.2.1.4) and bringing to volume with Methylene Chloride in a 5ml volumetric flask.

7.2.2.3.1 For SIM analysis inject 20ul of this solution (50ppm) per ml of sample extract prior to analysis resulting in a concentration of 1ppm (ug/ml) in the extract.

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7.2.3. Calibration Standards (Isotope Dilution SIM – 1,4-Dioxane): The Edison lab currently analyzes only for 1,4-dioxane by 8270E isotope dilution SIM (see Sections 1.0 and 2.0). Stock analytical isotope dilution SIM standard solutions are purchased mainly from Accustandard and Restek. Working standards are prepared from these solutions as listed in the tables below.

Table 14 - Stock 1,4-Dioxane Isotope Dilution SIM Standards			
Standard Name	**Concentration**	**Vendor**	**Catalog #**
1,4-Dioxane	1000ppm*	Accustandard	APP-9-096

* 1,4-Dioxane is diluted (10x) to 100ppm prior to SIM Standards prep

Table 15 - Stock Labeled 1,4-Dioxane SIM Surrogate/Internal Standard (added at prep)			
Standard Name	**Concentration**	**Vendor**	**Catalog #**
1,4-Dioxane-d8	2000ppm	Restek	30614

Table 16 - Stock 1,4-Dioxane Isotope Dilution SIM Internal Standard (added to extract)			
Standard Name	**Concentration**	**Vendor**	**Catalog #**
1,4-Dichlorobenzene-d4	2000ppm	Accustandard	AZ-014J-3

7.2.3.1 Individual calibration standards for 1,4-dioxane isotope dilution SIM analysis are prepared at the concentrations detailed in the following tables. Prepare by combining the appropriate volumes of each stock solution using volumetric flask. Dilute to the volume marker with methylene chloride.

Table 17 Reduced Volume Extraction/LVI – 1,4-Dioxane Isotope Dilution SIM ICAL Standard Concentrations (ug/ml)									
	Lev 1	**Lev 2**	**Lev 3**	**Lev 4**	**Lev 5**	**Lev 6**	**Lev 7**	**Lev 8**	**ICV***
1,4-Dioxane	0.02	0.04	0.1	0.2	0.5	1	2	10	0.2
1,4-Dioxane-d8	4	4	4	4	4	4	4	4	4
1,4-Dichlorobenzene-d4	0.2	0.2	0.2	0.2	0.2	0.2	0.2	0.2	0.2

***:** The ICV is prepared from the second source stock in Table 13.

7.2.4. GC/MS Instrument Performance Check (DFTPP): The DFTPP standard is prepared by is prepared at 25 ppm by adding 2.5ml of EPA 8270 GC/MS Tuning Solution II (Restek Catalog # 31615) to a 100ml volumetric flask and bringing to volume with Methylene Chloride.

7.2.5. Information on prepared standard solutions must be recorded in the TALS Reagent Module. Information such as standard supplier, lot number, original concentration, a description of how the standard was made, are required along with the laboratory lot number, analyst's initials, date prepared, expiration date and verification signature. Standards must be remade every 6 months, or sooner, if the standards expire or begin to show signs of unacceptable degradation. Class "A" volumetric must be used at all times and syringes, preferably gas-tight syringes when available, should be checked for accuracy using an analytical balance. Class "A" pipettes should also be used if volumes permit.

7.2.6. Please refer to Eurofins Edison SOP No. ED-GEN-008, Standard Operating Procedure for Preparation, Purity and storage of Reagents and Standards.

- Shelf Life of Standard: 1 year after preparation or stock standard manufacture expiration, whichever comes first;
- Storage Requirements: Stock standards are stored at 4°C and Working Standards stored at -10°C to -20°C.

8.0 Sample Collection, Preservation, Shipment and Storage

8.1 Samples from chlorinated water sources must be treated with sodium thiosulfate (0.008% solution) at the time of collection to remove chlorine. NOTE: containers pre-preserved with sodium thiosulfate must be requested in bottle orders for samples from chlorinated water sources.

8.2 All samples must be stored at 4ºC (± 2ºC) upon receipt.

8.3 Sample Extract Storage. Samples extracts must be protected from light and refrigerated at 4ºC (± 2ºC) from time of extraction until analysis.

8.4 Sample Extract Holding Time. All sample extracts must be analyzed within 40 days of extraction.

Matrix	Sample Container	Min. Sample Size	Preservation	Holding Time	Reference
Waters	Amber glass, 1L	1000 ml or 250 ml (1)	Cool 4 ± 2ºC	7 days to extraction; Analyze within 40 days of extraction	EPA Method SW846 8270E
Solids	Wide mouth glass, 8 or 16 oz.	50g	Cool 4 ± 2ºC	14 days to extraction; Analyze within 40 days of extraction	EPA Method SW846 8270E

(1) : Reduced volume extraction (RVE) LVI option

9.0 Quality Control

9.1. **Sample QC** - The following quality control samples are prepared with each batch of samples.

Quality Controls	Frequency	Control Limit
Method Blank (MB)	1 in 20 or fewer samples	< Rpt. Limit
Laboratory Control Sample (LCS)[1]	1 in 20 or fewer samples	Statistical Limits [4]
Matrix Spike (MS)[2]	1 in 20 or fewer samples	Statistical Limits [4]
MS Duplicate (MSD)[2]	1 in 20 or fewer samples	Statistical Limits [4]
Surrogates	every sample[3]	Statistical Limits [4]
Internal Standards	Every sample	Response within -50% to +100% of CCV

[1] LCS Duplicate (LCD) is performed only when insufficient sample is available for the MS/MSD or when requested by the client/project/contract.

[2] The sample selection for MS/MSD are randomly selected, unless specifically requested by a client….predetermined by the extraction lab.
[3] Analytical and QC samples (MB, LCS, MS/MSD, Method Blank)
[4] Statistical control limits are determined annually and are updated into TALS limit group..

9.1.1. Method blanks are extracted with every sample batch on each day that samples are extracted. To be considered acceptable, the method blank must contain less than the reporting limit of all target compounds except for phthalates, which can be present at up to 5x the MDL. For method 8270E the method blank is generally acceptable if target analyte concentrations are less than the one half the reporting limit.

If method blanks are unacceptably contaminated with target compounds that are also present in field samples, all affected samples must be re-extracted and re-analyzed. Corrective action must be taken to identify and eliminate the contamination source. Demonstrate that acceptable blanks can be obtained before continuing with sample extraction and analysis. Method blanks must be analyzed on each instrument on which the associated samples are analyzed.

9.1.1.1. Surrogate recoveries for the method blank are compared to laboratory generated limits. If two or more surrogates for any one fraction (base-neutral or acid) are outside of recovery limits or if any one surrogate recovers at <10%, the sample must be re-extracted and re-analyzed to confirm matrix interference.. If any surrogate is still outside limits, all samples and QC samples associated with that method blank must be re-extracted (volume permitting).

9.1.2. Matrix Spike (MS)/Matrix Spike Duplicate (MSD): A matrix spike/matrix spike duplicate (MS/MSD) pair is extracted and analyzed with every 20 environmental samples of a specific matrix (defined as a sample batch). Full compound list spiking is employed for MS/MSDs and LCSs. These spikes are prepared and extracted concurrent with sample preparation. MS and MSD recoveries are calculated and compared to lab generated acceptance criteria. See the current active TALS 8270 Method Limit Group for QC limits. The MS/MSD spiking solution should the same as used for the calibration standards.

9.1.2.1 A Laboratory Control Sample Duplicate (LCSD) is extracted and analyzed only when insufficient client sample is available for preparation of an MS/MSD pair. The LCS/LSCD is evaluated in the same manner as the MS/MSD (see Section 9.1.2)

9.1.2.2 An LCS/LCSD may be substituted for the MS/MSD if insufficient sample volume is available.

9.1.3. Laboratory Control Sample (LCS)/Laboratory Control Sample Duplicate (LCSD): A Laboratory Control Sample (LCS) (aka blank spike) must be extracted and analyzed with each batch of 20 environmental samples. The LCS data is used to assess method performance if the MS/MSD recoveries fall outside of the lab generated limits (See the current active TALS 8270 Method Limit Group for QC limits). If the LCS recovery is within the current lab generated limits, the MS/MSD recoveries are attributed to matrix interference.

9.1.3.1 A Laboratory Control Sample Duplicate (LCSD) is extracted and analyzed only when insufficient client sample is available for preparation of an MS/MSD pair. The LCS/LSCD is evaluated in the same manner as the MS/MSD (see Section 9.1.2)

9.1.3.2 Spike recovery limits are lab generated and are updated annually. Certain state regulatory programs have defined recovery limits which, where applicable, are used for spike recovery evaluations. The TALS Method Limit Groups detail these regulatory program criteria.

9.1.4. Surrogate Standards: All full scan samples, blanks and QC samples are spiked with a six (6) component surrogate standard mix (see Section 7.2.1.3). The percent recovery of the surrogate standards is calculated and compared to lab generated limits (See the current active TALS 8270 Method Limit Group for QC limits).

If any two or more surrogates for any one fraction (base-neutral or acid) are outside of recovery limits or if any one surrogate recovers at <10%, the sample must be re-extracted and re-analyzed to confirm matrix interference. If a surrogate is diluted to a concentration below that of the lowest calibration standard, no corrective action is necessary.

9.1.4.1 Surrogate recovery limits are lab generated and are updated annually. Certain state regulatory programs have defined recovery limits which, where applicable, are used for spike recovery evaluations. The TALS Method Limit Groups detail these regulatory program criteria.

9.1.5. Internal Standards: The response (area count) of each internal standard in the sample must be within -50 +100% of its corresponding internal standard in the CCV or, the ICAL midpoint for samples analyzed under the initial calibration range. Failure to meet these criteria is indicative of sample matrix effects. All samples failing these criteria must be reanalyzed to confirm matrix effects.

9.2. Instrument QC

9.2.1 GC/MS Instrument Performance Check (DFTPP): (**Note**: the DFTPP performance check applies only to full scan analyses and is not evaluated for SIM analysis). The GC/MS system is tuned using Perfluortributylamine

(PFTBA) such that an injection of 50ng of Decafluorotriphenylphosphine (DFTPP) meet the abundance criteria listed in the table below. Prior to the analysis of any calibration standards or samples, the GC/MS system must meet all DFTPP key ion abundance criteria. This analysis will verify proper tuning of the system for a period of 12 hours post-injection. After 12 hours, the instrument performance must again be verified prior to the analysis of standards, QC or samples. Daily tune verification is not required for 8270E CCV.

DFTPP Key Ions and Abundance Criteria	
Mass	**Ion Abundance Criteria**
51	30-60% of mass 198
68	<2% of mass 69
69	reference only
70	<2% of mass 69
127	40-60% of mass 198
197	<1% of mass 198
198	Base Peak, 100% relative abundance
199	5-9% of mass 198
275	10-30% of mass 198
365	>1% of mass 198
441	present but less than mass 443
442	>40% of mass 198
443	17-23% of mass 442

9.2.1.1. Evaluate DFTPP using three scan averaging and background subtraction techniques. Select the scan at the peak apex, add +1 scan from the apex and -1 scans from the apex.

9.2.1.2. The mass spectrum of DFTPP may be background subtracted to eliminate column bleed or instrument background ions. Background subtract DFTPP by selecting a scan for subtraction ≤20 scans before the apex scan of DFTPP.

9.2.1.3. Check column performance using pentachlorophenol and the benzidine peaks (these compounds are included in the DFTPP solution). Benzidine & Pentachlorophenol should respond normally without significant peak tailing (Tailing Factor should be <2 measured at 10% peak height). If responses are poor and excessive peak tailing is present, corrective action for the GC/MS instrument may be required. Corrective actions may include:

9.2.1.3.1 Retune the GC/MS;
9.2.1.3.2 Clip the injector end of the GC column;
9.2.1.3.3 Replace the septum and injection port liner;
9.2.1.3.4 Change the injection port seal;

9.2.1.3.5 Replace the GC column;
9.2.1.3.6 Clean the injection port with MeCl2
9.2.1.3.7 Clean the MS ion source;
9.2.1.3.8 Place a service call.

9.2.1.4. The breakdown of 4, 4-DDT into 4,4-DDD and 4,4'DDE may also be used to assess GC column performance and injection port inertness. If so evaluated the breakdown must be <20%.

9.2.1.5. DFTPP parameter settings are stored in a tune file, which will be used in all subsequent analysis of standards and sample extracts.

9.2.2 Initial Calibration Range and Initial Calibration Verification

9.2.2.1. Initial Calibration: The initial calibration range consists of a minimum of five concentration levels of analytical standards (six for second order regression) prepared as described in Section 7.2. and analyzed once the DFTPP instrument performance check has met the criteria in Section 9.2.1. .

9.2.2.2. Initial Calibration Verification (ICV): An Initial Calibration Verification (ICV) standard is analyzed immediately after the Initial Calibration Range and before any samples are analyzed. The ICV is prepared as detailed in Section 7.2. The ICV must be from a source (or lot) separate from the standards used in the Initial Calibration Range.

9.2.3 Continuing Calibration Verification (CCV) and Low Level Continuing Calibration Verification (LLCCV): A mid-point Continuing Calibration Verification (CCV) must be analyzed every 12 hours after the DFTPP instrument performance check (when applicable).. The CCV is prepared as detailed in Section 7.2. (typically, 50 ug/ml for full volume aqueous and soils, 10 ug/ml for LV, 0.02 ug/ml for LVI SIM) and 0.2 for isotope dilution SIM). Additionally a Low Level Continuing Calibration Verification (LLCCV) is analyzed after the CCV for full scan analysis. The LLCCV is the same as the lowest calibration level analyzed with the initial calibration range (See Section 7.2).

9.2.4 Calibration Acceptance Summary

9.2.4.1. Retention Time Windows: Retention time windows must be established to compensate for minor shifts in absolute retention times as a result of sample loading and normal chromatographic variability. Obtain the retention time for all compounds from the analysis of the midpoint standard for the calibration curve. Establish the center of the retention time window by using the absolute retention time for each analyte, internal standard and surrogate from the calibration verification standard at the beginning of the analytical shift. For samples run during the

same shift as an initial calibration, use the retention time of the mid-point standard of the initial calibration. For qualitative identification to be acceptable the retention time of the relative retention time (automatically calculated in Chrom) must be within 0.8 - 1.2 RRT units of its assigned internal standard. The relative retention times of each compound in the five calibration standards must agree within .06 relative retention time units.

9.2.4.2. Initial Calibration Range: Internal standard calibration is employed for this method. After the initial calibration range has been analyzed the relative response factor (RRF) for each target/surrogate compound at each concentration level is determined using the following equation.

$$RRF = \frac{A_x}{A_{is}} \times \frac{C_{is}}{C_x}$$

Where:
A_x = Area characteristic ion (see Table 21) for the compound
Ais = Area characteristic ion (see Table 21) of associated internal standard
Cis = Concentration of internal standard
Cx = Concentration of compound in standard

9.2.4.2.1. Determine the mean RRF for each compound. Minimum response factors must be met for each of the compounds listed in Table 18 (below). Any compound that fails the minimum response factor must be reported as estimated for detects and must have a demonstration of sensitivity in the analytical batch to report non-detects. To demonstrate adequate sensitivity for out of criterion compounds analyze the low level point of the calibration (LLCCV) in the analytical sequence. The criterion for the LLCCV is detection only but the standard qualitative identification criteria in the method must be met.

Table 18:
Minimum Response Factors

Compound	Minimum Response Factor
Benzaldehyde	0.010
Phenol	0.800
Bis(2-chloroethyl) ether	0.700
2-Chlorophenol	0.800
2-Methylphenol	0.700
2,2-Oxybis-(1-chloropropane)	0.010
Acetophenone	0.010
4-Methylphenol	0.600

Table 18: Minimum Response Factors	
Compound	**Minimum Response Factor**
N-Nitroso-di-n-propylamine	0.500
Hexachloroethane	0.300
Nitrobenzene	0.200
Isophorone	0.400
2-Nitrophenol	0.100
2,4-Dimethylphenol	0.200
Bis(2-chloroethoxy)methane	0.300
2,4-Dichlorophenol	0.200
Naphthalene	0.700
4-Chloroaniline	0.010
Hexachlorobutadiene	0.010
Caprolactam	0.010
4-chloro-3-methylphenol	0.200
2-Methylnaphthalene	0.400
Hexachlorocyclopentadiene	0.050
2,4,6-Trichlorophenol	0.200
2,4,5-Trichlorophenol	0.200
1,1'-Biphenyl	0.010
2-Chloronaphthalene	0.800
2-Nitroaniline	0.010
Dimethyl phthalene	0.010
2,6-Dinitrotoluene	0.200
Acenaphthylene	0.900
3-Nitroaniline	0.010
Acenaphthene	0.900
2,4-Dinitrophenol	0.010
4-Nitrophenol	0.010
Dibenzofuran	0.800
2,4-Dinitrotoluene	0.200
Diethyl phthalate	0.010
1,2,4,5-Tetrachlorobenzene	0.010
4-chlorophenyl-phenyl ether	0.400
Fluorene	0.900
4-Nitroanailine	0.010
4,6-Dinitro-2-methylphenol	0.010
4-Bromophenyl-phenyl ether	0.100
N-Nitrosodiphenylamine	0.010
Hexachlorobenzene	0.100
Atrazine	0.010
Pentachlorophenol	0.050
Phenanthrene	0.700
Anthracene	0.700
Carbazole	0.010
Di-n-butyl phthalene	0.010

Table 18: Minimum Response Factors	
Compound	**Minimum Response Factor**
Fluoranthene	0.600
Pyrene	0.600
Butyl benzyl phthalate	0.010
3,3'-Dichlorobenzidine	0.010
Benzo(a)anthracene	0.800
Chrysene	0.700
Bis-(2-ethylhexyl)phthalate	0.010
Di-n-octyl phthalate	0.010
Benzo(b)fluoranthene	0.700
Benzo(k)fluoranthene	0.700
Benzo(a)pyrene	0.700
Indeno(1,2,3-cd)pyrene	0.500
Dibenz(a,h)anthracene	0.400
Benzo(g,h,i)perylene	0.500
2,3,4,6-Tetrachlorophenol	0.010
Pentachloronitrobenzene	0.050

9.2.4.2.2. Calculate the Standard Deviation (SD) and Percent Relative Standard Deviation (% RSD) of the response factors for each compound:

$$\% \text{ RSD} = \frac{\text{Standard Deviation of RRFs}}{\text{Mean RRF}}$$

9.2.4.2.3. The % RSD of the RRF's must be ≤20% for each target analyte listed in Table 18. The % RSD of each target analytes must be ≤20% in order for the calibration range to be acceptable. Additionally for 8270E, the calculated concentration or amount of each analyte of interest in the CCV standard should fall within ±20%. If more than 10% of the compounds included with the initial calibration exceed the 20% RSD limit or do not meet the minimum correlation coefficient (0.99) or Relative Standard Error (≤20%) for alternate curve fits (see below) then appropriate corrective maintenance action must be performed. If more than 10% of the compounds included with the initial calibration exceed the 20% RSD limit **AND** do not meet the minimum correlation coefficient (0.99)) or Relative Standard Error (≤20%) then recalibration is necessary.

9.2.4.2.4. If the above listed criteria is met, the system can be assumed to be linear and sample analysis may begin and the average RF from the initial calibration range is used to quantitate all samples.

9.2.4.2.4.1 Certain state regulatory programs have defined calibration acceptance limits which, where applicable, are used for calibration evaluations. The TALS ICAL Limit Groups detail these regulatory program criteria.

9.2.4.2.5. An alternative calibration technique may be employed for those any compounds exceeding the 20% RSD criteria:

9.2.4.2.5.4 Calculate the first order linear regression for any compound which did not meet the 20% criteria. First order linear regression calibration may be employed if alternative average response calibration procedures were not applicable. The r value (Correlation Coefficient) of the equation must be ≥0.99 or the Relative Standard Error (RSE) (≤20%) for the calibration to be employed.

9.2.4.2.5.2 Second order regression calibration can be used for any compound that has an established history as a non-linear performer.

9.2.4.2.5.3 If second order regression calibration is used a minimum of six (6) calibration levels must be analyzed.

9.2.4.2.5.4 If second order regression calibration is used, the r^2 (Correlation Coefficient) value must be ≥ 0.99 or the Relative Standard Error (RSE) (≤20%) for the calibration to be employed.

9.2.4.2.5.5 Any compound that fails to meet the 20% RSD or 0.99 correlation coefficient or RSE (≤20%) criteria must be flagged as estimated for detects (or must be noted in the narrative). If there are non-detects the compounds may be reported if there is adequate sensitivity to detect at the quantitation limit. To demonstrate adequate sensitivity analyze the low level point of the initial calibration in each analytical batch (LLCCV) The criteria for demonstrating adequate sensitivity is detection in the LLCCV using the standard qualitative identification criteria.

9.2.4.2.5.6. When calculating the calibration curve using the linear calibration model a minimum quantitation check on the viability of the lowest calibration point should be performed by re-fitting the response from the low concentration back into the curve. The recalculated concentration of the low calibration point should be within ±50% of the

standard's concentration. This evaluation can be checked using the Initial Calibration %Drift Report in Chrom. Any detects for analytes calibrated using the linear model and failing this readback criterion must be flagged as estimated or detailed in the narrative.

9.2.4.3. **Calibration Point Read-back Criteria**: Whichever calibration model above is selected, it should be subjected to an additional check to establish the representativeness of the data that were used to produce it. This check is the refitting of each calibration point response back to the calibration model, or the comparison of the calculated amount of the standard against the expected amount.

- CHROM software provides an Initial Calibration %Drift report which shows the % Error for each calibration point. This report must be reviewed in addition to the %RSD / Linear Response Factor.

- The absolute value of the % Error for each calibration point should be < 30%. For the lowest calibration point, the % Error may be <50%.

- See Section 11.8 for the Calculation of Percent (%) Error.

9.2.4.4. **Initial Calibration Verification (ICV):**.Once the initial calibration has been analyzed and has met the above criteria, a second source Initial Calibration Verification (ICV) (as prepared in Section 7.2) must be analyzed and evaluated. The ICV must meet the criteria of 70-130% recovery for all compounds with the exception of the poor performing compounds listed in Attachment 1 which are allowed to be within 50-150% : An NCM must be initiated to denote any ICV non-conformances.

9.2.4.5. The ICV must meet the criteria of 70-130% recovery for all compounds however up to 10% of the compounds are allowed to exceed these criteria as long as their recoveries are within 65-135%. For the poor performers (see Attachment 1) the range is 50-150%. If the criterion is not met, a second ICV may be analyzed after corrective measures are taken. If a second ICV analysis fails to meet criteria proceed with corrective action and the analysis of a new initial calibration range. Flagging: If the ICV limits are outside of criteria (high) for an analyte and that analyte is undetected in the sample, no flagging or narration is required. If the ICV limits are outside of criteria (low) for an analyte and that analyte is undetected in a sample, narrate the non-conformance in an NCM. When that out of spec analyte is detected in a sample, describe the issue in the narrative, or flag as estimated.

9.2.4.6. Continuing Calibration Verification (CCV): A CCV consisting of a standard at or near the midpoint of the Initial Calibration Range is analyzed every 12 hours of instrument operation or at the beginning of an analytical sequence to verify the initial calibration. The calibration verification consists of a DFTPP instrument performance check (not required for 8270E), and analysis of a calibration verification standard. **Note:** Certain state regulatory programs have defined calibration acceptance limits which, where applicable, are used for calibration evaluations. The TALS ICAL Limit Groups detail these regulatory program criteria.

9.2.4.5.1 Tune Verification: Follow the procedure for verifying the instrument tune described in section 9.2.1 using a 50 ng injection of DFTPP. If the tune cannot be verified, analysis must be stopped, corrective action taken and a return to "control" demonstrated before continuing with the calibration verification process. For 8270E analysis only, tune verification is required just prior to ICAL.

9.2.4.5.2 Calibration Verification: Analyze the calibration verification standard immediately after a DFTPP that meets criteria. Daily analysis of the DFTPP is not required as part of the CCV for 8270E analysis. .When samples are analyzed after an ICAL the last ICAL standard may be used as the starting time reference for evaluation. Use the mid point calibration standard (approximately 50ug/l). NOTE: The calibration standard contains internal standards; Dichlorobenzene d_4, Naphthalene d_8, Acenaphthene d_{10}, Phenanthrene d_{10}, Chrysene d_{12}, and Perylene d_{12} at 40ug/l (0.1ug/L for SIM). The calibration check standard must also include all the target analytes from the original calibration.

9.2.4.5.3 The RFs must meet the criteria for the compounds in Table 18. Any compound that fails the minimum response factor must be reported as estimated for detects and must have a demonstration of sensitivity to report non-detects. To demonstrate adequate sensitivity for out of criterion compounds analyze the low level point of the calibration (LLCCV) in the analytical sequence. The criterion for the LLCCV is detection only but the standard qualitative identification criteria in the method must be met

9.2.4.5.4 The percent difference (when using average response factor) or percent drift (when using linear regression) of the compounds in Table 18 must be ≤20% for at least 80% of the total analyte list. If more than 20% of the compound list fail to 20% difference or drift criterion then appropriate corrective action must be taken prior to the analysis of the samples. Any individual compound that fails must be reported as estimated for detects and must have a demonstration of sensitivity to report non-detects. To demonstrate adequate sensitivity for out of criterion compounds analyze the low level point of the calibration (LLCCV) in the analytical sequence. The criterion for the LLCCV is detection only (%D criteria are not applied) but the standard qualitative identification criteria in the method must be met.

9.2.4.5.5 **CCV Poor Performers**: Refer to Attachment 1 for the identification of poor and/or erratic performing analytes. These analytes are allowed a %D >20% but must be <50 %D to be acceptable. If there are poor performers that exceed 50%D, the data may be reported provided results are noted as estimated. An NCM must be initiated to denote this situation.

9.2.4.5.6 The retention times of the internal standards from the calibration check must be within ±30 seconds of the internal standards from the mid point standard of the original calibration. If the retention time for any internal standard changes by more than 30 seconds from the latest daily (12 hour) calibration standard, the chromatographic system is inspected for malfunctions, and corrections made as required. If corrective action does not result in the retention time criteria being achieved, the system must be re-calibrated using four additional standards.

9.2.4.5.7 The response (area count) of each internal standard in the calibration verification standard must be within 50 - 100% of its corresponding internal standard in the mid-level calibration standard of the active calibration curve. If the EICP area for any internal standard changes by more than a factor of two (-50% +100%), the mass spectrometer system must be inspected for malfunction and corrections made as appropriate. When corrections are made, re-analysis of samples analyzed while the system was malfunctioning is required.

9.2.4.5.8 The relative retention times of each compound in the calibration verification standard must agree within .06 relative retention time units of its value in the initial calibration.

9.2.4.5.9 Use the average response factors from the original five-point calibration for quantitative analysis of target analytes identified in field samples.

9.2.4.5.10 Prepare a calibration summary or list indicating which compounds did not meet the 20% average percent difference criteria. Record this information in that run log.

9.2.4.7. Low Level Continuing Calibration Verification (LLCCV): An LLCCV consisting of the low level standard from the initial calibration range is analyzed every 12 hours of instrument operation after the CCV. The purpose and evaluation of the LLCCV is described in Section 9.2.4.4.4.

10.0 Procedure

10.1. Gas Chromatograph/Mass Spectrometer Operation

10.1.1. The sequence of events for GC/MS analysis involves many steps. First the injection system and column performance and calibration must be verified. Maintenance operations are performed as needed.

10.1.2. Preparation of the Injection Port Liner and Installation Procedure: Prior to the start of initial calibration and each daily analysis of sample extracts, a new liner for the injection port must be prepared. Once a liner has been used it is no longer inert and will cause serious chromatography problems with phenols and other compounds. When preparing the liner, proper laboratory protection must be worn and the liner must be prepared in a well-ventilated hood. When the procedure is completed all traces of toluene, Sylon-Ct and methanol will be removed immediately so that extraction solvents and preparation of sample extracts will not come into contact with these solvents and become contaminated.

10.1.2.1 Remove one liner from a 40ml VOA bottle containing other liners immersed in Sylon-Ct solution. Rinse off the liner with Toluene and wipe dry. Insert 1cm of pre-silanized glass wool partially into one end of the liner and trim neatly. Push the glass wool into the center of the liner so that it is 1 1/4" from the bottom. Do not use glass wool or solvents that are dirty (i.e. suspended particles) or use liners which are chipped on the ends, deformed or fractured. Inspect the glass wool for cleanliness after it has been inserted.

10.1.2.2 Using a Pasteur pipette flush out the interior of the liner containing the glass wool with Sylon-Ct. Rest the liner horizontally on a small beaker and allow the Sylon-Ct to re-deactivate the interior surfaces and the glass wool. There should be no air bubbles caught in the glass wool. After several

minutes flush out the Sylon-Ct with toluene and finally with methanol. Dry the outer surface of the liner and rest it on the injection port housing until the remaining methanol is boiled off

10.1.2.3 Insert the liner with the newly silanized glass wool plug into the injection port. Verify that the column extends up into the injection port and is perpendicular. Inspect the graphite seal and replace it if the edges are knife-shaped.

10.1.2.4 The septum is always replaced daily. Bake out the column at 300^{O}C for 15 minutes after the vacuum in the analyzer has returned to normal.

10.1.2.5 Performance may enhanced by clipping a small portion of the column at the injection port end. Document this activity in the maintenance record.

10.1.3. Prior to calibration or sample analysis always verify that the analyzer is under sufficient vacuum and that the column has proper carrier gas flow.

10.1.4. Establish the following GC/MS operating conditions:

10.1.4.1 Full Scan Operating Mode

Full Scan Mode – Standard Injection Volume
Mass Range: 35 to 500amu
Scan Time: 1 sec/scan
Transfer Line Temperature: 300^{O}C
Source Temperature: Preset by H.P. at 280^{O}C
Scan start time: 1.0 minutes
Initial Column Temperature and Hold Time: 45^{O}C for 0.5 minutes
Column Temperature Program: 20°C /min to 100°C 25°C/min to 270°C 10° C/min to 310°C
Final Column Temperature Hold: 310^{O}C for 5 minutes
Carrier Gas: Ultra High Purity Grade Helium at 1.3ml/min
Injector Temperature: 275^{O}C
Injector: Grob-type, pulse, splitless
Injection Volume: 1ul
Splitless Valve Time: 0.3 minutes

Full Scan Mode – Large Volume Injection (LVI)
Mass Range: 35 to 500amu
Scan Time: 1 sec/scan
Transfer Line Temperature: 300^{O}C
Source Temperature: Preset by H.P. at 280^{O}C
Scan start time: 1.0 minutes
Initial Column Temperature and Hold Time: 45^{O}C for 0.5 minutes
Column Temperature Program: 20°C /min to 100°C 25°C/min to 270°C 10° C/min to 310°C
Final Column Temperature Hold: 310^{O}C for 5 minutes
Carrier Gas: Ultra High Purity Grade Helium at 1.3ml/min
Injector Temperature: 275^{O}C
Injector: Grob-type, pulse, splitless
Injection Volume: 5ul
Splitless Valve Time: 0.3 minutes

10.1.4.2 SIM Operating Mode

SIM Mode
Mass Range: 35 to 500amu
Scan Time: 1 sec/scan
Transfer Line Temperature: 300^{O}C
Source Temperature: Preset by H.P. at 280^{O}C
Scan start time: 1.5 minutes
Initial Column Temperature and Hold Time: 40^{O}C for 0.5 minutes
Column Temperature Program: 20°C /min to 100°C 25°C/min to 270°C 10° C/min to 310°C
Final Column Temperature Hold: 310^{O}C for 3 minutes
Carrier Gas: Ultra High Purity Grade Helium at 1.3ml/min
Injector Temperature: 275^{O}C
Injector: Grob-type, pulse splitless
Injection Volume: 1ul
Splitless Valve Time: 0.3 minutes

10.1.4.3 Isotope Dilution Selected Ion Monitoring Mode :

SIM Parameters

Group 1
Plot 1 Ion: 74.0

Ions/Dwell in Group	(Mass Dwell)	(Mass Dwell)	(Mass Dwell)
	42.0 50	43. 0 50	68.0 50
	74.0 50	128.0 50	129.0 50
	136.0 50	150.0 50	152.0 50
	93.0 50	66.0 50	
	58.0 50		
	88.0 50		

Group 2
Group Start Time: 6.00
Plot 1 Ion: 152.0

Ions/Dwell in Group	(Mass Dwell)	(Mass Dwell)	(Mass Dwell)
	151.0 50	152.0 50	153.0 50
	154.0 50	162.0 50	164.0 50
	165.0 50	166.0 50	

Group 3
Group Start Time: 7.80
Plot 1 Ion: 188.0

Ions/Dwell in Group	(Mass Dwell)	(Mass Dwell)	(Mass Dwell)
	94.0 50	101.0 50	142.0 50
	178.0 50	179.0 50	188.0 50
	202.0 50	264.0 50	266.0 50
	284.0 50		

Group 4
Group Start Time: 10.50
Plot 1 Ion: 228

Ions/Dwell in Group	(Mass Dwell)	(Mass Dwell)	(Mass Dwell)
	120.0 50	228.0 50	229.0 50
	240.0 50		

Group 5
Group Start Time: 12.00
Plot 1 Ion: 252.0

Ions/Dwell in Group	(Mass Dwell)	(Mass Dwell)	(Mass Dwell)
	138.0 50	139.0 50	252.0 50
	253.0 50	260.0 50	264.0 50
	267.0 50	276.0 50	278.0 50

Table 19: Target Compound - Primary and Monitoring Ions

Compound	1	2	3

Compound	1	2	3
1,4-Dioxane-d8	96	64	62
1,4-Dioxane	88	58	57
1,4-Dichlorobenzene-d4	152	150	

10.1.5. The above listed instrument conditions are used for all analytical standards for calibration and for all sample extracts analyzed by this method.

10.1.5.1 The column conditions, scan start time, and splitless valve time for analysis of DFTPP only are as follows are as follows:

Initial Column Temperature and Hold Time: 140^{o}C for 0.5 minutes
Column Temperature Program: 140^{o} to 320^{o}C at 22^{o}C/minute
Final Column Temperature Hold: 320C for 0.5 minutes
Scan Start Time: approx. 5 minutes
Splitless Valve Time: 0.3 minutes
Injection Volume: 2 ul

10.2. Analytical Sequence

10.2.1. Dilutions are made based on initial GC/MS analysis. Dilutions are made in 1-ml vials using microsyringes. Calculate the dilution factor using the equation below:

DF= Ph / 5 x Is
Where:
DF = Dilution Factor
Ph = Sample Peak Height
Is = Internal Standard Peak Height

When DF >1 but <2, combine 500ul of sample extract with 500ul methylene chloride in a 1 ml amber vial, add20 ul internal standard and crimp seal

Use **Table 20** to determine dilution and internal standard amount.

Table 20 Dilution Factor Calculations			
DF Value	**Volume of Sample (ul)**	**Volume of Methylene Chloride (ul)**	**Volume of ISTD (ul)**
<1	1,000	None	None
>1, <2	500	500	10
>4, <5	200	800	16
>10, <20	100	900	36
>20	500*	500	10
*Prepare this dilution by serially diluting the >10, <20 dilution			

10.2.2. Instrument Performance and Calibration Sequence

10.2.2.1. Once the GC/MS instrument has been setup and maintained as detailed in Section 10.1, the first operations to be performed are the performance checks and calibration standards.

10.2.2.2. Analyze the Instrument Performance Check Standard (DFTPP) as discussed in Section 9.2.1.

10.2.2.3. Initially and as required, analyze the Initial Calibration Range (minimum 5 points, six points for second order regression) as detailed in Sections 7.2.1 and 9.2.4.2. Evaluate the acceptability of the Initial Calibration Range as detailed in Section 9.2.4.2.

10.2.2.4. Immediately after the Initial Calibration Range only, analyze the Initial Calibration Verification (ICV) as detailed in Sections 7.2. and 9.2.4.3. Evaluate the acceptability of the ICV as detailed in Section 9.2.4.3.

10.2.2.5. Every 12 hours, reanalyze and evaluate the Instrument Performance Check Standard (DFTPP), not required for 8270E followed by the Continuing Calibration Verification (CCV) and Low Level Continuing Calibration Verification (LLCCV) as detailed in Section 9.2.3, 9.2.4.4 and 9.2.4.5. Evaluate the acceptability of the CCV and LLCCV as detailed in Section 9.2.4.4

10.2.2.6. Client samples and QC samples are analyzed (as detailed in Section 10.2.3) after acceptable Instrument Performance and Calibration Checks and until the 12 hour clock expires. Repeat the sequence as required. The automation of GC/MS runs is accomplished via the “SEQUENCE” macro of the ChemStation.

10.2.3. Sample Analysis Sequence

10.2.3.1. Sample extracts are normally prepared on the same day as analysis. The GC/MS operator will prepare the extracts that will be run on his or her instrument. Volume adjustments to the extracts will be made at the discretion of the supervisor.

10.2.3.2. Prior to the start of sample analysis the GC/MS operator will generate a sequence program containing the list of the sample extracts to be analyzed, the position on the autosampler tray, and the proper acquisition and tune methods that are to be used. This sequence program contains all the necessary information on the samples to be analyzed and how the GC/MS system is to analyze them. The sample extracts are loaded onto the autosampler (ALS) tray. Their position is verified by checking them against the ALS number on the sequence. This batch analysis will be performed automatically over the 12-hour period.

10.2.3.3. The analytical run log is printed as a record of samples analyzed. The analyst will annotate the run log with any required information regarding anomalies or unusual events. The run log must be signed by the analyst and a reviewed and signed by a trained peer or manager

10.3. Data Processing

10.3.1. Prior to processing any standards or samples, target compound lists and sublists must be assembled. Chrom's auto-processing system queries TALS (LIMS) for each sample's processing parameters (including target compounds lists) and downloads the required processing methods from LIMS to analyze data. These lists are required for processing of all data files including calibration files. The data includes compound names, retention time data, quantitation ions, qualitative identification ions, and the assigned internal standard for qualitative and quantitative identification.

10.3.2. Key data is manually entered the first time a compound list is used for data processing. Processing data using a compound list automatically generates response factor data and updates retention information.

10.3.3. The characteristic ions for target compounds, surrogate compounds, and internal standards which can be determined using SW 8270E are listed in Table 21.

10.4. Interpretation and Qualitative Identification: Qualitative identification of target compounds is based on retention time and mass spectral comparison with characteristic ions in the target compound list. The reference mass spectrum is taken from a standard of the target compound analyzed by this method. The

characteristic ions are the three ions of greatest relative intensity or any ions over 30% relative intensity if less than three such ions occur in the reference spectrum. Compounds are identified as present when the following criteria are met:

10.4.1 Target Analytes: Qualitative identification of target compounds is based on retention time and mass spectral comparison with characteristic ions in the target compound list. The reference mass spectrum is taken from a standard of the target compound analyzed by this method. The characteristic ions are the three ions of greatest relative intensity or any ions over 30% relative intensity if less than three such ions occur in the reference spectrum. Compounds are identified as present when the following criteria are met:

10.4.1.1. Once the GC/MS instrument has been setup and maintained as detailed in Section 10.1, the first operations to be performed are the performance checks and calibration standards.

10.4.1.2. The intensities of the characteristic ions of a compound maximize in the same scan or within one scan of each other.

10.4.1.3. The relative retention time (RRT) of the sample component is within ± 0.06 RRT units of the RRT of the standard component.

10.4.1.4. The most abundant ion in the standard target spectrum that equals 100% MUST also be present in the sample target spectrum.

10.4.1.5. All other ions that are greater than 10% in the standard target spectra should also be present in the sample.

10.4.1.6. The relative intensities of the characteristic ions agree within 30% of the relative intensities of these ions in the reference spectrum. (Example: For an ion with an abundance of 50% in the reference spectrum, the corresponding abundance in a sample spectrum can range between 20% and 80%).

10.4.1.7. If the compound does not meet all of the criteria listed above, but is deemed a match in the technical judgment of the mass spectral interpretation specialist, the compound will be positively identified and reported with documentation of the identification noted in the raw data record.

10.4.2 Non-Target Analytes: Upon client request a library search to identify non-target Tentatively Identified Compounds (TIC) is performed. The NIST/EPA/NIH mass spectral library is used to identify non-target compounds (not including internal standard and surrogate compounds) of greatest apparent concentration by a forward search of the library. The following guidelines are used by the analyst when making TIC identifications:

10.4.2.1. Relative intensities of major ions in the reference spectrum (ions greater than 10% of the most abundant ion) should be present in the sample spectrum.

10.4.2.2. The relative intensities of the major ions should agree within ±20%. (Example: For an ion with an abundance of 50% in the standard spectrum, the corresponding sample ion abundance must be between 30 and 70%).

10.4.2.3. Molecular ions present in the reference spectrum should be present in the sample spectrum.

10.4.2.4. Ions present in the sample spectrum but not in the reference spectrum should be reviewed for possible background contamination or presence of co-eluting compounds.

10.4.2.5. Ions present in the reference spectrum but not in the sample spectrum should be reviewed for possible subtraction from the sample spectrum because of background contamination or co-eluting peaks. Data system library reduction programs can sometimes create these discrepancies.

10.4.2.6. If, in the technical judgement of the mass spectral interpretation specialist, no tentative identification can be made, the compound will be reported as 'Unknown'. If the compound can be further classified the analyst may do so (i.e, 'Unknown hydrocarbon', 'Unknown acid' , etc.).

10.5. Data Reporting

10.5.1. Final Report. The Chom data system automatically produces a data report consisting of hardcopy reports corresponding to specific data reporting requirements, which is uploaded to the TALS LIMS System for the report production group.

10.5.1.1. Total Ion Chromatogram. Full length chromatogram depicting the full length of the GC/MS acquisition.

10.5.1.2. Spectra of all detected target compounds. A page for each detected target compound spectra with a standard reference spectrum for comparison.

10.5.1.3. The calculations of the concentrations of each target compound in the sample, reported in units of ppb, ug/kg or ug/l.

10.5.1.4. Data summaries for each method blank indicating which samples were extracted with the indicated blank.

10.5.1.5. A copy of the initial calibration range together with the calibration verification report, and tune report.

10.5.1.6. Quality Control (QC) data report for each batch including surrogate recoveries, internal standard area summaries, LCS, MS/MSD and RPD summaries.

10.6. The low-level calibration standard establishes the reporting limit. All reported data must be at a concentration at or above the low concentration standard. Any quantitative values below the report limit must be qualified as estimated.

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11.0. Calculations/Data Reduction

11.1. Target Compounds: are quantitated using the internal standard method (see the formula in Section 11.3).

11.1.1. Identified target compounds are quantitated using the integrated abundance from the EICP of the primary characteristic ion. The internal standard used shall be the one nearest the retention time of the analyte).

11.1.2. The average response factor (RRF) from the initial calibration is used to calculate the target analyte concentration in client samples using the formula found in Section 11.3. See Section 9.2.4 for discussion of RRF.

11.1.3. Secondary ion quantitation is utilized only when there are sample interferences preventing use of the primary characteristic ion. If secondary ion quantitation is used an average relative response factor (RRF) must be calculated using that secondary ion.

11.2. Non-Target Compounds (Tentatively Identified Compounds): An estimated concentration for non-target (tentatively identified compounds) is calculated using the internal standard method (see formula in Section 11.3). For quantiation, the nearest eluting internal standard free of interferences is used. The procedure used for calculating the concentration of non-target compounds is the same as that used for target compounds (see Section 11.1) with the following revisions:

11.2.1. The total area count of the non-target compound is used for As (instead of the area of a characteristic ion).

11.2.2. The total area count of the chosen internal standard is used as Ais (instead of the area of a characteristic ion).

11.2.3. A RF on 1.0 is assumed.

11.2.4. The resulting concentration is qualified as estimated ('J') indicating the quantitative uncertainties of the reported concentration.

11.3. Internal Standard Calculation:

11.3.1. Aqueous Samples

$$\text{Concentration } (\mu g/L) = \frac{(As)(Cis)(D)}{(Ais)(RF)(Vs)\ (Vi)\ (1000)}$$

Where:

As	=	Area of the characteristic ion for the target analyte in the sample
Cis	=	Concentration of the internal standard (ug/L)
D	=	Dilution factor, if the sample or extract was diluted prior to analysis. If no dilution is performed, D = 1.
Vi	=	Volume of the extract injected (ul)
Ais	=	Area of the characteristic for the associated internal standard
RF	=	Average response factor from the initial calibration.
Vs	=	Volume of sample extracted (ml)

The 1000 in the denominator represents the number of ul in 1 ml.

11.3.2. Solid Samples

$$\text{Concentration } (\mu g/KG) = \frac{(As)(Cis)(D)(Vt)}{(Ais)(RF)(Ws)\ (Vi)\ (1000)}$$

Where:

As	=	Area of the characteristic ion for the target analyte in the sample
Cis	=	Concentration of the internal standard (ug/L)
D	=	Dilution factor, if the sample or extract was diluted prior to analysis. If no dilution is performed, D = 1.
Vi	=	Volume of the extract injected (ul)
Ais	=	Area of the characteristic for the associated internal standard
RF	=	Average response factor from the initial calibration.
Vt	=	Volume of concentrated extract (ul)
Ws	=	Weight of sample (g)

The 1000 in the denominator represents the number of ul in 1 ml.

11.4. Relative Response Factors

$$RRF = \frac{A_x}{A_{is}} \times \frac{C_{is}}{C_x}$$

Where:

A_x = Area characteristic ion for the compound (see Table 21)
Ais = Area characteristic ion of associated internal std (See Table 21)
Cis = Concentration of internal standard
Cx = Concentration of compound in standard

11.5. Percent Relative Standard Deviation (% RSD) : as discussed in Section 9.2.4.4 (Initial calibration):

$$\% \text{ RSD} = \frac{\text{Standard Deviation of RRFs}}{\text{Mean RRF}}$$

11.6. Percent Difference (% D):as discussed in Section 9.2.4.4 (Continuing calibration):

$$\% D = \frac{RRF_c - \overline{RRF_i}}{\overline{RRF_i}} \times 100$$

Where: RRFc = RRF from continuing calibration

$\overline{RRF_i}$ = Mean RRF from current initial calibration

11.7. Percent Recovery (% R): Surrogates and Spikes

$$\text{Recovery (\%)} = \frac{\text{Concentration (or amount) found}}{\text{Concentration (or amount) added}} \times 100$$

11.8. Calculation of Relative Standard Error (RSE)

$$\%\,RSE = 100\% \times \sqrt{\frac{\sum_{i=1}^{N}\left[\frac{C_i - PC_i}{C_i}\right]^2}{(N-P)}}$$

Where:

N = Number of points in the curve
P = Number of parameters in the curve (= 1 for average response factor, 2 for linear, 3 for quadratic)
CI = True concentration for level i
PCi = Predicted concentration for level

11.9. Calculation of Percent (%) Error

$$\%Error = \frac{x_i - x_i'}{x_i} x\,100$$

Where:

x_i ' = Measured amount of analyte at calibration level *i*, in mass or concentration units

x_i = True amount

11.10. Dry Weight Correction: All solid samples must be corrected for dry weight using the following formula for dry weight determination.

$$DW = \frac{Gd}{Gw} \times 100$$

Where:

DW	=	Percent % Dry Weight
Gd	=	Dry weight of selected sample aliquot
Gw	=	Wet weight of selected sample aliquot

Multiply the DW value times the wet weight of the sample extracted. NOTE: This calculation can also be performed automatically by the target system provided the DW value is available and entered into the system.

12.0. Method Performance

12.1. Method Detection Limit Study (MDL)

A Method Detection Limit (MDL) study, as described in the Eurofins corporate Detection and Quantitation Limits SOP, CA-Q-S-006, must be performed initially and whenever a significant change affecting sensitivity is made to the analytical system. The MDL must be re-evaluated from quarterly MDL points at least every 12 months.

12.2. Demonstration of Capabilities

For DOC procedure refer to Section 19 in the most current revision of Eurofins Edison's Quality Assurance Manual (ED-QA-LQM).

12.3. Lower Limit of Quantitation Verification

The lowest calibration standard analyzed establishes the LLOQ or Reporting Limit. The capability to reliably detect this concentration through the preparation, clean-

up and analytical procedure is verified through the annual analysis of a standard at the LLOQ/RL. The LLOQ verification shall also be performed whenever significant changes are made to the preparation and/or analytical procedure.

12.3.1 The LLOQ verification standard shall be prepared at a concentration 0.5-2 times the LLOQ/RL, and be taken through all of the same preparation and clean-up methods as client samples.

12.3.2 The LLOQ verification standard for aqueous matrix shall be prepared using laboratory deionized water and for the solid matrix using clean Ottawa sand. Other clean matrices may be used in addition, for project specific requirements.

12.3.3 The annual LLOQ verification is completed and documented with the required annual MDL evaluation.

12.3.4 Recovery of each analyte must meet the laboratory established LCS recovery limits + 20%. (For example, if the LCS recovery limits are 70-130%, the LLOQ verification must meet recovery limits of 50-150%.) Once sufficient points have been generated, LLOQ based statistical limits may be used in place of limits based on LCS recovery.

NOTE: The lower recovery limit for the LLOQ can be no lower than 10%.

12.4. Training Requirements

Refer to Eurofins SOP No. ED-GEN-022, (*Training*), for the laboratory's training program.

13.0. Pollution Control

13.1 It is Eurofins's policy to evaluate each method and look for opportunities to minimize waste generated (i.e., examine recycling options, ordering chemicals based on quantity needed, preparation of reagents based on anticipated usage and reagent stability). Employees must abide by the policies in Section 13 of the Corporate Environmental Health and Safety Manual (CW-E-M-001) for "Waste Management and Pollution Prevention."

14.0. Waste Management

14.1 Waste management practices are conducted consistent with all applicable rules and regulations. Excess reagents, samples and method process wastes are disposed of in an accepted manner. Waste description rules and land disposal restrictions are followed. Waste disposal procedures are incorporated by reference to Eurofins Edison SOPs Nos. ED-SPM-007 (*Disposal of Samples and Associated Laboratory Waste, current revision)* and ED-SPM-008 (*Laboratory Waste Disposal Procedures, current revision).* The following waste streams are produced when this method is carried out:

- Auto sampler vials and expired standards: These vials are collected in satellite accumulation within the instrument laboratory. The vials are then placed into a 55 steel open top drum in the waste room. When the drums are full, the drum will be collected by the waste vendor for disposal. This waste is treated for incineration.

  Teris Profile Number: 50016652
  Onyx Profile WIP Number: 282493

- Mixed Solvent Waste: Mixed solvent waste is collected in a small beaker inside the bench top hood. This waste is then transferred into the satellite accumulation container in the Organic Prep. Lab. on a daily basis. This material is transferred into 5 gallon solvent cans as satellite accumulation. These cans are emptied every 24 hours into a steel drum in the waste room. This drum is kept in the walk in hood until it is full. The full drum is then removed from the hood and placed on secondary containment in the waste room.

  Teris Profile Number: 50016624
  Onyx Profile WIP Number: 545240

14.1. Pollution Prevention

14.2.1. Pollution prevention encompasses any technique that reduces or eliminates the quantity or toxicity of waste at the point of generation. Numerous opportunities for pollution prevention exist in laboratory operation. The USEPA has established a prevention hierarchy of environmental management techniques that places pollution prevention as the management option of first choice. Whenever feasible, laboratory personnel should use pollution prevention techniques to address their waste generation. When wastes cannot be feasibly reduced at the source, the agency recommends recycling as the next best option.

14.2.2. The quantity of chemical purchased should be based on expected usage during its shelf life and disposal cost of unused material. Actual reagent preparation volumes should reflect anticipated usage and reagent stability.

15.0. References / Cross-References

15.1. United States Environmental Protection Agency, “Method SW8270D, Semivolatile Organic Compounds by Gas Chromatography/Mass Spectrometry”, Test Methods for Evaluating Solid Wastes, SW846 Third Edition, Laboratory Manual, Physical/Chemical Methods, Revision 5, July 2014..

15.2. United States Environmental Protection Agency, “Method SW8270E, Semivolatile Organic Compounds by Gas Chromatography/Mass Spectrometry”, Test Methods for Evaluating Solid Wastes, SW846 Update IV, Laboratory Manual, Physical/Chemical Methods, Revision 6, June 2018.

15.3. United States Environmental Protection Agency, "Method SW8000D: Determinative Chromatographic Separations", Test Methods for Evaluating Solid Wastes, SW846, Laboratory Manual, Physical/Chemical Methods, Update V, Revision 4, October 2012..

15.4. Eurofins Edison Document No. ED-QA-LQM, *Laboratory Quality Manual*, current revision.

15.5. Eurofins Edison SOP No. ED-ORP-002, *SW846 Method 3510C-Extraction of Semi-Volatile Organic Compounds in Water by Separatory Funnel,* current revision.

15.6. Eurofins Edison SOP No. ED-ORP-043, *SW846 Method 3580A - Waste Dilution Prep for Analysis of BNAs by SW846 Method 8270,* current revision.

15.7. Eurofins Edison SOP No. ED-ORP-044, *Procedure for the Microwave Extraction of Solids, SW3546, current revision.*

15.8. Eurofins Document No. CW-E-M-001, Corporate Environmental Health and *Safety Manual,* current revision.

15.9. Eurofins Corporate Quality SOP No. CA-Q-S-001, *Solvent & Acid Lot Testing & Approval*, current revision.

15.10. Eurofins Edison SOP No. ED-GEN-023 (*Bulk Solvent Testing and Approval*), current revision.

15.11. Eurofins Edison SOP No. ED-GCS-001, *Preparation and Screening of Semivolatile Organic Extracts for GC/MS Analysis*, current revision.

15.12. Eurofins Edison Work Instruction Document No. EDS-WI-012, *Client Complaint/Corrective Action Form,* current revision.

15.13. Eurofins Edison SOP No. ED-GEN-003, *Standard Operating Procedure for Control of Non-Conformances and Corrective Action,* current revision.

15.14. Eurofins Edison SOP No. ED-ORP-001, *Extraction of Semivolatile Organic Compounds in Water, EPA Method 625.1,* current revision.

15.15. Eurofins Edison SOP No. ED-GEN-022, *Training,* current revision.

15.16. Eurofins Corporate Quality Memorandum, CA-Q-QM-002, *GC/MS Tuning Policy*, current revision.

15.17. Eurofins Corporate Quality SOP No. CA-Q-S-006, *Detection and Quantitation Limits,* current revision.

16.0. Method Modifications:

Method 8270E requires the DFTPP tune standard to be analyzed once prior to an ICAL

and not daily prior to sample analysis. Until such time as 8270D is removed from lab capabilities and in order to satisfy both 8270D and 8270E The laboratory will analyze the DFTPP tune daily, prior to QC and sample analysis. The laboratory will use the tighter criteria from Methods 8270C/8270D for tune evaluation, rather than the criteria suggested in Table 3 of Method 8270E.

17.0. Attachments

Attachment 1 Poor Performing Analytes

18.0. Revision History

- Revision 10, date 10/18/2022
    - Updated to Eurofins branding throughout.
    - Section 8.1: updated to include procedure for sampling chlorinated water sources.
    - Section 9 updated throughout to include ICAL evaluation with Relative Standard Error (RSE).
    - Section 11.8: added formula for calculation of Relative Standard Error (RSE)

- Revision 9, date 03/15/2021
    - Updated as needed to reflect 1,4-dioxane RL of 0.2 ug/l.
    - Updated Tables 11 and 12 to reflect new low ICAL standard concentration of 1,4-dioxane..

- Revision 8, date 06/29/2020
    - Updated to Eurofins branding.
    - Updated throughout to include 8270E requirements.
    - Removed references to SW846 3550B/C prep methods (no longer in use for this method at Edison lab).
    - Update equipment listed in Section 6.0.Updated analytical column in Section 6.1.2.
    - Updated, deleted and renumbered tables as required.
    - Made extensive updates to Standards (sources and preparation) in Section 7.2.
    - Removed all references to Aromatic Amines. Deleted all tables specific to Aromatic Amine analysis. Renumbered remaining tables in document and updated text references.
    - Throughout document clarified tune requirements for 8270E.
    - Following added to Section 9.1.1: For method 8270E the method blank is generally acceptable if target analyte concentrations are less than the one half the reporting limit.
    - Calibration Point Read-back Criteria was added to Section 9.2.4.3. The calculation for percent error was added to Section 11.8.
    - Section 9.2.4.2.3: added following for 8270E: the calculated concentration or amount of each analyte of interest in the CCV standard should fall within ±20%.
    - Section 9.2.4.2.5.6: added ‘The recalculated concentration of the low calibration point should be within ±50% of the standard’s concentration.”

- Section 12.1 revised to reflect the updated MDL procedure.
- Added Section 12.3: annual Lower Limit of Quantitation Verification
- Added Corporate SOP CA-Q-S-006, Detection and Quantitation Limits to references.
- Section 16.0: added a Method Modification regarding tuning check requirements.

- Revision 7, date 06/08/2018

  - Section 2.3: revised to clarify that RVE/LVI is lab standard procedure.
  - Section 9.1.3: removed statement regarding allowance for up to five analytes to recover outside of lab acceptance limits in LCS/LCSD.
  - Section 9.2.4.3: Replace table 'ICV Poor Performers (50-150% Recovery) with expanded list of 'Poor Performing Analytes' in Attachment 1.
  - Added Section 9.2.4.4.5: CCV Poor Performers
  - Corrected number in section 9.2.4.5
  - Added Attachment 1 – Poor Performing Analytes

- Revision 6, date 01/12/2018:

  - Section 7.2.5 included to specify reagent and standard storage conditions.
  - Revised Section 9.1.3 to clarify requirements for specific LCS/LCSD evaluation criteria regarding the # of out of criteria analytes.
  - Revised Section 9.2.4.3 to add 2,4-Dimethylphenol as a poor performing analyte, increased the range for the poor performers to 50-150 and also expanded the guidelines for flagging the ICV outliers.

- Revision 5, dated 09/29/2017:
  - Revised Section 9.1.1 to clarify requirements for surrogate recovery in method blanks.

- Revision 4, dated 08/21/2017:
  - Updated throughout to add a procedure for the analysis of 1,4-dioxane by isotope dilution selected ion monitoring (SIM)
  - Added tables for isotope dilution SIM standards. Renumbered all tables as necessary.
  - Section 7.2.1: added a list of full scan calibration list options.
  - Table 3: Renamed 'Full Scan Stock Standards'.
  - Section 9.2.1: noted that DFTTP applies only to full scan analysis.
  - Section 9.2.3: updated CCV concentrations
  - Added reference to GC/MS Tuning Policy in Section 15.16.

- Revision 3, dated 01/07/2016:
  - Tables 1 and 2: added SIM as option for 1,4-Dioxane.
  - Section 2.3: removed SW3541 (Soxtherm) as option for soils prep (lab has discontinued use of this method). Also removed SW3541 SOP reference from Section 15.0.
  - Tables 19 and 20: added source and prep instructions for 1,4-Dioxane SIM standard. Updated source and prep instructions for 4,6-Dinitro-2-methylphenol.
  - Table 22: added prep instructions for 1,4-Dioxane and 4,6-Dinitro-2-methylphenol

SIM ICV standard.
- Corrected the information in the 'DFTPP Key Ions and Abundance Criteria' table in Section 9.2.1 to match the info found in SW846 8270C.
- Section 10.1.4.2: updated "SIM Parameters" to included ion masses/dwell times for 1,4-Dioxane.

- Revision 2, dated 01/28/2015:
  - Extensively reformatted the SOP. Placed tables that had been in rear of document into the body of the text. Renumbered tables as applicable and fixed text references to tables.
  - Section 1.1, Table 1: Revised table to include all current analytes. Also footnoted those compounds which are currently analyzed by SIM.
  - Section 2.3: added options for extraction of solids by SW846 3456 (Microwave Extraction) and by SW3580A (Waste Dilution) and added SOP references. Deleted reference to SOP ED-ORP-005 (SW3550B – Low Level); Updated Section 15 (References).
  - Section 2.5: added text detailing the RVE/LVI options.
  - Section 2.6: added table which includes all analytes routinely analyzed by SIM.
  - Section 6: updated to include newer GC, MS and autosampler models currently in use.
  - Section 6.1.3: added Zebron ZB column as an option.
  - Section 7.2: extensively revised standards information to reflect switch to Restek standards.
  - Table 3:Added Custom Aromatic Amine Surrogate Standard and revised Table 8 to include initial calibration prep instructions for the Aromatic Amine surrogates.
  - Throughout document: removed references to Target and replaced with Chrom.
  - Section 7.2.1: Added reference to section 10.2.1.2 for LVI.
  - Added Section 7.2.1.3.1 and Table 17A both of which discuss use of Aromatic Amine surrogates.
  - Section 7.2.1.2: Added reference to Tables 9,10 and 11 (ICV Preparation)
  - Section 8.0: Added Sample container and minimum sample size (250 ml) for Reduced volume extraction.
  - Sections 9.1.2, 9.1.3, 9.1.4 and 9.2.4: added statement that certain state regulatory programs have defined recovery limits which, where applicable, are used for spike and calibration evaluations.
  - Section 9.1.2: Deleted sentence "A minimum of 16 spiked analytes are reported to in client reports (the full list is reported at least once during each 2 year period because we employ full spiking list.
  - Section 9.1.4: Added note regarding use of Aromatic Amine Surrogates.
  - Section 9.2.2.2: Added reference to ICV Preparation tables in Section 7.2.
  - Section 9.2.3: added more specific info as to the concentration of the CCVs for all techniques.
  - Section 9.2.4.2.1: Changed to reflect that each analyte should meet minimum RF's, not the average across the calibration. Added LLCCV requirement.
  - Section 10.3.1: added explanation of Chrom's interaction with TALS. Removed references to Target.
  - Section 9.2.4.2.5.5: Added: (or can be noted in the narrative)
  - Section 9.2.4.2.5.6: Revised last sentence to read: "This evaluation can be checked using the Initial Calibration %Drift Report in Chrom."
  - Section 9.2.4.3: Removed 65-135% criteria and added "poor performing" analyte list and associated criteria of 60-140%.

- Section 9.2.4.4.3: Added LLCCV criterion for RFs
- Section 9.2.4.4.4: Added LLCCV criterion for %D
- Section 10.1.4: Updated GC/MS operating conditions for full scan, SIM and DFTPP.
- Section 10.1.4.1: added a table detailing operating conditions for LVI option.
- Table 2: Added 2-ethylaniline, 2,4-dimethylaniline, 3,4-dimethylaniline, 2,3-dimethylaniline, 2,4,5-trimethylaniline and 4-chloro-o-toluidine to Working Standards preparation information.
- Table 25: updated to include all current analytis/surrogates/internal standards and associated ions.
- Throughout document: updated LQM section references as appropriate as some have changed with the latest LQM revision.

- Revision 1, dated 11/07/2011

  - Section 1.1, Table 1: Added Pentachloronitrobenzene and associated CAS# to the analyte list.
  - Section 7.2.1: Added Pentachloronitrobenzene standard information.
  - Table 2: Added Pentachloronitrobenzene to Working Standards preparation information.
  - Table 4: Added Pentachloronitrobenzene and associated minimum RF.
  - Table 8: Added Pentachloronitrobenzene and associated ions.

- Revision 0, dated 02/22/2011: NEW

Table 21
Characteristic Ions Of Semi-Volatile Organic Compounds

Compound	Primary Ion	Secondary Ion(s)
1,1'-Biphenyl	154	153,76
1,2,4,5-Tetrachlorobenzene	216	214, 179
1,2,4-Trichlorobenzene	180	182, 145
1,2-Dichlorobenzene	146	148, 111
1,2-Diphenylhydrazine	77	105, 182
1,3-Dichlorobenzene	146	148, 111
1,3-Dimethylnaphthalene	156	141, 115
1,4-Dichlorobenzene	146	148, 111
1,4-Dichlorobenzene d4 **(ISTD)**	152	150, 115
1,4-Dioxane	88	58, 43
1-Methylnaphthalene	142	141, 115
1-Naphthylamine	143	115, 116
2,2'-oxybis[1-chloropropane]	45	77, 121
2,3,4,6-Tetrachlorophenol	232	131, 230
2,3,7,8-TCDD (screen)	320	322, 324
2,3-Dihydroindene		
2,3-Dimethylaniline	106	129

Table 21 Characteristic Ions Of Semi-Volatile Organic Compounds		
Compound	**Primary Ion**	**Secondary Ion(s)**
2,4,5-Trichlorophenol	196	198, 200
2,4,5-Trimethylaniline	102	55, 56
2,4,6-Tribromophenol **(Surrogate)**	330	132, 141
2,4,6-Trichlorophenol	196	198, 200
2,4-Dichlorophenol	162	164, 98
2,4-Xylidine	121	120, 106
2,4-Dimethylphenol	122	107, 121
2,4-Dinitrophenol	184	63, 154
2,4-Dinitrotoluene	165	63, 89
2,6-Dinitrotoluene	165	63, 89
2-Chloronaphthalene	162	127, 164
2-Chlorophenol	128	64, 130
2-Ethylaniline	106	122,104
2-Fluorobiphenyl **(Surrogate)**	172	171
2-Fluorophenol **(Surrogate)**	112	64
2-Methylnaphthalene	142	141
2-Methylphenol	108	107
2-Naphthylamine	143	115, 116
2-Nitroaniline	65	108, 138
2-Nitrophenol	139	109, 65
2-tert-butyl-4-Methylphenol	149	121, 91
2-Toluidine	107	106, 77
3,3'-Dichlorobenzidine	252	254, 126
3,4-Dimethylaniline	106	129, 127
3,5-Di-tert-butyl-4-Hydroxytol	205	220, 145
3-Nitroaniline	138	108, 65
4,6-Dinitro-2-methylphenol	198	51, 105
4-Bromophenyl phenyl ether	248	250, 141
4-chloro-2-methylaniline	106	144, 142
4-Chloro-3-methylphenol	107	144, 142
4-Chloroaniline	127	129
4-Chloroaniline-d4 **(Surrogate)**	131	133
4-Chlorophenyl phenyl ether	204	206, 141
4-Methylphenol	108	107
4-Nitroaniline	138	108, 65
4-Nitrophenol	139	109, 65
Acenaphthene	154	153, 152
Acenaphthene d10 **(ISTD)**	164	162, 160
Acenaphthylene	152	151, 153
Acetophenone	105	77, 51
Aniline	93	66
Aniline-d5 (**Surrogate**)	98	71,42
Anthracene	178	176, 179
Atrazine	200	173,215
Benzaldehyde	77	105,106
Benzidine	184	92, 185

Table 21 Characteristic Ions Of Semi-Volatile Organic Compounds		
Compound	**Primary Ion**	**Secondary Ion(s)**
Benzo(a)anthracene	228	229, 226
Benzo(a)pyrene	252	253, 125
Benzo(b)fluoranthene	252	253, 125
Benzo(g,h,i)perylene	276	138, 277
Benzo(k)fluoranthene	252	253, 125
Benzoic Acid	122	105, 77
Benzyl Alcohol	108	79, 77
Bis(2-chloroethoxy)methane	93	95, 123
Bis(2-chloroethyl)ether	93	63, 95
Bis(2-ethylhexyl)phthalate	149	167, 279
Bisphenol-A	213	228, 119
Butyl benzyl phthalate	149	91, 206
Caprolactam	113	55,56
Carbamazepine	193	236, 135
Carbazole	167	166, 139
Chrysene	228	226, 229
Chrysene d12 **(ISTD)**	240	120, 136
Coumarin	146	118, 63
Dibenz(a,h)anthracene	278	139, 279
Dibenzofuran	168	139
Diethylphthalate	149	177, 150
Dimethylphthalate	163	194, 164
Di-n-butylphthalate	149	150, 104
Di-n-octylphthalate	149	167, 43
Fluoranthene	202	101, 203
Fluorene	166	165, 167
Hexachlorobenzene	284	142, 249
Hexachlorobutadiene	225	223, 227
Hexachlorocyclopentadiene	237	235, 272
Hexachloroethane	117	201, 199
Indeno(1,2,3-cd)pyrene	276	138, 227
Isophorone	82	95,138
Kepone	272	237, 355
N,N-Dimethylaniline	120	122, 104
Naphthalene	128	129, 127
Naphthalene d8 **(ISTD)**	136	68
n-decane	43	57
Nitrobenzene	77	123, 65
Nitrobenzene-d5 **(Surrogate)**	82	128, 54
N-Nitrosodimethylamine	42	74, 44
N-Nitroso-di-n-propylamine	170	42,101,130
N-Nitrosodiphenylamine	169	168, 167
n-Octadecane	57	43, 85
o-Toluidine-d9 **(Surrogate)**	114	112, 42
Pentachloronitrobenzene	237	214,295
Pentachlorophenol	266	264, 268

Table 21 Characteristic Ions Of Semi-Volatile Organic Compounds		
Compound	**Primary Ion**	**Secondary Ion(s)**
Perylene d12 **(ISTD)**	264	260, 265
Phenanthrene	178	179, 176
Phenanthrene d10 **(ISTD)**	188	94, 80
Phenol	94	65, 66
Phenol-d5 **(Surrogate)**	99	42, 71
Phenyl ether	170	77, 115
Pyrene	202	200, 203
Pyridine	79	52, 51
Terphenyl-d14 **(Surrogate)**	244	122, 212

Attachment 1
Poor Performing Compounds

1,2,4,5-Tetrachlorobenzene
1,4-Dioxane
1-Naphthylamine
2,3,4,6-Tetrachlorophenol
2,4-Dimethylphenol
2,4-Dinitrophenol
2-Chloroaniline
2-Naphthylamine
3&4-Methylphenol
3'3-Dichlorobenzidine
4,6-Dinitro-2-methyl- phenol
4-Chloroaniline
4-Nitrophenol
Aniline
Atrazine
Benzaldehyde
Benzidine
Benzoic Acid
Benzyl Alcohol
Biphenyl
Caprolactam
Diphenylamine
Hexachlorocyclopentadiene
Hexachloroethane
n-Decane
n-Nitrosodimethylamine
o,o,o-Triethylphosphorothioate
o-Toluidine
Pentachloronitrobenzene
Pentachlorophenol
Phenol
Pyridine

These analytes are exempt from the ICV and CCV criteria as detailed in this SOP

Title: Purge and Trap for Aqueous Samples, SW846 Method 5030B and 5030C

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Approvals (Signature/Date):

Mary Baron Department Manager	2/9/21 Date	Dan Helfrich Health & Safety Manager / Coordinator	2/9/21 Date
Carl Armbruster Quality Assurance Manager	2/9/21 Date	Mark Acierno Laboratory Director	2/9/21 Date

Sylvanus Klusey — 2/9/21 Date
Organics Operations Manager

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1.0 Scope and Application

1.1 Analytes, Matrix(s), and Reporting Limits

1.1.1 This method describes purge and trap techniques for the analysis of volatile organic compounds in aqueous samples by SW846 Method 5030B and 5030C and high concentration soil sample extracts prepared using SW846 Methods 5035 or 5035A.

1.1.2 SW846 Methods 5030B and 5030C can be used for most volatile compounds that have boiling points below 200° C and are insoluble or slightly soluble in water. Water-soluble compounds can be analyzed for by this method; however, quantitation limits will be higher due to poor purging efficiency.

1.1.3 This sample introduction step is to be followed by determinative methods such as SW-846 Method 8015 for GC analysis or SW-846 8260 for GC/MS analysis.

1.1.4 On occasion clients may request modifications to this SOP. These modifications are handled following the procedures outlined in Sections 9 (*Review of Work Request*) and 19 (*Test Methods and Method Validation*) of TestAmerica Edison's Quality Assurance Manual (ED-QA-LQM).

2.0 Summary of Method

2.1 An aliquot of the aqueous sample is purged with helium (or nitrogen, where applicable) as in a closed sparging vessel. For medium or high level samples, an aliquot of the extract prepared by SW846 Methods 5035 or 5035A is combined with organic free reagent water and then purged. The volatile compounds are transferred from the aqueous phase to the vapor phase. The vapor is swept through a sorbent column where the volatiles are trapped. After purging is complete, the sorbent trap is heated and backflushed with helium (or nitrogen, where applicable) to desorb the volatiles onto a gas chromatographic column.

3.0 Definitions

3.1 For a complete list of definitions refer to Appendix 5 in the most current revision of TestAmerica Edison's Quality Assurance Manual (ED-QA-LQM).

4.0 Interferences

4.1 This method and the determinative methods that follow are susceptible to contamination from a number of sources. Potential sources of contamination include organic solvents used in other laboratory procedures, impurities in the purge gas, improper cleaning of syringes or purge vessels, and carryover from high level samples. Samples can be contaminated by the diffusion of volatile organics through the septum

during shipment or storage. Steps have been taken to ensure that these potential problems are eliminated from the laboratory.

4.2 The volatile laboratory is housed in a separate building, away from the organic extraction area where large quantities of organic solvents are used. No organic solvents are used or stored in the volatile laboratory.

4.3 The helium (or nitrogen, where applicable) used as purge gas passes through a solvent trap prior to its inlet into the purge and trap units.

4.4 A trip blank prepared from organic-free reagent water is carried through the sampling, storage, and analysis of each group of samples to check for such contamination.

4.5 Individual samples are each handled with a unique syringe that has been baked in a drying oven at 105°C to ensure the absence of volatile compounds.

4.6 Purge vessels are removed from the autosampler units after each use, rinsed, baked, returned to the units and pre-purged before the next use.

4.7 Carryover can occur anytime a high level sample is analyzed. Screening procedures are employed to ensure that a sample is analyzed at an appropriate dilution to minimize potential carryover. When a high level sample is analyzed, it is followed by the analysis of a reagent water blank. If another sample was analyzed after the high level sample, this sample is inspected carefully for signs of carryover. If this sample does not contain any of the compounds found in the high level sample, the system can be considered contamination free.

4.8 The analytical system is checked daily with the analysis of a method blank. This blank must meet all quality control criteria for the method before sample analysis may take place.

5.0 Safety

Employees must abide by the policies and procedures in the Corporate Environmental Health and Safety Manual (CW-E-M-001) and this document. This procedure may involve hazardous material, operations and equipment. This SOP does not purport to address all of the safety problems associated with its use. It is the responsibility of the user of the method to follow appropriate safety, waste disposal and health practices under the assumption that all samples and reagents are potentially hazardous. Safety glasses, gloves, lab coats and closed-toe, nonabsorbent shoes are a minimum.

Any questions pertaining to safety issues or procedures should be brought to the department manager or Edison Safety Officer.

5.1 Specific Safety Concerns or Requirements

- Latex, nitrile and vinyl gloves all provide adequate protection against the methanol used in this method.

- Purge vessels on purge-and-trap instruments can be pressurized by the time analysis is completed. Vent the pressure prior to removal of these vessels to prevent the contents from spraying out.

5.2 Primary Materials Used

The following is a list of the materials used in this method, which have a serious or significant hazard rating. **Note: This list does not include all materials used in the method. The table contains a summary of the primary hazards listed in the MSDS for each of the materials listed in the table.** A complete list of materials used in the method can be found in the reagents and materials section. Employees must review the information in the MSDS for each material before using it for the first time or when there are major changes to the MSDS.

Material (1)	Hazards	Exposure Limit (2)	Signs and symptoms of exposure
Hydrochloric Acid	Corrosive Poison	5 ppm-Ceiling	Inhalation of vapors can cause coughing, choking, inflammation of the nose, throat, and upper respiratory tract, and in severe cases, pulmonary edema, circulatory failure, and death. Can cause redness, pain, and severe skin burns. Vapors are irritating and may cause damage to the eyes. Contact may cause severe burns and permanent eye damage.
Methanol (MeOH)	Flammable Poison Irritant	200 ppm-TWA	A slight irritant to the mucous membranes. Toxic effects exerted upon nervous system, particularly the optic nerve. Symptoms of overexposure may include headache, drowsiness and dizziness. Methyl alcohol is a defatting agent and may cause skin to become dry and cracked. Skin absorption can occur; symptoms may parallel inhalation exposure. Irritant to the eyes.
1 – Always add acid to water to prevent violent reactions.			
2 – Exposure limit refers to the OSHA regulatory exposure limit.			

6.0 Equipment and Supplies

6.1 Instrumentation

Purge and trap unit that consists of three parts: the sample purger, the trap, and the concentrator.

6.1.1 Purge and trap units from several different manufacturers are used, depending upon the sample matrix and preparatory technique required.

6.1.2 Purge and trap units used include the the OI 4451 autosampler/4560 concentrator, the Archon 5100A automatic sampler/OI 4560 concentrator, OI 4551A autosampler/4660 Concentrator, Centurion

Autosampler/Encon, Archon/Encon, Solatek autosampler/Stratum, Aquatek 70/Tekmar 3000 concentrator and Archon/Evolution.

6.1.3 The purge chambers of each unit are designed to accept a 5mL sample with a water column at least 3cm deep. The headspace above the water has a volume less than 15 mL. The purge gas is introduced no more than 5 mm from the base of the water column. The purge gas passes through the water column as finely divided bubbles, each with a diameter of less than 3mm at the origin.

6.1.4 Different sorbent traps are used, depending on the determinative method that follows the purge and trap procedure. Alternate traps may be used provided the adsorption and desorption characteristics are equivalent to those of the trap recommended by the method.

6.1.5 The VOCARB 3000 (Supelco) is used in the Encon, Tekmar 3000 and Evolution. This trap is 25cm long with an inside diameter of 0.105 inches. The trap is packed with 10.0cm Carbopack B, 6.0 cm Carboxin 1000, and 1cm Carboxin 1001.

6.1.6 The OI Analytical purge trap #10 is used in the OI 4560 and 4660. This trap is 25cm long and has an inside diameter of 0.105 inches. The trap is packed to contain the following adsorbents: Tenax/silica gel/ carbon molecular sieve.

6.1.7 The concentrator of each unit is capable of rapidly heating the trap to 260°C and holding at that temperature for the duration of the desorb time.

6.1.8 Gas chromatograph. HP 5890/Agilent 6890N/7890 equipped with temperature programming capability.

6.1.9 GC column:

- 75M long x 0.53mm ID, J&W DB-624 capillary column with 3um film thickness or similar phase.
- 20M long x 0.18 ID, DB-624 or Rtx-VMS with 1 um film thickness.
- 30M long x 0.25 mm ID x 1.4 um film thickness Rtx-624.

6.1.10 Injection port liners. HP 18740-80200 or equivalent.

6.2 Supplies

- Microsyringes. 10 uL to 1000 uL.
- Syringes. 5mL and 30mL gas-tight.
- Volumetric flasks. Class “A” glassware, 10mL, 50mL, and 100 mL.

- Vials. 2mL amber glass with screw cap with Teflon-faced septa.
- VOA vials. 40ml glass with PTFE-faced septa

7.0 Reagents and Standards

7.1 Organic free reagent water. Distilled water purchased from Poland Spring.

7.2 Methanol: Purge and trap grade, purchased from JT Baker. (Cat # 9077-02)

7.3 Standard preparation and use is described in detail in each of the determinative methods.

8.0 Sample Collection, Preservation, Shipment and Storage

8.1 Aqueous samples are collected in the appropriate containers and preserved according to the determinative method requirements.

8.2 All aqueous samples are protected from light and stored at 4°C in an area free of organic solvents. No standards or solvents are stored in the sample refrigerators or in the surrounding area.

8.3 All aqueous samples are analyzed within 14 days of collection.

8.4 The collection, preservation and hold times for soil samples prepared via SW846 Method 5035 or 5035A are addressed in the corresponding TestAmerica Edison SOPs (see references in Section 15).

9.0 Quality Control

9.1 Specific quality control procedures are outlined in each of the determinative methods that follow this purge and trap technique of sample introduction. Standard quality assurance practices are used with all methods.

9.2 An initial demonstration of accuracy and precision is performed for each determinative method. This demonstration is done for each sample introduction technique.

9.3 Reagent water blanks are analyzed to ensure that reagents and/or sample dilutions are free of interferences.

9.4 Method blanks are prepared and analyzed in the same manner as samples, and are carried through the entire analytical procedure to show that each system is in control.

9.5 Each analysis batch contains spike and duplicate data as outlined in the method, as well as a laboratory control sample (blank spike) to show that replicate data can be generated and appropriate concentrations of target analytes recovered.

10.0 Procedure

10.1 This section provides guidance for the analysis of aqueous samples and/or methanol extracts prepared by SW846 Method 5035 by purge and trap techniques. Determinative methods SW846 Methods 8015 or 8260 will follow this purge and trap procedure

10.2 Instrument operating parameters are set at the beginning of a method of analysis and remain constant throughout the calibration and sample analysis steps. See each determinative method to verify specific instrument operating parameters.

Table 1: Example Instrument Operating Parameters

Purge and trap unit	
Purge Time	11 minutes
Dry Purge	1 Minutes
Purge Gas	Helium (or nitrogen, where applicable)
Purge Flow	40-45 ml/min
Purge Temp	Water: Ambient, Solids: 40^{0}C
Trapping Temp	Ambient, <30^{0}C
Desorb Time	1 Minute
Desorb Temp	VOCARB: 260^{0}C
Gas chromatograph	
Injector	180^{0}C
Carrier Gas	Helium
Carrier Flow	6 ml/min
Oven Program	35 - 250^{0}C with 2 ramps
Run Time	26 Minutes
Mass Spectrometer	
Electron Energy	70 volts (nominal)
Mass range	35-260 AMU
Scan time	0.9 sec./scan
Source Temp	200^{0}C
Separator Temp	180^{0}C

10.3 Screen all samples according to TestAmerica Edison SOP No. ED-GCV-001, (*Screening for Volatile Organics, Static Headspace with GC FID,, SW846 Method 5021* **,** current revision). Analysts are furnished with approximate dilution factors prior to analysis.

10.4 Allow all samples and standards solutions to warm to ambient temperature before use.

10.5 Prior to performing any purge and trap analysis, prepare each system in the following manner:

10.5.1 Condition concentrators by baking the trap at their bake temperatures for 5-12 minutes. Also condition the GC column by baking at 200°C.

10.5.2 Access the FLUSH menu on the Archon Autosampler. Rinse the syringe barrel with two 6mL portions of rinse water. Turn off the helium switch, and fill the water reservoir with reagent water. Visually inspect the standard vial and fill with the appropriate internal standard/surrogate mix. Turn the helium switch back on. Prime the standard loop by accessing the SYSTEM/MAINTENANCE/STANDARD CONTROL menu. Empty the waste reservoir.

10.5.3 Prepare other referenced autosamplers by filling internal standard reservoirs with the appropriate IS/SURR, empty the waste reservoir and fill the water rinse reservoir.

10.6 Prior to using this sample introduction technique, the GC or GC/MS system must be tuned and calibrated as required by each of the determinative methods (reference the applicable TestAmerica Edison SOP; see references in Section 15). All method requirements for tune, calibration, and blank analysis must be met prior to sample analysis.

10.7 Aqueous samples

10.7.1 Load the sample vials directly into the autosampler tray. The appropriate internal standard and/or surrogate solution will be added to the sample by the autosampler unit immediately before a sample is purged.

10.7.2 The process of using autosamplers also destroys the validity of the sample by puncturing the septum. Any re-analysis should be done using second vial of sample. If only one vial exists, do not use this autosampler.

10.8 Medium or high level sample

10.8.1 Prepare samples by adding the appropriate amount of sample to a 50mL volumetric flask containing reagent water.

10.8.2 Add the appropriate amount of sample to the flask. Dilute to volume with reagent water, cap, and invert three times. Pour the diluted sample into a 5mL syringe or a 40mL VOA vial.

10.8.3 If a dilution is required, reduce the volume of sample added to the

10.8.4 Load sample onto the autosampler.

10.8.5 Purge the sample for 11 minutes, with one minute dedicated to removing moisture from the purge and trap system (dry purge). Times may vary among different determinative methods.

10.8.6 After purging is complete, desorb the sample onto the GC column by rapidly heating the trap to 260°C and backflushing it with helium (or nitrogen, where applicable). (Trap temperatures will vary based on type used)

10.8.7 Begin the GC temperature program and data acquisition.

10.8.8 Re-condition the trap by baking at 260°C for 8-12 minutes.

10.8.9 Cool the trap to (<31°C). The trap is now ready for the next sample.

10.8.10 Process data using Chrom software, and evaluate as per each determinative method.

10.9 Technical acceptance criteria for sample analysis.

10.9.1 The samples must be analyzed on a GC or GC/MS system meeting the initial calibration, continuing calibration and blank technical acceptance criteria of the determinative methods (reference the applicable TestAmerica Edison SOP; see references in Section 15).

10.9.2 The sample must be analyzed within the required holding time.

10.9.3 The sample must have an associated method blank meeting the blank technical acceptance criteria.

10.9.4 The percent recovery of each of the system monitoring compounds in the sample must be within the acceptance windows.

10.9.5 After analyzing a sample that exceeds the initial calibration range the analyst must either analyze an instrument blank (using the same purge inlet if using an auto sampler) which must meet technical acceptance criteria for blank analysis or monitor the sample analyzed immediately after the contaminated sample for all compounds that were in the contaminated sample that exceeded the calibration range.

10.10 Corrective Action for Sample Analysis

10.10.1 Samples must meet technical acceptance criteria of the determinative methods (reference the applicable TestAmerica Edison SOP; see references in Section 15) before reporting data.

10.10.2 Corrective action must be completed prior to sample analysis if analysis failed to meet instrument performance checks, initial calibration, continuing calibration and method blanks.

10.10.3 Corrective action for system monitoring compounds and internal standard compounds that fail to meet acceptance criteria must be completed prior to sample analysis.

10.11 If any of the system monitoring compounds and internal standard compounds fail to meet acceptance criteria of the determinative methods (reference the applicable TestAmerica Edison SOP; see references in Section 15):

10.11.1 Check all calculations, instrument logs, the system monitoring compound and internal standard compound spiking solutions and the instrument operation. If the calculations were incorrect, correct calculations and verify that the system monitoring compound recoveries and internal standard compound responses meet acceptance criteria

10.11.2 Check the preparation of the internal standards and system monitoring compounds for concentration and expiration.

10.11.3 Verify that the instrument is operating correctly.

10.12 Data that fails to meet minimum acceptance criteria will be annotated (flagged) with qualifiers and/or appropriate narrative comments defining the nature of the outage. If applicable, a *Non-Conformance Memo* (NCM) (TestAmerica Edison Work Instruction No. EDS-WI-012) will be initiated in order to provide for investigation and follow-up. For further corrective actions and contingencies for handling out-of-control or unacceptable data see *Standard Operating Procedure for Control of Non-Conformances and Corrective Action* (TestAmerica Edison SOP No. EDS-GEN-003).

11.0 Calculations / Data Reduction

11.1 For specific calculations refer to the applicable determinative methods (reference the applicable TestAmerica Edison SOP; see references in Section 15

12.0 Method Performance

12.1 Method Detection Limit Study (MDL)

The method detection limit (MDL) is the lowest concentration that can be detected for a given analytical method and sample matrix with 99% confidence that the analyte is present. The MDL is determined according to the laboratory's MDL procedure in Section 19 (Test Methods and Method Validation) of TestAmerica Edison's Quality Assurance Manual (ED-QA-LQM). MDLs reflect a calculated (statistical) value determined under ideal laboratory conditions in a clean matrix, and may not be achievable in all environmental matrices. The laboratory maintains MDL studies for analyses performed; these are verified at least annually unless method requirements require a greater frequency.

12.2 Demonstration of Capabilities

For DOC procedure refer to Section 19 in the most current revision of TestAmerica Edison's Quality Assurance Manual (ED-QA-LQM).

12.3 Training Requirements

Refer to TestAmerica SOP No. ED-GEN-022, Training, for the laboratory's training program.

13.0 Pollution Control

13.1 Pollution prevention encompasses any technique that reduces or eliminates the quantity or toxicity of waste at the point of generation. Numerous opportunities for pollution prevention exist in laboratory operation. The USEPA has established a prevention hierarchy of environmental management techniques that places pollution prevention as the management option of first choice. Whenever feasible, laboratory personnel should use pollution prevention techniques to address their waste generation. When wastes cannot be feasibly reduced at the source, the agency recommends recycling as the next best option.

13.2 The quantity of chemical purchased should be based on expected usage during its shelf life and disposal cost of unused material. Actual reagent preparation volumes should reflect anticipated usage

14.0 Waste Management

14.1 The U.S. Environmental Protection Agency requires that laboratory waste management practices conducted be consistent with all applicable rules and regulations. All waste will be disposed of in accordance with Federal, State and Local regulations. Where reasonably feasible, technological changes have been implemented to minimize the potential for pollution of the environment. Employees will abide by this method and the policies in section 13 of the Corporate Environmental Health and Safety Manual (CW-E-M-001) for "Waste Management and Pollution Prevention."

14.2 The following waste streams are generated as a result of this analysis:

- Laboratory Generated Aqueous Waste (aqueous VOA vials – used and unused). This waste may have a pH of less than 2.0. These vials are collected in satellite accumulation. The vials are then transferred to the waste room. These vials are passed through a vial crusher and the liquid portion is separated from the solid portion. The solid is dumped into the municipal garbage. The liquid is pumped into the neutralization system where it is neutralized to a pH of 6 to 9 with sodium bicarbonate (Seidler Chemical SC-0219-25). When neutralization is complete, the material is transferred to the municipal sewer system.

- Expired Standards – The vials are collected in a 1 gallon polyethylene bucket. These vials are then transferred to an open top 55 gallon steel or polyethylene

waste drum. These drums are transported to a waste facility for proper disposal.

- Soil Retain Samples - These samples if not flagged in the system for any hazardous constituents are transferred to poly-lined cubic yard boxes. These boxes when full are sent to stabilization or incineration. These materials are sent out as hazardous for lead and chromium
  Teris Profile Number (incineration): 50016710
  Onyx Profile Number: (stabilization) 402535

- Methanol Preserved Samples - Methanol preserved sample vials are collected in satellite accumulation and then transferred to a 55 gallon open top steel waste drum in the waste room. This drum is then removed by a waste vendor for incineration.

  Teris Profile Number: 50016652
  Onyx Profile Number: 282493

- Returned Methanol Preservative - Methanol preserved vials are collected in satellite accumulation and then transferred to 55 gallon open top steel waste drums in the waste room. These waste drums are then removed by a waste vendor for incineration.

  Teris Profile Number: 50016652
  Onyx Profile Number: 282493

15.0 References / Cross-References

15.1 United States Environmental Protection Agency, *Method 5030C: Purge and Trap for Aqueous Samples*, Test Methods for Evaluating Solid Wastes, SW846 Third Edition, Volume 1B: Laboratory Manual, Physical/Chemical Methods, Revision 3 2003.

15.2 United States Environmental Protection Agency , *Method 5035A*: *Closed-System Purge-and-Trap and Extraction for Volatile Organics in Soil and Waste Samples*, Test Methods for Evaluating Solid Wastes, SW846 Third Edition, Volume 1B: Laboratory Manual, Physical/Chemical Methods, Revision 0, July 2002.

15.3 United States Environmental Protection Agency, “Method 8260D, Volatile Organic Compounds by Gas Chromatography/Mass Spectrometry (GC/MS)”, Test Methods for Evaluating Solid Waste, SW846, Update VI, Revision 4, June 2018.

15.4 United States Environmental Protection Agency, *Method 8015D*: *Non-Halogenated Organics using GC-FID,* Test Methods for Evaluating Solid Wastes, SW846, Laboratory Manual, Physical/Chemical Methods, Revision 4, June 2003.

15.5 TestAmerica Edison SOP No. ED-GCV-008, *Gasoline Range Organics using GC-FID Method, SW846 Method 801D,* current revision.

15.6 TestAmerica Edison SOP No. ED-MSV-002, *Closed System Purge and Trap and Extraction for Volatile Organics in Soil, SW846 Method 5035A,* current revision.

15.7 TestAmerica Edison SOP No. ED-GCV-001, *Screening for Volatile Organics, Static Headspace with GC FID, SW846 Method 5021* **,** current revision.

15.8 TestAmerica Edison Document No. ED-QA-LQM, *Laboratory Quality Manual*, most current revision.

15.9 TestAmerica Edison SOP No. EDS-GEN-003, *Standard Operating Procedure for Control of Non-Conformances and Corrective Action*, most current revision

16.0 Method Modifications

Not applicable

17.0 Attachments

Not applicable

18.0 Revision History

Revision 10, dated: 02/09/2021

- Updated to Eurofins branding;
- Updated Lab Quality Manual section references as applicable throughout
- Updated Section 15 (References) as needed.

Revision 9, dated: 06/26/2013:

- Revised title of SOP to include reference to SW846 5030C.
- Section 1.1.1 and throughout document as appropriate: added reference to SW846 5030C.
- Section 1.1.1: inserted word ‘extracts’ in reference to analysis of high concentration and soil samples by SW846 5035/5035A.
- Section 10.8.10: replaced Target reference with Chrom (newly implemented chromatography data system)
- Section 15.3: corrected method reference to ‘5035A’.
- Section 15.14: added reference to SW8465030C

Revision 8, dated: 11/27/2012:

- Updated referenced Lab Quality Manual section and appendix numbers as appropriate.

Revision 7, dated: 10/02/2008

- Revised SOP format in accordance with TestAmerica Corporate Quality SOP No. CW-QS-002 (Rev 0) Writing a Standard Operating Procedure (SOP).

- Section 1.2 Added reference to Quality Assurance Manual for method modifications. Added references to related methods and SOPs for determinative methods.
- Section 2 (and throughout document): added option of purging with nitrogen gas as applicable.
- Section 3: revised to reference new location for definitions.
- Section 5: Revised to include most up to date corporate health and safety references and information.
- Section 6: Newer instrumentation and GC column types added. See sections 6.1.2 and 6.1.9. All references to Tekmar instruments 2016 and 2000 and procedures to use with it removed in all relevant sections.
- Section 11: Removed reference to Organic Calculation SOP.
- Section 12: updated and revised the MDL requirements to reflect text in the current revision of the TestAmerica Edison Laboratory Quality Manual (LQM).
- References: Expanded to include more specific SOP references
- Section 18: Added this Revision History section

Title: Closed System Purge and Trap and Extraction for Volatile Organics in Soil , SW846 Method 5035A

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Approvals (Signature/Date):

Reddy Pakanati	04/11/19 Date	Dan Helfrich	04/11/19 Date
VOA Department Manager		Health & Safety Manager / Coordinator	
Carl Armbruster	04/11/19 Date	Mark Acierno	04/11/19 Date
Quality Assurance Manager		Laboratory Director	
Sylvanus Klusey	04/11/19 Date		
Operations Manager - Organics			

1.0 Scope and Application

1.1 Analytes, Matrix(s), and Reporting Limits

1.1.1 This method describes a closed system purge and trap procedure for the analysis of volatile organic compounds in soils, sediments and solid wastes.

1.1.2 This method is designed for use on samples containing low levels of volatile compounds (0.5 to 200ug/kg), and requires the use of hermetically sealed sample vials, which limits a samples exposure to the atmosphere, thereby minimizing the loss of volatile compounds.

1.1.3 Method 5035A can be used for most volatile organic compounds that have boiling points below 200°C and are insoluble or slightly soluble in water. Water-soluble compounds can be included in this technique, but will have higher quantitation limits due to poor purging efficiency.

1.1.4 Method 5035A also includes sample collection and preparation procedures for medium and high concentration soil samples (i.e., >200 ug/kg). Medium and high level soil samples are prepared either by field preservation of a soil sample in methanol or by creating a methanol extract from a soil sample that was collected as bulk soil.

1.1.5 The preparation steps described for medium and high level samples will be followed by the aqueous purge and trap procedure detailed in TestAmerica Edison SOP No. ED-MSV-001(*Purge and Trap for Aqueous Samples, SW846 Method 5030, current revision*).

1.1.6 The preparation and sample introduction steps described for low-level soils will be followed by analysis using TestAmerica Edison SOP Nos. ED-GCV-006 (*Gasoline Range Organics using GC FID Method SW846 8015, current revision*) or ED-MSV-005 (*SW846 Method 8260B Volatile Organic Compounds by Gas Chromatography/Mass Spectrometry, current revision*) as applicable.

1.1.7 On occasion clients may request modifications to this SOP. These modifications are handled following the procedures outlined in Sections 7 (*Review of Work Request*) and 19 (*Test Methods and Method Validation*) of TestAmerica Edison's Quality Assurance Manual (ED-QA-LQM).

2.0 Summary of Method

2.1 **Low Level Soils:** Volatile organic compounds are determined by collecting a 5g-soil sample with a specially designed sampling device (ex., En Core® Sampler) in which the sample is stored without headspace. Upon receipt and within 48 hours of sampling, the soil is placed in a pre-weighed vial containing 5 mL of reagent water and a stir bar. The vial containing the soil is then frozen extending the hold time to 14 days. The same samples may be received from the field in a 40 mL vial with 5 mL water (ex., Terra Core® Sampler). Immediately prior to analysis, the soil is thawed, reagent water, surrogate spiking solution and/or internal standard

spiking solution are added to the vial by the automatic sampler, without opening the vial to the atmosphere. The sample vial is heated and purged with helium while being magnetically stirred. The volatile components travel through a transfer line to a sorbent trap. When purging is complete, the trap is heated and backflushed with helium to desorb the sample components on to a GC column for separation and analysis by the appropriate determinative method.

2.2 **Medium Level Soils:** Medium level soils are soils that were originally intended to be analyzed by a low level soil method, but show contaminants >200ug/kg when screened. A methanol extract is prepared by extracting 5g of soil with 10mL of methanol. The volatiles are effectively transferred from the soil to the methanol. A portion of the methanol extract is introduced into the GC or GC/MS system by using the purge and trap Method SW846 5030 followed by the appropriate determinative method.

2.2.1 Samples may also arrive from the field as 5 mL or 10 mL methanol preserved samples. These are considered to be medium level prepared samples.

2.3 **High Level Soils:** The high level soil method acts as a combined preservation and extraction technique. 25mL of methanol /surrogate solution is placed in each VOA vial and weighed prior to field sampling. Approximately 10g of soil is added to each vial in the field. Upon returning to the lab, the vial is weighed again, and the difference between the two weights is recorded as the initial weight of sample. As with the medium level method, the volatiles are effectively transferred to the methanol. A portion of the methanol extract is introduced into the GC or GC/MS system by using the purge and trap Method SW846 5030 followed by the appropriate determinative method.

2.4 **Waste Dilution:** Organic waste samples are prepared by extracting 1g of sample with 10mL of methanol / surrogate solution. A portion of the methanol extract is introduced into the GC or GC/MS system by using the purge and trap Method SW846 5030 followed by the appropriate determinative method.

3.0 Definitions

3.1 For a complete list of definitions refer to Appendix 2 in the most current revision of TestAmerica Edison's Quality Assurance Manual (ED-QA-LQM).

4.0 Interferences

4.1 This method and the determinative methods that follow are susceptible to contamination from a number of sources. Potential sources of contamination include organic solvents used in other laboratory procedures, impurities in the purge gas, improper cleaning of syringes or purge vessels, and carryover from high level samples. Samples can be contaminated by the diffusion of volatile organics through the septum during shipment or storage. Steps have been taken to ensure that these potential problems are eliminated from the laboratory.

4.2 The volatile laboratory is located in a separate building, away from the organic extraction area where large quantities of organic solvents are used. No organic solvents are used or stored in the volatile laboratory.

4.3 The nitrogen used as purge gas passes through a solvent trap prior to its inlet into the purge and trap units.

4.4 A trip blank prepared from organic-free reagent water is carried through the sampling, storage, and analysis of each group of samples to check for such contamination.

4.5 Individual samples are each handled with a unique syringe that has been baked in a drying oven at 105°C to ensure the absence of volatile compounds.

4.6 Purge vessels are removed from the autosampler units after each use, rinsed, baked, returned to the units and pre-purged before the next use.

4.7 Carryover can occur anytime a high level sample is analyzed. Screening procedures are employed to ensure that a sample is analyzed at an appropriate dilution to minimize potential carryover. When a high level sample is analyzed, it is followed by the analysis of a reagent water blank. If another sample was analyzed after the high level sample, this sample is inspected carefully for signs of carryover. If this sample does not contain any of the compounds found in the high level sample, the system can be considered contamination free.

4.8 The analytical system is checked daily with the analysis of a method blank. This blank must meet all quality control criteria for the method before sample analysis may take place.

4.9 Potential cross-contamination of samples stored in lab refrigerators is monitored by preparation, analysis and evaluation of storage blanks. Storage blanks are prepared by filling 40 mL VOA vials with reagent water and placing one in each refrigerator. After one week, the storage blanks are removed and analyzed. Additional details can be found in TestAmerica Edison SOP No. ED-SPM-004, *Sample Storage & Handling Procedures for Mitigation of Sample and Laboratory Contamination,* current revision.

5.0 Safety

Employees must abide by the policies and procedures in the Corporate Environmental Health and Safety Manual (CW-E-M-001) and this document. This procedure may involve hazardous material, operations and equipment. This SOP does not purport to address all of the safety problems associated with its use. It is the responsibility of the user of the method to follow appropriate safety, waste disposal and health practices under the assumption that all samples and reagents are potentially hazardous. Safety glasses, gloves, lab coats and closed-toe, nonabsorbent shoes are a minimum.

Any questions pertaining to safety issues or procedures should be brought to the department manager or Edison Safety Officer.

5.1 Specific Safety Concerns or Requirements

5.1.1 Latex, nitrile and vinyl gloves all provide adequate protection against the methanol used in this method.

5.1.2 Purge vessels on purge-and-trap instruments can be pressurized by the time analysis is completed. Vent the pressure prior to removal of these vessels to prevent the contents from spraying out.

5.2 Primary Materials Used

The following is a list of the materials used in this method, which have a serious or significant hazard rating. **Note: This list does not include all materials used in the method. The table contains a summary of the primary hazards listed in the MSDS for each of the materials listed in the table.** A complete list of materials used in the method can be found in the reagents and materials section. Employees must review the information in the MSDS for each material before using it for the first time or when there are major changes to the MSDS.

Material (1)	Hazards	Exposure Limit (2)	Signs and symptoms of exposure
Methanol (MeOH)	Flammable Poison Irritant	200 ppm-TWA	A slight irritant to the mucous membranes. Toxic effects exerted upon nervous system, particularly the optic nerve. Symptoms of overexposure may include headache, drowsiness and dizziness. Methyl alcohol is a defatting agent and may cause skin to become dry and cracked. Skin absorption can occur; symptoms may parallel inhalation exposure. Irritant to the eyes.
1 – Always add acid to water to prevent violent reactions.			
2 – Exposure limit refers to the OSHA regulatory exposure limit.			

6.0 Equipment and Supplies

6.1 Instrumentation

6.1.1 Purge and trap units from several different manufacturers are used, depending upon the sample matrix and preparatory technique required. A purge and trap unit consists of three parts: the sample purge unit, the trap, and the concentrator. Unit configurations currently in use are:

- OI Analytical 4551 Automatic Sampler/4560 concentrator;
- Archon 5100A Automatic sampler/ OI Analytical 4560 concentrator;
- EST Centurion Autosampler/ EST Encon concentrator;
- Archon Autosampler/EST Encon concentrator.

6.1.2 A VOCARB 3000 trap from Supelco is used in the Encon concentrator. The trap is 25cm long with an inside diameter of 0.105 inches. The trap is packed with 10.0cm Carbopack B, 6.0 cm Carboxin 1000, and 1cm Carboxin 1001

6.1.3 An OI analytical purge trap #10 is used for the OI 4560 concentrator. The trap is 25cm long with an inside diameter of 0.105 inches. The trap is packed to contain the following absorbents: Tenax/silica gel/carbon molecular sieve.

6.1.4 Alternate traps may be used provided the adsorption and desorption characteristics are equivalent to those of the trap recommended by the method.

6.1.5 Both the Encon and OI concentrators are capable of rapidly heating the trap to 260°C and holding at that temperature for the duration of the desorb time.

6.1.6 Gas chromatograph: HP 5890/Agilent 6890/7890 equipped with temperature programming capability.

6.1.7 GC column: reference the applicable determinative method for column specifics.

6.1.8 Detector: Flame Ionization Detector or Mass Spectrometer (HP5971/5972/Agilent 5973). Reference the applicable determinative method for detector specifics.

6.1.9 Data system: HP Chemstation II for data acquisition and HP UNIX based TARGET software for data processing.

6.2 Equipment and Supplies

6.2.1 Freezer: Capable of holding a temperature of -7℃ to -20℃.

6.2.2 Refrigerator: Capable of holding a temperature of 4℃ ±2℃

6.2.3 Top loading analytical balance.

6.2.4 Portable field balance. Capable of weighing to 0.01g.

6.2.5 Microsyringes: 10 uL to 1000 uL.

6.2.6 Syringes: 5mL, 10mL, and 30mL gas-tight.

6.2.7 Volumetric flasks: Class “A” glassware, 10mL , 50mL , and 100 mL.

6.2.8 VOA vials: 40 mL glass with PTFE –faced septum.

6.2.9 En Core® Sampler. Designed to take a 5g-soil sample. Sealed to prevent loss of volatiles.

6.2.10 Vials: 2mL amber glass with screw cap with Teflon-faced septa.

6.2.11 Spatula: Narrow, stainless steel.

6.2.12 Stir bars: PTFE coated, small enough to spin freely inside a VOA vial.

7.0 Reagents and Standards

7.1 Reagents

7.1.1 Organic free reagent water. Distilled water purchased from Poland Spring.

7.1.2 Methanol. Purge and trap grade, purchased from JT Baker. (Cat # 9077-02)

7.1.2.1 Each lot of methanol and hydrochloric acid is screened for contaminants before being used for analysis as detailed in TestAmerica Corporate Quality SOP No. CA-Q-S-001 (*Solvent & Acid Lot Testing & Approval*) and TestAmerica Edison SOP No. ED-GEN-023 (*Bulk Solvent Testing and Approval*).

7.2 Standards

7.2.1 Calibration standard preparation and use is described in detail in each of the determinative methods.

7.2.2 Method 8260 Methanol/Surrogate solution (2.5 ppm) is prepared using purge and trap grade methanol and neat a,a,a-Triflurotoluene solution. Initially a primary solution is prepared at 2500 ppm:

Standard Name	Vendor	Catalog #	Volume added	Concentration of Stock Std.	Concentration of Standard	Total Volume Volume of MeOH
1°Surrogate Mix: 4-Bromofluorobenzene Toluene-d8 1,2-Dichloroethane-d4	Sigma Aldrich	 B67201 151998 396540	 1585 ul 2678 ul 1932 ul	Neat	2500ppm	1000 ml

The 2500 ppm solution is used to prepare a 2.5 ppm Method 8260 Methanol/Surrogate working solution by diluting 1.0 ml of 2500 ppm stock to 1000ml with purge and trap grade methanol.

7.2.3 Method 8015 (GRO) Methanol/Surrogate solution (1.25 ppm) is prepared by diluting 0.5ml of the 2500 ppm stock (see Section 7.1.4) to 1000 ml with purge and trap grade methanol.

8.0 Sample Collection, Preservation, Shipment and Storage

8.1 Low Level Soils

Sample containers used to collect low-level soils are 40 mL VOA vials with PTFE-faced septa (closed system purge and trap), En Core® Samplers, Terra Core® kits or bulk soil.

8.1.1 For the 40 mL VOA vial (closed system purge and trap) collection procedure, add 1 clean magnetic stir bar and 5 mL of reagent water to each vial.

8.1.1.1. Cap the vial and weigh to the nearest 0.01g. Record this as the tare weight.

8.1.1.2. Collect approximately 5g of soil using an appropriate sampling device and add 5g to each of 3 vials for low level analysis.

8.1.1.3. Collect a fourth aliquot for field methanol preservation into a pre-weighed vial containing 10 mL of methanol.

8.1.1.4. Closed system purge and trap collection vials are re-weighed to determine actual sample weight. Record the sample weight on the sample vial.

8.1.1.5. Within 48 hours of sampling the collection vials are placed into a freezer (on their side to prevent breakage) at -7°C to -20°C. Freezing in this manner extends the holding time to 14 days from sampling.

8.1.2 For En Core® Sampler collection, collect up to 3 En Core® Sampler for low level preservation via freezing and a fourth for medium level preservation with methanol (Section 8.2 details the medium level preservation procedures for En Core® samplers). The low level preservation procedures are:

8.1.2.1. The En Core® device is received at the lab sealed without headspace with a cap and viton o-rings.

8.1.2.2. For each En Core® requiring low level preservation, add 5ml of reagent water and a magnetic stir bar to a 40 ml vial. Cap the vial.

8.1.2.3. Weigh each vial to the nearest 0.01g. Record this as the tare weight.

8.1.2.4. Transfer the contents of each En Core® to separate preweighed vials within 48 hours of sampling.

8.1.2.5. Reweigh the vial. Record the weight on each sample vial.

8.1.2.6. The vials are placed into a freezer (on their side to prevent breakage) at -7°C to -20°C. Freezing in this manner extends the holding time to 14 days from sampling.

8.1.2.7. A bulk soil sample must also be collected for the purposes of percent moisture analysis for dry weight determination.

8.1.2.8. Vials are removed from the freezer the day of analysis for thawing.

8.1.2.9. Stir bars may be reused after the vial is analyzed. Retrieve the stir bar from each vial using the magnetic stir bar retriever, rinse the stir bar with methanol and bake at 105°C for 2 hours.

8.1.3 Terra Core® Kits. This option requires samples to be field preserved:

8.1.3.1. The sample kit typically includes two pre-weighed 40 mL vials containing 5 mL of water with a small magnet stir bar and one or two pre-weighed 40 mL vials containing pre-measured and recorded amount of methanol.

8.1.3.2. Approximately 5g of sample is placed into each vial by the field sampler.

8.1.3.3. The vials containing the samples are re-weighed in the lab and the net sample weight is recorded for each vial. The vials containing the sample and water are frozen up until the time of analysis.

8.1.3.4. The methanol preserved samples are considered to be medium level prepared samples and do not require freezing but they must be refrigerated at 4℃ until time of analysis.

8.1.3.5. A bulk soil sample must also be collected for the purposes of percent moisture analysis for dry weight determination.

8.1.3.6. Stir bars may be reused after the vial is analyzed. Retrieve the stir bar from each vial using the magnetic stir bar retriever, rinse the stir bar with methanol and bake at 105°C for 2 hours.

8.1.4 Bulk soil. Typically consists of soil collected in 8-oz or 16-oz glass jars. At client request bulk soil samples can be sub-sampled, preserved and analyzed for volatile organics. Soils collected for low level VOA analysis as bulk soil in jars will be preserved as described above in Section 8.1.1. Documentation of preservation is recorded in the Soil Preservation Logbook.

8.2 Medium Level Soils

8.2.1 Medium level refers to any soil sample extracted in methanol not preserved in the field. The fourth Encore sample collected (as described in Section 8.1.2 above) will be prepared as a medium level soil in case the analysis of a low level preserved aliquot exhibits concentrations of target compounds exceeding 200 ug/kg. All medium level extracts must be refrigerated at 4℃ until time of analysis.

8.2.1.1. Prepare the medium level soil by adding 5 g of soil or Encore to 10 mL of Methanol / Surrogate solution (either the 8260 solution as described in Section 7.1.4 ***or*** the 8015 solution as described in Section 7.1.5. depending upon the method to be analyzed). All medium level extracts shall be prepared within 48 hours of collection. The weight of soil used to prepare the extract is recorded on the sample vial.

8.2.1.2. Since methanol extract results tend to increase with time as the sample contact time increases, a minimum contact time of one day is allowed ***or*** the soil is sonicated for 20 minutes.

8.2.1.3. Any soil requiring medium level preservation but collected out of compliance with method 5035A (bulk soil without preservative or not in an Encore device) will be prepared in the same manner as in section 8.2.1.1. Documentation will be noted in the soil preservation log.

8.3 High Level Soils

8.3.1 High level soils are field collected using laboratory prepared 40 ml VOA vials (with PTFE-faced septa) containing 25 mL of the Methanol/Surrogate solution (either the 8260 solution as described in Section 7.1.4 ***or*** the 8015 solution as described in Section 7.1.5. depending upon the method to be analyzed).

8.3.2 The vials containing the Methanol/Surrogate solution are weighed using a barcode reader and an analytical balance. The bottle ID and weight (Weight 1) are recorded in the methanol-tracking program.

8.3.3 The containers are issued to the field as required.

8.3.4 Field samplers are directed to weigh 10 g of sample using a field balance. They must carefully add the 10 g sample to the sample vial containing the Methanol/Surrogate solution, taking care not to spill any of the solution from the vial.

8.3.5 Field samplers are directed to wipe the neck of the vial carefully to remove any soil around the threads. This helps to ensure a good seal once the vial is capped. Field samplers are further directed to cap the vial, shake gently, dispersing the soil into the methanol. An additional aliquot of soil without any methanol or preservative must be collected for use in determining percent moisture (i.e., for dry weight determination).

8.3.6 Upon the return to the laboratory, the sample vials (now containing both the Methanol/Surrogate solution and approximately 10g of soil) are reweighed using the barcode reader and analytical balance. Record this weight (Weight 2) in the methanol tracking system. Calculate the initial weight of soil by subtracting Weight 1 from Weight 2.

8.3.7 All samples for volatile analysis are protected from light and stored at 4°C±2°C in an area free of organic solvents. No standards or solvents are stored in the sample refrigerators or in the surrounding area.

8.3.8 All samples preserved in this manner must be analyzed within 14 days of collection.

9.0 Quality Control

9.1 Specific quality control procedures are outlined in each of the determinative methods that follow this purge and trap technique of sample introduction. Standard quality assurance practices are used with all methods.

9.2 Reagent water blanks are analyzed to ensure that reagents and/or sample dilutions are free of interferences.

9.3 Method blanks are prepared and analyzed in the same manner as samples, and are carried through the entire analytical procedure to show that each system is in control.

9.4 Potential cross-contamination of samples stored in lab refrigerators is monitored by preparation, analysis and evaluation of storage blanks. Storage blanks are prepared by filling 40 mL VOA vials with reagent water and placing one in each refrigerator. After one week, the storage blanks are removed and analyzed. Additional details can be found in TestAmerica Edison SOP No. ED-SPM-004, *Sample Storage & Handling Procedures for Mitigation of Sample and Laboratory Contamination,* current revision.

9.5 An initial demonstration of accuracy and precision is performed for each determinative method. This demonstration is done for each sample introduction technique.

10.0 Procedure

10.1 This section provides guidance for the analysis of low level soils using a closed system purge and trap procedure. Reference the SOPs for the determinative methods for additional details as applicable: TestAmerica Edison SOP Nos. ED-GCV-006 (*Gasoline Range Organics using GC FID Method SW846 8015, current revision*) or ED-MSV-005 (*SW846 Method 8260B Volatile Organic Compounds by Gas Chromatography/Mass Spectrometry, current revision*).

10.2 Instrument operating parameters are set at the beginning of a method of analysis and remain constant throughout the entire analytical procedure. Again, reference the SOP for the applicable determinative method for specifics. Sample operating conditions are listed here:

Example Instrument Operating Parameters

Purge and trap unit	
Purge Time	11 minutes
Dry Purge	1 Minutes
Purge Gas	Helium
Purge Flow	40-45 ml/min
Purge Temp	Water - Ambient; Solids - 40^{0}C
Trapping Temp	Ambient, <30^{0}C
Desorb Time	1-2 Minutes
Desorb Temp	Vocarb - 260^{0}C
Gas chromatograph	
Injector	180^{0}C
Carrier Gas	Helium
Carrier Flow	6 ml/min
Oven Program	35 - 250^{0}C
Run Time	30 Minutes
Mass Spectrometer	
Electron Energy	70 volts (nominal)
Mass range	35-260 AMU
Scan time	0.9 sec./scan
Source Temp	200^{0}C
Separator Temp	180^{0}C

10.3 All samples are screened prior to analysis, using the procedure outlined in TestAmerica Edison SOP No. ED-GCV-001, *Screening for Volatile Organics, Static Headspace with GC FID SW846 Method 5021(modified),* current revision. In addition to reducing excessive instrument contamination and/or extensive instrument clean up, screen data provides the analyst with an approximate dilution factor and is used to determine which sample preparation technique should be used for a particular sample. If the estimated concentration from the screening procedure shows concentrations below 200 ug/kg, follow the low concentration closed system purge and trap method. If the screen result shows concentrations >200 ug/kg, follow the medium level procedure.

10.4 Low Level Soil Procedure

10.4.1 Low level soils are field collected as described in Section 8.1 using 40 mL VOA vials with PTFE-faced septa (closed system purge and trap), En Core® Samplers, Terra Core® kits or bulk soil.

10.4.2 Set up the purge and trap autosampler (Archon 5100A or EST Centurion, see Section 6.1) for the closed system method for low-level soils. The purge and trap autosampler will add 5 mL reagent water, and 1uL of the internal standard and/or surrogate spiking solution to the sample vials. The heating block will heat each sample to 40° C and hold for 0.5 minutes before purging begins.

10.4.3 Tuning, calibration, and method blank analysis:

- Tuning and calibration is accomplished as detailed in the SOP for the applicable determinative method.

- The volume of water used when purging a calibration standard must be the same as the volume of water purged during a sample analysis. Since the low-level soils have 5 mL water added at vial preparation plus the 5 mL of reagent water added by the purge and trap autosampler prior to purging, a total volume of 10 mL is required for calibration standards.

- Internal standard (if required by the determinative method) is added to each calibration standard by the purge and trap autosampler, in the same manner it will be added to the samples.

- After successful calibration, analyze a method blank. The method blank must meet all criteria in the determinative method. A successful method blank shows that the system is free of interferences and sample analysis may begin.

10.5 Medium Level Soil Procedure

10.5.1 Medium level soils are typically field collected using laboratory prepared 40 ml VOA vials (with PTFE-faced septa) containing 25 mL of the Methanol/Surrogate solution (either the 8260 solution as described in Section 7.1.4 ***or*** the 8015 solution as described in Section 7.1.5 depending upon the method to be analyzed).

10.5.2 When preparing a medium level soil from a bulk soil sample, do not decant any supernatant liquids. Quickly mix the contents of the sample container with a narrow spatula. Weigh out 5g of soil into a 40ml vial, add 10ml of Methanol/Surrogate mix solution (either the 8260 solution as described in Section 7.1.4 ***or*** the 8015 solution as described in Section 7.1.5 depending upon the method to be analyzed) and cap the container and proceed with Section 10.5.6.

10.5.3 For samples collected in the Encore sampling device, break open one end and push the aliquot of soil out into a vial. Add 10ml of the Methanol/Surrogate mix solution (either the 8260 solution as described in Section 7.1.4 ***or*** the 8015 solution as described in Section 7.1.5 depending upon the method to be analyzed) and cap the vial and proceed with Section 10.5.6.

10.5.4 For Method 8260 samples collected as Terra Cores® in 5 mL or 10 mL methanol add 5 uL and 10 uL of the 2500 ppm 8260 Methanol/Surrogate mix (see Section 7.1.4) respectively to extract and proceed with Section 10.5.6.

10.5.5 For Method 8015 (GRO) samples collected as Terra Cores® in 5 mL or 10 mL methanol add 2.5 uL and 5 uL of the Methanol/Surrogate Mix surrogate solution (see Section 7.1.5) respectively and proceed with Section 10.5.6.

10.5.6 Cap and shake the extract for 2 minutes. Allow soil to settle.

10.5.7 Transfer 2 mL of the extract to a 2 mL amber glass vial for storage with minimal headspace.

10.5.8 Refrigerate at 4°C until the time of analysis.

10.5.9 Analyze a portion of this extract using Method 5030 purge and trap for aqueous samples (TestAmerica Edison SOP No. ED-MSV-001, *Purge and Trap for Aqueous Samples, SW846 Method 5030, current revision*). The appropriate dilution factor is calculated from the low-level analysis.

10.6 High Level Soil Procedure

10.6.1 High level soils have been preserved in the field by sampling either 10 g of soil into vials containing 25 mL Methanol/Surrogate mix solution (either the 8260 solution as described in Section 7.1.4 ***or*** the 8015 solution as described in Section 7.1.5 depending upon the method to be analyzed) or 5 g of soil into vials containing 10 mL Methanol/Surrogate mix solution (again, either the 8260 solution as described in Section 7.1.4 ***or*** the 8015 solution as described in Section 7.1.5 depending upon the method to be analyzed).

10.6.2 Screen high level soils by Method 5021 to determine the proper dilution factor prior to analysis (see TestAmerica Edison SOP No. ED-GCV-001, *Screening for Volatile Organics,Static Headspace with GC FID SW846 Method 5021(modified),* current revision.)

10.6.3 Transfer 2 ml of the extract to a 2 mL amber glass vial for storage with minimal headspace.

10.6.4 Analyze a portion of this extract using Method 5030 purge and trap for aqueous samples.

10.7 Sample Analysis

10.7.1 Remove sample vials from storage and allow thawing to ambient temperature before analysis. Shake the vial gently to ensure that the contents move freely within the vial. Place the sample vials in the autosampler tray.

10.7.2 Schedule the autosamplers to run the samples using the low level soil method. 5 mL of reagent water plus 1 uL of internal standard/ surrogate spiking solution is added to the sample vial directly through the septum.

10.7.3 Prior to purging, the sample is heated to 40°C for 0.5 minutes.

10.7.4 Purge the sample with helium for 11 minutes while agitating with magnetic stirring.

10.7.5 The purgeable compounds will flow out of the vial through a glass lined transfer line to a sorbent trap.

10.7.6 Desorb the contents of the trap on to the GC column by rapidly heating the trap to 260 °C and backflushing it with helium.

10.7.7 Begin the GC temperature program and data acquisition.

10.7.8 Re-condition the trap after desorb by baking at 260° C for 8-12 minutes.

10.7.9 Perform quantitative and qualitative analysis using TARGET software. Evaluate data according to each determinative method.

10.7.10 If a re-analysis is required, perform the re-analysis using the second frozen sample vial.

10.7.11 If a dilution is required, analyze the methanol preserved aliquot by Method 5030.

10.7.12 Technical acceptance criteria for sample analysis. (Note: these are general guidelines; please see the SOP for the determinative method for more specific acceptance criteria)

- The samples must be analyzed on a GC or GC/MS system meeting the initial calibration, continuing calibration and blank technical acceptance criteria.
- The sample must be analyzed within the required holding time.
- The sample must have an associated method blank meeting the blank technical acceptance criteria.
- The percent recovery of each of the system monitoring compounds in the sample must be within the acceptance windows.
- After analyzing a sample that exceeds the initial calibration range the analyst must either analyze an instrument blank (using the same purge inlet if using an auto sampler) which must meet technical acceptance criteria for blank analysis or monitor the sample analyzed immediately after the contaminated sample for all compounds that were in the contaminated sample that exceeded the calibration range.

10.7.13 Corrective Action for Sample Analysis (Note: these are general guidelines; please see the SOP for the determinative method for more specific corrective actions):

- Samples must meet technical acceptance criteria before reporting data.

- Corrective action for failure to meet instrument performance checks, initial, continuing calibration and method blanks must be completed prior to sample analysis.

- Corrective action for system monitoring compounds and internal standard compounds that fail to meet acceptance criteria must be completed prior to sample analysis.

- If any of the system monitoring compounds and internal standard compounds fail to meet acceptance criteria, check all calculations, instrument logs, the system monitoring compound and internal standard compound spiking solutions and the instrument operation. If the calculations were incorrect, correct calculations and verify that the system monitoring compound recoveries and internal standard compound responses meet acceptance criteria.

- Check the preparation of the internal standards and system monitoring compounds for concentration and expiration.

- Verify that the instrument is operating correctly.

- Data that fails to meet minimum acceptance criteria will be annotated (flagged) with qualifiers and/or appropriate narrative comments defining the nature of the outage. If applicable, a Corrective Action Reports will be initiated in order to provide for investigation and follow-up.

11.0 Calculations / Data Reduction

11.1 Methanol volume correction for soil moisture content (medium/high level methods):

$$Vt\ (\mu L\ solvent/water) = [\ ml\ of\ solvent + \frac{(\%moisture \times g\ of\ sample)}{100}] \times 1000\ uL/mL$$

12.0 Method Performance

12.1 Method Detection Limit Study (MDL)

The method detection limit (MDL) is the lowest concentration that can be detected for a given analytical method and sample matrix with 99% confidence that the analyte is present. The MDL is determined according to the laboratory's MDL procedure in Section 20 (Test Methods and Method Validation) of TestAmerica Edison's Quality Assurance Manual (ED-QA-LQM). MDLs reflect a calculated (statistical) value determined under ideal laboratory conditions in a clean matrix, and may not be achievable in all environmental matrices. The laboratory maintains MDL studies for analyses performed; these are verified at least annually unless method requirements require a greater frequency.

12.2 Demonstration of Capabilities

For DOC procedure refer to Section 19 in the most current revision of TestAmerica Edison's Quality Assurance Manual (ED-QA-LQM).

12.3 Training Requirements

Refer to TestAmerica SOP No. ED-GEN-022, *Training* (latest revision) for the laboratory's training program.

13.0 Pollution Control

13.1 It is TestAmerica's policy to evaluate each method and look for opportunities to minimize waste generated (i.e., examine recycling options, ordering chemicals based on quantity needed, preparation of reagents based on anticipated usage and reagent stability). Employees must abide by the policies in Section 13 of the Corporate Environmental Health and Safety Manual (CW-E-M-001) for "Waste Management and Pollution Prevention."

14.0 Waste Management

14.1 Waste management practices are conducted consistent with all applicable rules and regulations. Excess reagents, samples and method process wastes are disposed of in an accepted manner. Waste description rules and land disposal restrictions are followed. Waste disposal procedures are incorporated by reference to TestAmerica Edison SOPs Nos. ED-SPM-007 (*Disposal of Samples and Associated Laboratory Waste, current revision)* and ED-SPM-008 (*Laboratory Waste Disposal Procedures, current revision).* The following waste streams are produced when this method is carried out.

- Laboratory Generated Aqueous Waste (aqueous VOA vials – used and unused). This waste may have a pH of less than 2.0. These vials are collected in satellite accumulation. The vials are then transferred to the waste room. These vials are passed through a vial crusher and the liquid portion is separated from the solid portion. The solid is dumped into the municipal garbage. The liquid is pumped into the neutralization system where it is neutralized to a pH of 6 to 9 with sodium bicarbonate (Seidler Chemical SC-0219-25). When neutralization is complete, the material is transferred to the municipal sewer system**.**

- Expired Standards – The vials are collected in a 1 gallon polyethylene bucket. These vials are then transferred to an open top 55 gallon steel or polyethylene waste drum. These drums are transported to a waste facility for proper disposal.

- Soil Retain Samples - These samples if not flagged in the system for any hazardous constituents are transferred to poly-lined cubic yard boxes. These

boxes when full are sent to stabilization or incineration. These materials are sent out as hazardous for lead and chromium

Teris Profile Number (incineration): 50016710
Onyx Profile Number: (stabilization) 402535

- Methanol Preserved Samples - Methanol preserved sample vials are collected in satellite accumulation and then transferred to a 55 gallon open top steel waste drum in the waste room. This drum is then removed by a waste vendor for incineration.

  Teris Profile Number: 50016652
  Onyx Profile Number: 282493

- Returned Methanol Preservative - Methanol preserved vials are collected in satellite accumulation and then transferred to 55 gallon open top steel waste drums in the waste room. These waste drums are then removed by a waste vendor for incineration.

  Teris Profile Number: 50016652
  Onyx Profile Number: 282493

15.0 References / Cross-References

15.1 United States Environmental Protection Agency, *Method 5035A: Closed System Purge and Trap and Extraction for Volatile Organics in Soil and Waste Samples*, Test Methods for Evaluating Solid Wastes, SW846 Third Edition, Volume 1B: Laboratory Manual, Physical/Chemical Methods, Revision 0, December 1996.

15.2 TestAmerica Edison Document No. ED-QA-LQM, *Laboratory Quality Manual*, most current revision.

15.3 TestAmerica Edison SOP No. ED-GCV-001, *Screening for Volatile Organics,Static Headspace with GC FID SW846 Method 5021(modified),* current revision.

15.4 TestAmerica Edison SOP No. ED-MSV-001, *Purge and Trap for Aqueous Samples, SW846 Method 5030, current revision*.

15.5 TestAmerica Edison SOP Nos. ED-GCV-006, *Gasoline Range Organics using GC FID Method SW846 8015, current revision.*

15.6 TestAmerica Edison SOP No ED-MSV-005, *SW846 Method 8260B Volatile Organic Compounds by Gas Chromatography/Mass Spectrometry, current revision.*

15.7 United States Environmental Protection Agency, *Method 8000C: Determinative Chromatographic Determinations*, Test Methods for Evaluating Solid Wastes, SW846 Third Edition, Volume 1B: Laboratory Manual, Physical/Chemical Methods, Revision 3, March 2003

15.8 TestAmerica Edison SOP No. ED-SPM-004, *Sample Storage & Handling Procedures for Mitigation of Sample and Laboratory Contamination,* current revision.

15.9 TestAmerica Corporate Environmental Health and Safety Manual (CW-E-M-001)

15.10 TestAmerica SOP No. ED-GEN-022, *Training* ,latest revision.

15.11 TestAmerica Edison SOPs Nos. ED-SPM-007, *Disposal of Samples and Associated Laboratory Waste, current revision.*

15.12 TestAmerica Edison SOP No. ED-SPM-008, *Laboratory Waste Disposal Procedures,* current revision.

16.0 Method Modifications:

16.1 N/A

17.0 Attachments

17.1 N/A

18.0 Revision History

- Revision 10, effective date: 4/11/2019
  - Section 8.2.1: Added temperature preservation requirement for Medium level extracts.

- Revision 9, effective date: 11/27/2012
  - Updated referenced Lab Quality Manual section and appendix numbers as appropriate

- Revision 8, effective date: 12/6/2010
  - Section 3: revised to reference new location for definitions.
  - Sections 8.2.1.4 and 8.3.9 referencing methanol solvent adjustment for solid samples with significant moisture content (>10%) was removed.

- Revision 7, effective date: 11/4/2008:
  - Revised SOP format in accordance with TestAmerica Corporate Quality SOP No. CW-QS-002 (Rev 0) Writing a Standard Operating Procedure (SOP).
  - Updated the SOP to comply with SW846 Method 5035A (was 5035). Retitled SOP accordingly.
  - Section 1.1.5: Added reference to TestAmerica Edison SOP No. ED-MSV-001(*Purge and Trap for Aqueous Samples, SW846 Method 5030, current revision*).
  - Section 1.1.6: Added references to TestAmerica Edison SOP Nos. ED-GCV-006 (*Gasoline Range Organics using GC FID Method SW846 8015, current*

revision) and ED-MSV-005 (*SW846 Method 8260B Volatile Organic Compounds by Gas Chromatography/Mass Spectrometry, current revision*)

- Section 1.1.7: Added reference to Quality Assurance Manual for method modifications.
- Section 3: revised to reference new location for definitions.
- Section 4: revised to include a reference to storage blanks and the applicable SOP: TestAmerica Edison SOP No. ED-SPM-004, *Sample Storage & Handling Procedures for Mitigation of Sample and Laboratory Contamination*, current revision.
- Section 5 (Safety): Revised to include most up to date corporate health and safety references and information.
- Section 6: Updated with current instrumentation and configurations.
- Section 7.0: added details of the solvent testing and approval program.
- Section 7.2: Updated standards sources and catalog numbers.
- Section 8: Updated with additional details for each type of preservation/analysis (low level, medium level, high level). Added used of Terra Core® Sampling Kits (Section 8.1.3).
- Section 8: Updated to include requirement to adjust the methanol volume (medium/high level) for soil moisture content.
- Section 9.0: Added details of storage blanks and reference to TestAmerica Edison SOP No. ED-SPM-004, *Sample Storage & Handling Procedures for Mitigation of Sample and Laboratory Contamination,* current revision..
- Section 10: Revised and expanded to include instrument operating conditions and specific sample prep details for low level, medium and high level preps.
- Section 11: Deleted reference to SOP for Organic Calculations. Added statement that calculations can now be found in the applicable determinative methods.
- Section12.0: added reference to Training SOP.
- Section 13.0: Revised to include most up to date company policy on Pollution Control as well as to include corporate health and safety references and information.
- Section 14.0: Revised to include most up to date company policy on Waste Management as well as to include corporate health and safety references and information.
- References: Expanded to include more specific SOP references.
- Revision history: updated.

Title: Volatile Organic Compounds by Gas Chromatography/Mass Spectrometry (GC/MS) by SW846 Method 8260D

Once printed, this is considered an uncontrolled document

Approvals (Signature/Date):

Sylvanus Klusey Organics Operations Manager	10/17/22 Date	Dan Helfrich Health & Safety Manager / Coordinator	10/17/22 Date
Carl Armbruster Quality Assurance Manager	10/17/22 Date	Mark Acierno Laboratory Director	10/17/22 Date

1.0 Scope and Application

1.1 Analytes, Matrix(s), and Reporting Limits

1.1.1 USEPA SW846 Method 8260D is used for the determination of volatile organic compounds in a variety of aqueous and solid matrices by purge and trap gas chromatography (GC)/mass spectrometery (MS). The methods are applicable to the compounds listed in Table 1 (below). Actual target compound lists are determined through regulatory or project specifications. Method performance criteria for each target analyte will be determined prior to sample analysis.

1.1.2 This SOP also describes the optional procedure for analyses of compounds using 8260D Selected Ion Monitoring (SIM). SIM analyses is specific to target compounds: 1,2-dibromoethane, 1,2-dibromo-3-chloropropane, 1,2,3-Trichloropropane and 1,4-Dioxane. Benzene and Chloroform if needed.

Table 1: Method Analytes

Compound	CAS #	Compound	CAS #
1,1,1,2-Tetrachloroethane	630-20-6	cis-1,2-Dichloroethene	156-59-2
1,1,1-Trichloroethane	71-55-6	cis-1,3-Dichloropropene	10061-01-5
1,1,1-Trifluoro-2,2-dichloroethane	306-83-2	Cyclohexane	110-82-7
1,1,2,2-Tetrachloroethane	79-34-5	Cyclopentene	142-29-0
1,1,2-Trichloro-1,2,2-trifluoroethane	76-13-1	Dibromomethane	74-95-3
1,1,2-Trichloroethane	79-00-5	Dichlorobromomethane	75-27-4
1,1-Dichloroethane	75-34-3	Dichlorodifluoromethane	75-71-8
1,1-Dichloroethene	75-35-4	Dichlorofluoromethane	75-43-4
1,1-Dichloropropene	563-58-6	Dimethylnaphthalene (total)	28804-88-8
1,1-Difluoroethane	75-37-6	Epichlorohydrin	106-89-8
1,2,3-Trichlorobenzene	87-61-6	Ethanol	64-17-5
1,2,3-Trichloropropane **(1)**	96-18-4	Ethyl acetate	141-78-6
1,2,3-Trimethylbenzene	526-73-8	Ethyl acrylate	140-88-5
1,2,4,5-Tetramethylbenzene	95-93-2	Ethyl ether	60-29-7
1,2,4-Trichlorobenzene	120-82-1	Ethyl methacrylate	97-63-2
1,2,4-Trimethylbenzene	95-63-6	Ethylbenzene	100-41-4
1,2-Dibromo-3-Chloropropane **(1)**	96-12-8	Ethylene Dibromide **(1)**	106-93-4
1,2-Dichloro-1,1,2,2-tetrafluoroethane	76-14-2	Hexachlorobutadiene	87-68-3
1,2-Dichloro-1,1,2-trifluoroethane	354-23-4	Hexane	110-54-3
1,2-Dichlorobenzene	95-50-1	Indan	496-11-7
1,2-Dichloroethane	107-06-2	Iodomethane	74-88-4
1,2-Dichloroethene, Total	540-59-0	Isobutyl alcohol	78-83-1
1,2-Dichloropropane	78-87-5	Isopropyl acetate	108-21-4
1,3,5-Trichlorobenzene	108-70-3	Isopropyl alcohol	67-63-0
1,3,5-Trimethylbenzene	108-67-8	Isopropyl ether	108-20-3
1,3-Dichlorobenzene	541-73-1	Isopropylbenzene	98-82-8
1,3-Dichloropropane	142-28-9	Methacrylonitrile	126-98-7
1,3-Dichloropropene, Total	542-75-6	Methyl acetate	79-20-9

Compound	CAS #	Compound	CAS #
1,4-Dichlorobenzene	106-46-7	Methyl acrylate	96-33-3
1,4-Dioxane **(1)**	123-91-1	Methyl methacrylate	80-62-6
1-Chloropropane	540-54-5	Methyl tert-butyl ether	1634-04-4
2,2,4-Trimethylpentane	540-84-1	Methylcyclohexane	108-87-2
2,2-Dichloropropane	594-20-7	Methylene Chloride	75-09-2
2,4,4-Trimethyl-1-pentene	107-39-1	Methylnaphthalene (total)	1321-94-4
2-Butanone (MEK)	78-93-3	Monochloropentafluoroethane	76-15-3
2-Chloro-1,3-butadiene	126-99-8	m-Xylene & p-Xylene	179601-23-1
2-Chloroethyl vinyl ether	110-75-8	Naphthalene	91-20-3
2-Chloropropane	75-29-6	n-Butanol	71-36-3
2-Chlorotoluene	95-49-8	n-Butyl acetate	123-86-4
2-Hexanone	591-78-6	n-Butyl acrylate	141-32-2
2-Methyl-1,3-butadiene	78-79-5	n-Butylbenzene	104-51-8
2-Methyl-2-propanol	75-65-0	n-Heptane	142-82-5
2-Nitropropane	79-46-9	n-Propyl acetate	109-60-4
2-Octanol	123-96-6	N-Propylbenzene	103-65-1
2-Octanone	111-13-7	o-Xylene	95-47-6
4-Chlorotoluene	106-43-4	p-Diethylbenzene	105-05-5
4-Ethyltoluene	622-96-8	Pentane	109-66-0
4-Isopropyltoluene	99-87-6	Propene	115-07-1
4-Methyl-2-pentanone (MIBK)	108-10-1	Propionitrile	107-12-0
Acetaldehyde	75-07-0	sec-Butylbenzene	135-98-8
Acetone	67-64-1	Styrene	100-42-5
Acetonitrile	75-05-8	Tert-amyl methyl ether	994-05-8
Acrolein	107-02-8	Tert-butyl ethyl ether	637-92-3
Acrylonitrile	107-13-1	tert-Butylbenzene	98-06-6
Allyl chloride	107-05-1	Tetrachloroethene	127-18-4
Amyl acetate (mixed isomers)	628-63-7	Tetrahydrofuran	109-99-9
Benzene **(1)**	71-43-2	Toluene	108-88-3
Benzyl chloride	100-44-7	Total BTEX	STL00431
Bromobenzene	108-86-1	trans-1,2-Dichloroethene	156-60-5
Bromoform	75-25-2	trans-1,3-Dichloropropene	10061-02-6
Bromomethane	74-83-9	trans-1,4-Dichloro-2-butene	110-57-6
Butadiene	106-99-0	Trichloroethene	79-01-6
Butyl Methacrylate	97-88-1	Trichlorofluoromethane	75-69-4
Camphene	79-92-5	Vinyl acetate	108-05-4
Camphor	76-22-2	Vinyl chloride	75-01-4
Carbon disulfide	75-15-0	Xylenes, Total	1330-20-7
Carbon tetrachloride	56-23-5	1,4-Dichlorobenzene-d4 **(ISTD)**	3855-82-1
Chlorobenzene	108-90-7	1,4-Dioxane-d8 **(ISTD)**	17647-74-4
Chlorobromomethane	74-97-5	2-Butanone-d5 **(ISTD)**	24313-50-6
Chlorodibromomethane	124-48-1	Chlorobenzene-d5 **(ISTD)**	3114-55-4
Chlorodifluoromethane	75-45-6	Fluorobenzene **(ISTD)**	462-06-6
Chloroethane	75-00-3	TBA-d9 **(ISTD)**	25725-11-5
Chloroform **(1)**	67-66-3	1,2-Dichloroethane-d4 **(Surrogate)**	17060-07-0
Chloromethane	74-87-3	4-Bromofluorobenzene **(Surrogate)**	460-00-4

Compound	CAS #	Compound	CAS #
Chlorotrifluoroethene	79-38-9	Dibromofluoromethane **(Surrogate)**	1868-53-7
Chlorotrifluoromethane	75-72-9	Toluene-d8 **(Surrogate)**	2037-26-5

(1) Compound can be analyzed by full scan or Selected Ion Monitoring (SIM).

1.1.3 Method 8260D can be used to quantitate most volatile organic compounds that have boiling points below 200°C, and that are insoluble or slightly soluble in water. Water-soluble compounds can be included in this method, but quantitation limits will be higher due to poor purging efficiency.

1.1.4 The standard reporting limit (RL) is established at or above the low-level standard in the calibration curve (1 ug/l for most compounds). For a complete list of method detection limits (MDLs) and RLs, please see reference the current TALS (LIMS) active Method Limit Group database.

1.1.5 On occasion clients may request modifications to this SOP. These modifications are handled following the procedures outlined in Sections 7 (*Review of Work Request*) and 20 (*Test Methods and Method Validation*) of Eurofins Edison's Quality Assurance Manual (ED-QA-LQM).

1.1.6 Any variation in procedure shall be completely documented using an NCM. The NCM is approved by the supervisor and then automatically sent to the laboratory Project Manager by e-mail so that the client can be notified as appropriate. The QA department also receives NCMs by e-mail for tracking and trending purposes. The NCM process is described in more detail in Eurofins Edison SOP No. ED-GEN-003, *Standard Operating Procedure for Control of Non-Conformances and Corrective Action,* current revision NCM shall be filed in the project file and addressed in the case narrative. Any unauthorized deviations from this procedure must also be documented as a nonconformance, with a cause and corrective action described.

2.0 Summary of Method

2.1 Method 8260D is used to determine volatile organic compounds in aqueous, non-aqueous and solid matrices. Sample preparation techniques vary, depending on the matrix and the level of contamination expected. Purge and trap techniques are used to introduce the sample to the GC/MS system. Refer to Eurofins Edison SOP Nos. ED-MSV-001*, Purge and Trap for Aqueous Samples, SW846 Method 5030*, current revision and ED-MSV-002, *Closed-System Purge-and-Trap and Extraction for Volatile Organics in Soil and Waste Samples, SW846 Method 5035A,* current revision.

2.2 All samples extracts are screened by GC/FID static headspace analysis to provide the analyst with appropriate initial dilution factors. For additional details see

Eurofins Edison SOP No. ED-GCV-001, *Screening for Volatile Organics, Static Headspace with GC FID, SW846 Method 5021*, current revision.

2.3 An aliquot of sample containing internal standard and surrogate spiking solution is purged with nitrogen in a closed sparging vessel. The volatile compounds are transferred from the aqueous phase to the vapor phase. The vapor is swept through a sorbent column where the volatiles are trapped. After purging is complete, the sorbent column is heated and backflushed with helium to desorb the volatiles onto a gas chromatograph column.

2.4 Analytes eluted from the capillary chromatograhy column are introduced into the mass spectrometer via a direct connection. Identification of target analytes is accomplished by comparing their mass spectra with the electron impact spectra of authentic standards. Quantitation is accomplished by comparing the response of a major (quantitation) ion relative to an internal standard using a minimum of a five-point calibration curve.

2.5 For aqueous VOA samples submitted for New Jersey Groundwater Quality Standard (NJ GWQS) evaluation, a full scan analysis is initially performed using the 8260 methodology No further analysis by SIM is required if all of the following compounds are present above the full scan RL: 1,2-dibromoethane, 1,2-dibromo-3-chloropropane, 1,2,3-Trichloropropane and 1,4-dioxane, chloroform, vinyl chloride and benzene. If any of these compounds are undetected in the undiluted, full scan analysis, the sample must be analyzed via 8260D SIM for those compounds.

2.6 In order to meet lower reporting limits of 0.5ug/L for most analytes, 2.5 ug/L for ketones and generally lower limits for other non-routine analytical compounds, samples must be analyzed against an initial calibration with a low point at those levels. The corresponding TALS login method for low level aqueous analysis is 8260_LL. See Table 3b for initial calibration levels and spike amounts.

3.0 Definitions

3.1 For a complete list of definitions refer to Appendix 2 in the most current revision of Eurofins Edison's Quality Assurance Manual (ED-QA-LQM).

4.0 Interferences

4.1 This method is susceptible to contamination from a number of sources, including organic solvents used in other laboratory procedures, impurities in the purge gas, improper cleaning of syringes or purge vessels, and carryover from high level samples. Samples can be contaminated by the diffusion of volatile organics through the septum during shipment or storage. Steps have been taken to ensure that these potential problems are eliminated from the laboratory.

4.2 The volatiles analytical laboratory is housed in a separate building, away from the organic extraction lab area where large quantities of organic solvents are used. No organic solvents are used or stored in the volatiles laboratory.

4.3 The nitrogen used as purge gas passes through a solvent trap prior to its inlet into the purge and trap units.

4.4 Trip Blanks are shipped to clients with aqueous bottle ware as requested. The purpose of the trip blank is to detect and identify any VOC contamination of the samples while in transit to and from the lab. The blank is created at the laboratory by completely filling the volatile vial container with lab grade organic free deionized water and sealing the container Trip Blanks accompany bottle ware and samples through the sampling, storage and analysis stages as a check on contamination that may occur at these points.

4.5 Individual samples are each handled with a unique syringe that has been baked in a drying oven at 105°C to ensure the absence of volatile compounds.

4.6 Carryover can occur anytime a high level sample is analyzed. Screening procedures are employed to ensure that a sample is analyzed at an appropriate dilution to minimize potential carryover. When a high level sample is analyzed, it is followed by the analysis of a reagent water blank. If another sample was analyzed after the high level sample, this sample is inspected carefully for signs of carryover. If this sample does not contain any of the compounds found in the high level sample, the system can be considered contamination free.

4.7 The analytical system is checked daily with the analysis of a method blank. This blank must meet all quality control criteria for the method before sample analysis may take place.

5.0 Safety

Employees must abide by the policies and procedures in the Corporate Environmental Health and Safety Manual (CW-E-M-001) and this document. This procedure may involve hazardous material, operations and equipment. This SOP does not purport to address all of the safety problems associated with its use. It is the responsibility of the user of the method to follow appropriate safety, waste disposal and health practices under the assumption that all samples and reagents are potentially hazardous. Safety glasses, gloves, lab coats and closed-toe, nonabsorbent shoes are a minimum.

Any questions pertaining to safety issues or procedures should be brought to the department manager or Edison Safety Officer.

5.1 Specific Safety Concerns or Requirements

5.1.1 Latex, nitrile and vinyl gloves all provide adequate protection against the methanol used in this method.

5.1.2 Purge vessels on purge-and-trap instruments can be pressurized by the time analysis is completed. Vent the pressure prior to removal of these vessels to prevent the contents from spraying out.

5.1.3 The gas chromatograph and mass spectrometer contain zones that have elevated temperatures. The analyst needs to be aware of the locations of those zones, and must cool them to room temperature prior to working on them.

5.1.4 The mass spectrometer is under deep vacuum. The mass spectrometer must be brought to atmospheric pressure prior to working on the source.

5.1.5 There are areas of high voltage in both the gas chromatograph and the mass spectrometer. Depending on the type of work involved, either turn the power to the instrument off, or disconnect it from its source of power.

5.2 Primary Materials Used

The following is a list of the materials used in this method, which have a serious or significant hazard rating. **Note: This list does not include all materials used in the method. The table contains a summary of the primary hazards listed in the MSDS for each of the materials listed in the table.** A complete list of materials used in the method can be found in the reagents and materials section. Employees must review the information in the MSDS for each material before using it for the first time or when there are major changes to the MSDS.

Material (1)	Hazards	Exposure Limit (2)	Signs and symptoms of exposure
Methanol (MeOH)	Flammable Poison Irritant	200 ppm-TWA	A slight irritant to the mucous membranes. Toxic effects exerted upon nervous system, particularly the optic nerve. Symptoms of overexposure may include headache, drowsiness and dizziness. Methyl alcohol is a defatting agent and may cause skin to become dry and cracked. Skin absorption can occur; symptoms may parallel inhalation exposure. Irritant to the eyes.
1 – Always add acid to water to prevent violent reactions.			
2 – Exposure limit refers to the OSHA regulatory exposure limit.			

6.0 Equipment and Supplies

6.1 Instrumentation

6.1.1 Purge and trap units from several different manufacturers are used, depending upon the sample matrix and preparatory technique required. A purge and trap unit consists of three parts: the sample purge unit, the trap, and the concentrator. Unit configurations currently in use are:

- OI Analytical 4551, 4100 Automatic Sampler/4660,4760 concentrator;
- Archon 5100A Automatic sampler/ OI Analytical 4660,4760 concentrator;
- EST Centurion Autosampler/ EST Encon concentrator;

- Archon Autosampler/EST Encon concentrator.
- Archon/EST Evolution

6.1.2 A VOCARB 3000 trap from Supelco is used in the Encon concentrator. The trap is 25cm long with an inside diameter of 0.105 inches. The trap is packed with 10.0cm Carbopack B, 6.0 cm Carboxin 1000, and 1cm Carboxin 1001.

6.1.3 An OI analytical purge trap #10 is used for the OI 4560,4660 and 4760 concentrator. The trap is 25cm long with an inside diameter of 0.105 inches. The trap is packed to contain the following absorbents: Tenax/silica gel/carbon molecular sieve.

6.1.4 Alternate traps may be used provided the adsorption and desorption characteristics are equivalent to those of the trap recommended by the method.

6.1.5 Both the Encon and OI concentrators are capable of rapidly heating the trap to 260°C and holding at that temperature for the duration of the desorb time.

6.1.6 Gas chromatograph: HP Agilent 6890/7890 equipped with temperature programming capability.

6.1.7 GC column: 30M long x 0.25mm ID, 1.4um film thickness, 20M x 0.18mm x 1um DB-624 and 20M long x 0.18 mm ID Restek Rtx-VMS capillary column with 1um film thickness or similar phase.

6.1.8 Mass Spectrometer (Agilent 5973/5975/5977): scanning from 35-260 amu every 0.9 seconds, utilizing 70 volts (nominal) electron energy in the electron ionization mode and producing a mass spectrum which meets all EPA performance criteria when 50 ng of 4-Bromofluorobenzene (BFB) is injected through the gas chromatograph inlet.

6.1.9 GC/MS Interface: transfer lines heated to 180°C .

6.1.10 Data system: HP Chemstation II for data acquisition and Eurofins Chrom for data processing.

6.2 Supplies

- Microsyringes: 10 ul to 1000 ul.
- Syringes: 5 ml to 25 ml gas-tight.
- Injection port liners: HP 18740-80200 or equivalent
- Volumetric flasks: Class “A” glassware, 5 ml to 500 ml.

- VOA vials: 20-ml and 40-ml glass with PTFE – faced septum.
- Vials: 2-ml amber glass with screw cap with Teflon-faced septa.
- Top loading analytical balance.
- Spatula: Narrow, stainless steel.
- Stir bars: PTFE coated, small enough to spin freely inside a VOA vial.

7.0 Reagents and Standards

7.1 Reagents

7.1.1 Organic free reagent water: High purity water that meets the requirements for a method blank when analyzed. (See section 9.1.1) Reagent water is obtained from Millipore system. Other methods of preparing reagent water are acceptable, provided that the water produced meets method blank criteria.

7.1.2 Methanol: Ultra Resi-Analyzed, purge and trap grade, purchased from JT Baker or equivalent. (Cat # 9077-02)

7.1.2.1 Each lot of methanol is screened for contaminants before being used for analysis as detailed in Eurofins Corporate Quality SOP No. CA-Q-S-001 (*Solvent & Acid Lot Testing & Approval*) and Eurofins Edison SOP No. ED-GEN-023 (*Bulk Solvent Testing and Approval*).

7.2 Standards

7.2.1 **Calibration Standards** Stock target compound analytical standard solutions are purchased mainly from Restek, Supelco, Inc, Absolute Standards and Spex although standards of similar quality from other suppliers may be substituted as required. Standards noted with an asterisk (*) are custom mixes made especially for Eurofins Edison.

Target Analyte Standard Name	Concentration	Vendor	Catalog #
8260 List 1 / Std #3 Gases*	2500 ppm	Restek	569722
8260 List 1 / Std #3 Gases – (SS)*	2500 ppm	Restek	569722 sec
8260 List 1 / Std #1 MegaMix*	1250-62500 ppm	Restek	569720
8260 List 1 / Std #1 MegaMix (SS)*	1250-62500 ppm	Restek	569720 sec
8260 List 1 / Std #2 Ketones *	12500 ppm	Restek	569721
8260 List 1 / Std #2 Ketones * (SS)	12500 ppm	Restek	569721 sec
8260 List 1 / Std #5 Acrolein *	20,000 ppm	Restek	568720
8260 List 1 / Std #5 Acrolein (SS)	20,000 ppm	Restek	568720 sec
8260 List 1 /Std #4 2 CEVE *	2500 ppm	Restek	569723
8260 List 1 /Std #4 2 CEVE (SS) *	2500 ppm	Restek	569723 sec
8260 List 1 /Std #6 Vinyl Acetate *	5000 ppm	Restek	569724
8260 List 1 /Std #6 Vinyl Acetate (SS) *	5000 ppm	Restek	569724 sec

Target Analyte Standard Name	Concentration	Vendor	Catalog #
8260 List 2 / Std #1 Additions *	2500-62500ppm	Restek	568725
8260 List 2 / Std #1 Additions (SS) *	2500-62500 ppm	Restek	568725 sec
8260 List 3 / Std #1 Polar Additions *	2500-100000ppm	Restek	568728
8260 List 3 / Std #1 Polar Additions (SS) *	2500-100000 ppm	Restek	568728 sec
VOC Extra Standard 2015 *	2500-5000 ppm	Absolute	98593
VOC Extra Standard 2015 * (SS)	2500-5000 ppm	Absolute	98593
Epichlorohydrin	1000 ppm	Absolute	70377
Acrolein	5000 ppm	Restek	91980
Acrolein *	Neat	Sigma	110221
2-Freon Mix quote # 12258 *	2500ppm	Absolute	12258
2-Freon Mix quote # 12258 * (SS)	2500ppm	Absolute	12258
1,4-Dioxane	Neat	Sigma	360481
Epichlorohydrin	Neat	Sigma	45340
2-Chloroethylvinyl ether	Neat	Sigma	109983
1,4-Dioxane	1000 ppm	Absolute	70373
1,4-Dioxane	10000 ppm	Absolute	92785
Benzene	1000 ppm	Absolute	70025
Chloroform	1000 ppm	Absolute	70076

(1): The separate source for this material is not available as a distinct catalog number. Analyst must ensure that a separate lot of the material is selected and used as required.

An asterisk (*) indicates a custom standard mix.

7.2.1.1. Prepare stock solutions at volumes and concentrations indicated in Table 2 (Working Standards Preparation) by combining the indicated volumes of each stock solution into a volumetric flask corresponding to the total final volume. Dilute to the volume marker with methanol.

7.2.1.2. Prepare individual calibration standards as applicable per Section 9.2.2.1, Table 3, Initial Calibration Standards Preparation, Low Level Soil, Table 3a, Initial Calibration Standards Preparation (Low Level), Aqueous or Table 3B Initial Calibration Standards Preparation, Aqueous.

7.2.1.3. The 'Second Source' standards listed are used in the preparation of the Initial Calibration Verification (ICV) standard (see Tables 4 and 4a for ICV preparation instructions) and the Laboratory Control Standard (LCS) (see Section 9.1.3 and Tables 4 and 4a).

7.2.2 **Surrogate Standards:** Surrogate standard solutions are prepared from the stock solution (2500ppm)

Surrogate Standard Name	Concentration	Vendor	Catalog #
4-Bromofluorobenzene	2500ppm	Restek	567650
Toluene-d8			
1,2-Dichloroethane-d4			
Dibromofluoromethane			

7.2.2.1 A primary surrogate stock solution (2500 ppm each) is prepared from the neat standards as follows:

7.2.2.2 Secondary surrogate standard solutions are prepared at two (2) levels using the 2500 ppm primary stock solution as detailed in the table below:

Standard Name	Vendor	Catalog #	Volume added	Concentration of Stock Std.	Concentration of Standard	Total Volume Volume in MeOH/Total volume of MeOH
8260 Surrogate Mix: 4-Bromofluorobenzene Toluene-d8 1,2-Dichloroethane-d4 Dibromofluoromethane	Restek	567650	1ml	2500ppm	250ppm	10mL 9.0mL TV/M
8260 Surrogate Mix: 4-Bromofluorobenzene Toluene-d8 1,2-Dichloroethane-d4 Dibromofluoromethane	Restek	567650	1ml	2500ppm	50ppm	50mL 9.0mL TV/M

7.2.2.3 Methanol/Surrogate solution (2.5ug/mL): For methanol sampling field kits. Prepared by adding 1mL of 2500 ug/ml primary surrogate stock solution (see Section 7.2.2.1) to 1 L purge and trap grade methanol.

7.2.3 **Internal Standards:** Internal Standards Solutions are purchased from Restek:

Standard Name	Concentration	Vendor	Catalog #
8260 Internal Standard Mix: *Chlorobenzene-d5 *1,4-Dichlorobenzene-d4 *Fluorobenzene *1,4-Dioxane-d8 *TBA-d9	250-5000ppm	Restek	567649

7.2.4 **Internal Standard/Surrogate Mix (125 ppm each):** A solution containing both Internal Standards and Surrogates at 125 ppm is prepared in a 10ml volumetric flask as detailed below using the 2500 ppm surrogate stock solution prepared in Section 7.2.2.1 and the 2500 ppm internal standard mix detailed in Section 7.2.3:

Standard Name	Concentration of Stock Std.	Volume added to final volume of 20ml MeOH	Final Concentration of Standard
8260 Internal Standard/Surrogate Mix (125 ppm) For Aquatek Autosampler	2500 ppm Surrogate Mix	1.0ml	125 ppm each component
	250 Internal Std Mix	10 ml	

7.2.5 **Internal Standard/Surrogate Mix (SIM) (2.5/50 ppm each):** A solution containing both Internal Standards and Surrogates at 25 ppm is prepared in a 10ml volumetric flask as detailed below using the 2500 ppm surrogate stock solution prepared in Section 7.2.2.1 and the 250 ppm internal standard mix detailed in Section 7.2.3:

Standard Name	Concentration of Stock Std.	Volume added to final volume of 10ml MeOH	Final Concentration of Standard
8260 Internal Standard/Surrogate Mix (25 ppm) (SIM)	2500 ppm Surrogate Mix	10ul	2.5/50 ppm each component
	250 Internal Std Mix (Restek)	10ul	
1,4-Dioxane-d8	10000 ppm	50ul	

7.2.6 **GC/MS Instrument Performance Check (BFB):** The instrument performance check solution consists of 4-Bromofluorobenzene in addition to the other three surrogates in methanol. Prepare the solution at **50ppm as specified in section 7.2.2.2**. Assign an expiration date of 6 months.

7.2.7 All standards preparation information must be logged into the TALS Reagent Module. All pertinent information must be entered: Date prepared, Lot #'s, Expiration dates, Solvents used, Lab Lot # (expiration date), Manufacturer and Verification signature. Additionally, all prepped standards are typically given a unique Lot# and all information pertaining to standard preparation is entered into the GC/MS VOA Standard Preparation Log Book. Information such as standard supplier, lot number, original concentration, a description of how the standard was made, are required along with the laboratory lot number, analyst's initials, date prepared, expiration date and verification signature. Class "A" volumetric must be used at all times and syringes, preferably gas-tight syringes when available, should be checked for accuracy using an analytical balance. Class "A" pipettes should also be used if volumes permit.

7.2.8 Please refer to Eurofins Edison SOP No. ED-GEN-008, *Standard Operating Procedure for Preparation, Purity and Storage of Reagents and Standards*, current revision. For Method 8260D:

- Shelf Life of Standard:
  - Stock standards (Non-gases) - 6 months after opening vendor stock of up to 2000ppm, 3 years for 10,000ppm, 5 years for over 50,000ppm, or manufacturer's expiration date whichever comes first.
  - Stock Standards (Gases) - 2 months after opening vendor stock, or manufacturer's expiration date whichever comes first.
  - Working/Secondary dilution Standards (Non-gases) – 6 months after preparation, or manufacturer's expiration date whichever comes first.
  - Working/Secondary dilution Standards (Gases) – 1 week from the date of preparation for 50ppm and 2 weeks for 500ppm, or manufacturer's expiration date whichever comes first.
  - Daily Calibration Standards – 24 hours after preparation.

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- Storage Requirements:

  Aqueous standards are stored at 4°C and Methanol standards are stored at -10°C to -20°C.

8.0 Sample Collection, Preservation, Shipment and Storage

Sample container, preservation techniques and holding times may vary and are dependent on sample matrix, method of choice, regulatory compliance, and/or specific contract or client requests. Listed below are the holding times and the references that include preservation requirements.

Matrix	Sample Container	Min. Sample Size	Preservation	Holding Time	Reference
Waters	Glass 40 ml vials	40 mLs	HCl, pH < 2; Cool 4 °C ± 2°C	14 Days / preserved 7 Days / unpreserved	SW846 Method 5030
Waters	Glass 40 ml vials	40mLs	TSP,pH>11 Cool 4 °C ± 2°C	14 Days / preserved	SW846 Method 5030
Soils (Low)	Encore or Terracore (40 ml vials)	5 grams in 5 mls DI H_2O	Frozen Stored -7°C to -20°C	14 Days	SW846 Method 5035A

Matrix	Sample Container	Min. Sample Size	Preservation	Holding Time	Reference
Soils (Med)	Encore or Terracore (40 ml vials)	5 grams in 10 mls MeOH	Cool 4 °C ± 2°C	14 Days	SW846 Method 5030
Soils (High)	Glass (Lab Prepared Kits)	10 grams in 25 mls MeOH	Cool 4 °C ± 2°C	14 Days	SW846 Method 5030

8.1.1 There are several methods of sampling soil. The recommended method is to take samples using an EnCore™ sampler or using a Terra Core™ sampling kit. At specific client request, unpreserved soil samples in 4oz jars may be accepted. For EnCore and Terra Core sampling, a separate jar is required for percent solids/moisture determination, unless one is supplied for another analysis.

8.1.2 For EnCore™ samplers, the 5g sample is extruded into a pre-weighed 40mL vial containing 5mL of methanol (medium level analysis) or reagent water (for low level, <50 µg/kg, analysis). The exact samples weight is determined as the difference between the vial + preservative weight and weight after the sample is added.

- Samples must be transferred (extruded from the sampler) and preserved within 48 hours of sampling.
- Water preserved samples are then frozen at <10°C. Methanol preserved samples may be stored at > 0.0 °C but < 6° °C or frozen.
- Methanol preserved samples are shaken for at least 2 minutes, and a portion of the methanol extract after settling may be transferred to a smaller Teflon-lined capped vial for storage below 6 °C
- Normally one (1) medium level and two (2) low-level samples are taken and preserved.
- One vial with a clean matrix of each preservation type is prepared at the same time as samples, to be used for LCS analysis. Spikes are not added until the time of analysis.
- Samples are spiked with internal standards and surrogates at the time of analysis.

8.1.3 Terra Core™ sampling kits are pre-preserved for use and immediate samples preservation in the field. Kits are shipped that include one (1) methanol preserved and two (2) reagent water preserved vials, along with a 4oz jar for solids/moisture analysis volume.

- Terra Core™ vials are immediately placed in the freezer (<-10°C) upon receipt at the lab. Methanol preserved vials are shaken for at least two (2) minutes to break up the solid and create the methanol extract.
- Terra Core™ vials are labeled with the weight of the vial and preservative. The vials are re-weighed prior to analysis to determine the weight of the solid sample added. It is important that labels NOT

be added to these vials prior to weighing, because the weight of the label will add to the sample weight. Vials may be marked with indelible marker, or placed in a labeled, sectioned box until ID labels can be added after weighing.

8.2 Unpreserved soils - At client request, unpreserved soils packed into glass jars or brass tubes may be accepted and subsampled in the lab. A 5g portion of the sample is transferred to a 40mL vial and mixed with reagent water and/or methanol for analysis. Since this procedure is not compliant with SW5035A an NCM and case narrative statement describing the non-conformance must be included with any resulting data reported to the client.

8.3 Aqueous samples are stored in 40mL glass vials with Teflon lined septa at >0 and ≤ 6.0°C. Vials are required to have no headspace larger than a small pea.

8.3.1 Samples from chlorinated water sources must be treated with sodium thiosulfate (0.008% solution) at the time of collection to remove chlorine. NOTE: containers pre-preserved with sodium thiosulfate must be requested in bottle orders for samples from chlorinated water sources

8.3.2 Regulatory requirements for 2-Chloroethyl vinyl ether:

- 2-Chloroethyl vinyl ether: The stability of this compound is reduced when subjected to low pH, therefore samples for analysis to include 2-CEVE must be taken without acid preservation. Unpreserved samples must be analyzed within 7 days.

- SW846 Update V removed special preservation requirements for Acolein and Acylonitrile. These compounds may be analyzed for using a preserved sample vial.

8.4 Soil samples and water samples preserved to pH <2 with HCl have a maximum holding time is 14 days from sampling until the sample is analyzed. If water samples are known to be unpreserved, the holding time is 7 days from sampling to analysis.

8.4.1 Preserved water samples are checked to confirm the preservation pH AFTER analysis because the vials must not be opened prior to analysis. If the pH is found to be >2, this must be addressed in the case narrative.

8.5 Medium level solid methanol extracts, if taken at the time of preservation, are aliquoted into 4 mL glass vials with Teflon lined caps and stored at > 0.0 °C but ≤6.0°C or frozen. The extracts are stored with minimum headspace.

8.6 Storage blanks are prepared by filling 40 mL VOA vials with reagent water and placing one in each refrigerator. After 1-2 weeks, the storage blanks are removed and analyzed. Additional details can be found in Eurofins Edison SOP No. ED-SPM-004, *Sample Storage & Handling Procedures for Mitigation of Sample and Laboratory Contamination,* current revision.

9.0 Quality Control

9.1 **Sample QC** - The following quality control samples are prepared with each batch of samples:

Quality Controls	Frequency	Control Limit
Method Blank (MB)	1 in 20 or fewer samples	< Rpt. Limit
Laboratory Control Sample (LCS)[1]	1 in 20 or fewer samples	Statistical Limits [4]
Matrix Spike (MS)[2]	1 in 20 or fewer samples	Statistical Limits [4]
MS Duplicate (MSD)[2]	1 in 20 or fewer samples	Statistical Limits [4]
Surrogates	every sample[3]	Statistical Limits [4]
Internal Standards	Every samples	Response within -50% to +100% of CCV

[1] LCS Duplicate (LCD) is performed only when insufficient sample is available for the MS/MSD or when requested by the client/project/contract.
[2] The sample selection for MS/MSD are randomly selected, unless specifically requested by a client….predetermined by the extraction lab.
[3] Analytical and QC samples (MB, LCS, MS/MSD)
[4] Statistical control limits are updated annually and are updated into LIMS.

9.1.1. **Method blanks** are analyzed every 12 hours immediately after successful calibration verification (ICV and CCV) and before any samples are analyzed during the 12 hour clock. Analyze the blank in the same manner as the associated samples.

9.1.1.1. Prepare an aqueous blank by filling a 40 mL vial with reagent water and placing it in the autosampler. The autosampler will add the internal standard and/or surrogate standard.

9.1.1.2. Prepare a medium or high level blank in a 50 mL volumetric flask by adding 1.0 mL of purge and trap grade methanol to reagent water and bringing up to volume with the reagent water. The appropriate volume of this mix is added to the purge vessel. The autosampler will automatically internal standard and/or surrogate standard.

9.1.1.3. Prepare a low- level soil blank in a 40 ml VOA vial by adding a magnetic stir bar and 5 ml of reagent water and placing the vial in the autosampler tray. An additional 5mL of reagent water plus 1uL of 250ppm Internal Standard/Surrogate Mix (see Section 7.2.4) will be added by the Archon prior to purging.

9.1.1.4. To be considered acceptable, the method blank must not have any target analytes above the reporting limit. For method 8260D method blank is acceptable when target analyte concentrations are less than one-half of the reporting limit. Method blanks may contain analyte concentrations greater than acceptance limits if the associated samples in the batch are unaffected or sample

concentrations/responses are >10x the blank If method blanks are unacceptably contaminated with target compounds that are also present in field samples, all affected samples must be re-extracted and re-analyzed. Re-analysis is not necessary if the analyte concentration falls well below the action or regulatory limit or if the analyte is deemed not important for the project. Corrective action must be taken to identify and eliminate the contamination source. Demonstrate that acceptable blanks can be obtained before continuing with sample extraction and analysis. Method blanks must be analyzed on each instrument on which the associated samples are analyzed. Method blanks, trip blanks and other field blanks must be carried out through all stages of sample preparation and analysis.

9.1.1.5. Surrogate recoveries for the method blank must be within the laboratory generated limits. (Method 8260D requires the use of a minimum of three (3) surrogates. Since we are spiking with four (4) surrogates, either 1,2-Dichloroethane-d4 or dibromofluoromethane can be recovered outside of control limits without corrective action) .Internal standard area counts in the method blank must be within method specified limits. If any surrogate or internal standard is outside the limits, the method blank must re-analyzed.

9.1.2. Matrix Spike (MS)/Matrix Spike Duplicate (MSD): A matrix spike/matrix spike duplicate (MS/MSD) pair is extracted and analyzed with every 20 environmental samples of a specific matrix (defined as a sample batch which may contain up to 20 samples, and additional samples can be added to the batch for 14 days after the first sample was analyzed). Full compound list spiking is employed for MS/MSDs and LCSs. These spikes are prepared (as described in Section 9.1.2.1) concurrent with sample preparation. MS and MSD recoveries are calculated and compared to lab generated acceptance criteria which are updated annually. For acceptance limits, reference the current TALS (LIMS) active Method Limit Group database.

9.1.2.1. Prepare the MS/MSD as follows:

9.1.2.1.1 Low Level Soil: The low level soil MS/MSD is prepared as detailed in the following table. This is prepared in duplicate (one for the MS, the other for the MSD) in a 5 ml syringe filled with reagent water. Once prepped the solution is added to separate 40 ml vials each containing 5 gram aliquots of the sample to be spiked :

Standard Solution (Reference Table 2, Lab Names)	Concentration	Volume of Standard (ul) Added to 5.0 ml of Reagent Water	Final Concentration (ug/kg)
Gas Mix Li	50ppm	2	20

Standard Solution (Reference Table 2, Lab Names)	Concentration	Volume of Standard (ul) Added to 5.0 ml of Reagent Water	Final Concentration (ug/kg)
8260 combined	50ppm	2	20
Acrolein	500 ppm	3	300
Propenes	50ppm (varied)	2	20 (varied)
Freons	50 ppm	2	20

9.1.2.1.2 Aqueous Samples: The MS/MSD for aqueous samples is prepared as detailed in the following table. This is prepared in duplicate (one for MS, the other for MSD) in 50 ml volumetric flasks filled with an aliquot of sample to be spiked. Once prepped the solution is poured into a 40 ml VOA vial and loaded onto the purge and trap autosampler:

Standard Solution (Reference Table 2, Lab Names)	Concentration	Volume of Standard (ul) Added to 50 ml of Sample	Final Concentration(ug/L)
Gas Mix Li	50ppm	20	20
8260 combined	50ppm	20	20
Acrolein	500 ppm	4	40
Propenes	50ppm (varied)	20	20 (varied)
Freons	50	20	20

9.1.2.1.3 Medium & High Level Soils: The MS/MSD for medium/high level soils is prepared as detailed in the following table. This is prepared in duplicate (one for MS, the other for MSD) in 50 ml volumetric flasks filled with reagent water which has been previously spiked with the methanol sample extract. Once prepped the solution is poured into a 40 ml VOA vial, the and loaded onto the purge and trap autosampler:

Standard Solution (Reference Table 2, Lab Names)	Concentration	Volume of Standard (ul) Added to 50 ml of Reagent Water containing sample methanol extract	Final Concentration (ug/L)
Gas Mix Li	50ppm	20	20
8260 conbined	50ppm	20	20
Acrolein	500ppm	4	40
Propenes	50ppm (varied)	20	20 (varied)
Freons	50 ppm	20	20

9.1.2.1.4 SIM: The MS/MSD for SIM samples is prepared as detailed in the following table. This is prepared in duplicate (one for MS, the other for MSD) in 100 ml

volumetric flasks filled with an aliquot of sample to be spiked. Once prepped, two separate 10ml solution is poured into 40 ml VOA vials, 2ul of SIM IS/S is then added to each vial and loaded onto the purge and trap autosampler:

Standard Solution	Concentration	Volume of Standard Added to 100 ml of Sample (ul)	Final Concentration (ug/L)
8260SIM Mix1	10ppm	0.5	0.05
1,4-Dioxane	50ppm (varied)	10	5
Benzene/Chloroform	10ppm	0.5	0.05

9.1.2.2. An Laboratory Control Sample (LCS) /Laboratory Control Sample Duplicate (LCSD) may be substituted for the MS/MSD if insufficient sample volume is available (see Section 9.1.3).

9.1.3. Laboratory Control Sample (LCS)/Laboratory Control Sample Duplicate (LCSD): A Laboratory Control Sample (LCS) (aka blank spike) must be prepared analyzed with each batch of 20 environmental samples. The LCS data is used to assess method performance if the MS/MSD recoveries fall outside of the lab generated limits (see For acceptance limits, reference the current TALS (LIMS) active Method Limit Group database). If the LCS recovery is within the current lab generated limits, the MS/MSD recoveries are attributed to matrix interference. If the LCS recovery results are outside the method specified, the LCS is reanalyzed. If, upon reanalysis, the LCS is it is still outside of limits the entire batch must be reanalyzed. For 8260D, when an LCS is prepared in the same manner as CCV, the same standard can be used as both the LCS and CCV.

9.1.3.1 For LCS preparation instructions please refer to Section 9.1.2.1 for low level soil introduction technique (note: use reagent water only, no solid matrix is used when preparing the LCS) and Sections 9.1.2.1.2 and 9.1.2.1.3 as applicable for aqueous/medium or high level solids introduction (note: use reagent water only, no sample or sample extract is used when preparing the LCS).

9.1.3.2 The LCS for SIM samples is prepared as detailed in the following table. This is prepared in a 200 ml volumetric flasks filled with organic free reagent water. Once prepped, 10ml of the solution is poured into a 40 ml VOA vial and 2ul IS/SS added manually and loaded onto the purge and trap autosampler

Standard Solution	Concentration	Volume of Standard Added to 200 ml of Reagent Water (ul)	Final Concentration (ug/L)
8260 Mix1	10ppm	1	0.05
1,4-Dioxane	50ppm	20	5
Benzene/Chloroform	10ppm	1	0.05

9.1.3.3 A Laboratory Control Sample Duplicate (LCSD) is analyzed only when insufficient client sample is available for preparation of an MS/MSD pair. The LCS/LSCD is evaluated in the same manner as the MS/MSD (see Section 9.1.2)

9.1.4. Surrogate Standards: All samples, blanks and QC samples are spiked with a four (4) component surrogate standard mix (see Section 7.2.2). The percent recovery of the surrogate standards is calculated and compared to lab generated limits (For acceptance limits, reference the current TALS (LIMS) active Method Limit Group database).

9.1.4.1. Surrogate recovery limits are lab generated and are updated annually.

9.1.4.2. Surrogate recoveries are calculated for the blank, samples, and QC samples. Surrogate recovery is calculated as:

$$\frac{\text{Concentration found}}{\text{Concentration added}} \times 100 = \%\ \text{RECOVERY}$$

9.1.4.3. If the surrogate recoveries of any blank, sample, or QC sample fails to meet the current recovery criteria, the sample must be re-analyzed. If a surrogate is diluted to a concentration below that of the lowest calibration standard, no corrective action is necessary. Method 8260D requires the use of a minimum of three (3) surrogates. As we spike with four (4) surrogates, one can be recovered outside of control limits without corrective action.

9.1.5. Internal Standards: All samples, blanks, standards and QC samples are spiked with a five (5) component internal standard mix (See Section 7.2.3). The response (area count) and retention time of each internal standard in all samples, standards, blanks and QC samples are monitored.

9.1.5.1. The internal standard responses must be within -50 +100% of its corresponding internal standard in the mid-level calibration standard or the active calibration curve. Failure to meet these criteria is indicative of sample matrix effects. All samples failing these criteria must be reanalyzed to confirm matrix effects.

9.1.5.2. Internal standard retention time is evaluated immediately after acquisition. The retention times of the internal standards must be within ±30 seconds of the internal standards from the mid point standard of the initial calibration or the calibration verification standard. Any blank, sample, or QC sample that fails to meet these criteria must be re-analyzed.

9.2 Instrument QC

9.2.1 GC/MS Instrument Performance Check (BFB): The GC/MS system is tuned using Perfluortributylamine (PFTBA) such that an injection or purging of 50ng of 4-Bromofluorobenzene (BFB) meets the abundance criteria listed in the table below. Prior to the analysis of any calibration standards or samples, the GC/MS system must meet all BFB key ion abundance criteria. This analysis will verify proper tuning of the system for a period of 12 hours post-injection. After 12 hours, the instrument performance must again be verified prior to the analysis of standards, QC or samples. For method 8260D tune checks are only required prior to initial calibration. (**NOTE**: see Method Modifications in Section 16.0).

BFB Key Ions and Abundance Criteria	
Mass	**Ion Abundance Criteria**
50	15.0-40.0 percent of the base peak
75	30.0-60.0 percent of the base peak
95	Base peak, 100% relative abundance
96	5.0-9.0 percent of the base peak
173	Less than 2.0% of mass 174
174	Greater than 50% of the base peak
175	5.0-9.0 percent of mass 174
176	Greater than 95.0% but less than 101% of mass174
177	5.0-9.0 percent of mass 176

9.2.1.1. The BFB mass spectrum may be evaluated using one of the procedures listed below. The spectrum may be background subtracted using a single peak no more than 20 scans before the peak apex. The BFB spectrum must meet the technical acceptance criteria listed in the table above:

- A single scan on the peak;
- An average of the peak;
- Use of three scan averaging and background subtraction techniques. Select the scan at the BFB peak apex, add +1 scan from the apex and -1 scans from the apex;

9.2.1.2. BFB parameter settings are stored in a tune file, which ill be used in all subsequent analysis of standards and samples.

9.2.2 Initial Calibration Range and Initial Calibration Verification

9.2.2.1. **Initial Calibration:** The initial calibration range consists of a five-point concentrations (six points for second order regression) of analytical standards prepared as described in Tables 3, 3A and 3B as applicable (attached). The initial calibration range must be analyzed only after the BFB instrument performance check has met the criteria in Section 9.2.1. A separate initial calibration range is analyzed for each sample introduction technique. The last initial calibration standard may be used to be the start of the 12 hour clock for samples analyzed after initial calibration. Verify closely eluting isomers resolution in the mid-point concentration of the ICAL. Isomers are considered resolved if the peaks are at least 50% resolved (i.e., the height of the valley between two isomer peaks is less than or equal to 50% of the average of the two peak heights. This should also be checked in the daily CC's.

9.2.2.2. If analysis by the SIM technique is required, prepare calibration standards for, Vinyl Chloride, Chloroform, Benzene 1,2-dibromoethane, 1,2,3-Trichloropropane and 1,2-dibromo-3-chloropropane at concentrations of 0.02, 0.04, .05, 0.10, 0.20, 0.50, 1.0 and 2.0 ppb; 1,4-Dioxane at 0.4, 1, 5, 10, 20, 30, 40 and 50ppb. See Table 5 that summarizes the preparation information.

9.2.2.3. **Initial Calibration Verification (ICV):** An Initial Calibration Verification (ICV) standard is analyzed immediately after the Initial Calibration Range and before any samples are analyzed. The ICV is prepared as detailed in Section 7.2.1.3 and Tables 4 and 4a (full scan) and Table 6 (SIM) (attached). The ICV must be from a source separate from the standards used in the Initial Calibration Range.

9.2.3 **Continuing Calibration Verification (CCV):** A approximately mid-point (20ug/ml and 0.050/5ug/ml for SIM) Continuing Calibration Verification (CCV) must be analyzed every 12 hours after the BFB instrument performance check. BFB is not a requirement for 8260D CCV verification. The CCV is prepared as detailed in Section 7.2.1.1 and Table 3 (attached).

9.2.4 **Calibration Acceptance Summary**

9.2.4.1. **Retention Time:** The relative retention times of each compound in the five calibration standards must agree within 0.06 relative retention time units.

9.2.4.2. **Initial Calibration Range:** Internal standard calibration is employed for this method. After the initial calibration range has been analyzed as detailed in Section 10.3.3 the relative response factor (RRF) for each target/surrogate compound at each concentration level is determined using the following equation.

$$RRF = \frac{A_x}{A_{is}} \times \frac{C_{is}}{C_x}$$

Where:
A_x = Area characteristic ion for the compound (see attached Table 7)
Ais = Area characteristic ion of internal standard (see attached Table 7)
Cis = Concentration of internal standard
Cx = Concentration of compound in standard

9.2.4.2.1. Determine the mean RRF for each compound using the five or six RFs from the initial calibration range.

9.2.4.2.2. The average RFs of the target analytes listed in the table below must meet the indicated minimum RF criteria:

Minimum Relative Response Factor	
Common Target Analytes	**Minimum RF**
Dichlorodifluoromethane	0.100
Chloromethane	0.100
Vinyl Chloride	0.100
Bromomethane	0.100
Chloroethane	0.100
Trichlorofluoromethane	0.100
1,1-Dichloroethene	0.100
1,1,2-Trichloro-1,2,2-trifluoroethane	0.100
Acetone *	0.050
Carbon disulfide	0.100
Methyl Acetate *	0.005
Methylene chloride	0.100
trans-1,2-Dichloroethene	0.100
cis-1,2-Dichloroethene	0.100
Methyl tert-Butyl Ether	0.100
1,1-Dichloroethane	0.200
2-Butanone *	0.050
Chloroform	0.200
1,1,1-Trichloroethane	0.100
Cyclohexane	0.100
Carbon tetrachloride	0.100
Benzene	0.500
1,2-Dichloroethane	0.100
Trichloroethene	0.200
Methylcyclohexane	0.100
1,2-Dichloropropane	0.100
Bromodichloromethane	0.200
cis-1,3-Dichloropropene	0.200
trans-1,3-Dichloropropene	0.100
4-Methyl-2-pentanone *	0.050
Toluene	0.400

Minimum Relative Response Factor	
Common Target Analytes	**Minimum RF**
1,1,2-Trichloroethane	0.100
Tetrachloroethene	0.200
2-Hexanone*	0.050
Dibromochloromethane	0.100
1,2-Dibromoethane	0.100
Chlorobenzene	0.500
Ethylbenzene	0.100
meta-/para-Xylene	0.100
ortho-Xylene	0.300
Styrene	0.300
Bromoform	0.100
Isopropylbenzene	0.100
1,1,2,2-Tetrachloroethane	0.300
1,3-Dichlorobenzene	0.600
1,4-Dichlorobenzene	0.500
1,2-Dichlorobenzene	0.400
1,2-Dibromo-3-chloropropane	0.050
1,2,4-Trichlorobenzene	0.200

Note: Alternate ions chosen for the analytes in the table above may result in lower than recommended value

* These values are lower than method recommended values.

9.2.4.2.3. Any individual analyte that fails the minimum response factor above must have a demonstration of sensitivity in the analytical batch to report non-detects. The demonstration of sensitivity is analysis of a low level CCV (at or below the reporting limit). The criterion for a passing LLCCV is detection only, and a passing LLCCV allows non-detects to be reported without flagging. The low level CCV would normally be analyzed immediately after the mid-level CCV

9.2.4.2.4. Calculate the Standard Deviation (SD) and Percent Relative Standard Deviation (% RSD) of the response factors for each compound:

$$\% \text{ RSD} = \frac{\text{Standard Deviation of RRFs}}{\text{Mean RRF}}$$

The % RSD of the common target compounds listed above must be ≤20% for average RF in order for the calibration range to be acceptable. If more than 10% of the compounds exceed the 20%RSD limit and do not meet the minimum correlation coefficient (0.99) or Relative Standard Error (RSE) of ≤20% for alternative curve fits, appropriate instrument maintenance like source cleaning should be performed. Any compound that do not meet the 20%

RSD or 0.99 correlation coefficient/RSE criteria must be flagged as estimated for detects.

9.2.4.2.5. For all compounds (including those analyzed by SIM): in order to assume linearity, the % RSD of the RRF's for each target analyte must be ≤20%.

9.2.4.2.6. If the above listed criteria is met, the system can be assumed to be linear, sample analysis may begin and the average RF from the initial calibration range may be used to quantitate all samples.

9.2.4.2.7. An alternative calibration technique may be employed for those any compounds exceeding the 20% RSD criteria:

9.2.4.2.5.4 Linear regression: Calculate the first order linear regression for any compound which did not meet the 20% RSD criteria the r^2 (Correlation Coefficient) value must be $\geq$ 0.99 or the Relative Standard Error (RSE) (≤20%) for the calibration to be employed.

9.2.4.2.5.4 Quadratic (or second order) regression: may be used if the linear regression correlation coefficient exceeds criteria. Quadratic regression requires the use of a minimum six calibration points. If second order regression calibration is used, , the r^2 (Correlation Coefficient) value must be $\geq$ 0.99 or the Relative Standard Error (RSE) (≤20%) for the calibration to be employed.

9.2.4.2.8. If neither of the alternative calibration techniques meets acceptance criteria i.e for more than 10% of the analytes fail both 20%RSE and 0.990 the calibration is not valid. Corrective action must be taken and the initial calibration range reanalyzed.

9.2.4.2.9. Non-detect results for any analyte that fails both 20%RSD/RSE and 0.990 correlation coefficient may be reported without flagging if (and only if) there has been a successful analysis of a LLCCV (CCV at the reporting limit) in the same analytical batch. The criterion for the LLCCV is detection only (%D criteria are not applied) but the standard qualitative criteria in the method must be met. Flagging of detected analytes results as estimated is discouraged when the 20%RSD/RSE and 0.990 criteria fails. In general no more than one or two of the poorest performing analytes should fail both criteria.

9.2.4.2.10. Due to significant bias to the lower portion of a calibration curve using the linear regression fit model a quantitation check on the viability of the lowest calibration point should be performed by re-fitting the response from the low concentration calibration standard back into the curve as if it were an unknown sample (rename the lower point calibration file as a separate data file before re-processing). The results should be within ±30% of the standard's true concentration. This is not required for average RF or quadratic fits. Additionally forcing a linear regression through zero will meet the requirement of not re-fitting. Analytes which do not meet the minimum quantitation calibration re-fitting criteria should be considered 'out of control'. Report those target analyte outliers as estimated when the concentration is at or near the lowest calibration point and/or report to the next reporting level (i.e., the next higher calibration point for the analyte).

9.2.4.2.11. For additional detail refer to Eurofins Edison Work Instruction No. EDS-WI-096, *8260C ICAL Procedure*, latest revision.

9.2.4.3. Calibration Point Read-back Criteria: Whichever calibration model above is selected, it should be subjected to an additional check to establish the representativeness of the data that were used to produce it. This check is the refitting of each calibration point response back to the calibration model, or the comparison of the calculated amount of the standard against the expected amount.

- CHROM software provides an Initial Calibration %Drift report which shows the % Error for each calibration point. This report must be reviewed in addition to the %RSD Linear Response Factor.

- The absolute value of the % Error for each calibration point should be < 30%. For the lowest calibration point, the % Error may be < 50%. Relative standard error (RSE) can also be used and must be ≤ 20% for each calibration point. See section 11.10 for the Calculation of the %Error.

9.2.4.4. Initial Calibration Verification (ICV): Once the initial calibration has been analyzed and has met the above criteria, a second source Initial Calibration Verification (ICV) (as prepared in Section 9.2.2.2) must be analyzed and evaluated. The ICV must meet the criteria of 70-130% recovery for all compounds however up to 10% of the compounds are allowed to exceed

this criteria as long as their recoveries are within 65-135%. For the poor performers the range is 50-150%. If the criterion is not met, a second ICV may be analyzed after corrective measures are taken. If a second ICV analysis fails to meet criteria proceed with corrective action and the analysis of a new initial calibration range. Flagging: If the ICV limits are outside of criteria (high) for an analyte and that analyte is undetected in the sample, no flagging or narration is required. If the ICV limits are outside of criteria (low) for an analyte and that analyte is undetected in a sample, narrate the non-conformance in an NCM. When that out of spec analyte is detected in a sample, describe the issue in the narrative, or flag as estimated.

9.2.4.5. Continuing Calibration Verification (CCV): A CCV consisting of a standard at or near the midpoint of the Initial Calibration Range is analyzed every 12 hours of instrument operation or at the beginning of an analytical sequence to verify the initial calibration. The calibration verification consists of a BFB instrument performance check, and analysis of a calibration verification standard.

9.2.4.4.1 Tune Verification: Follow the procedure for verifying the instrument tune described in section 9.2.1 using a 50 ng injection of BFB. If the tune cannot be verified, analysis must be stopped, corrective action taken and a return to "control" demonstrated before continuing with the calibration verification process. For method 8260D, tune verification is not required for daily CCV.

9.2.4.4.1.1 Calibration Verification: Analyze the calibration verification standard immediately after a BFB that meets criteria. For method 8260D, BFB is not needed. Use the mid point calibration standard (20ug/L). **NOTE:** The same sample introduction technique employed for the initial six-point calibration must be used for the calibration verification.

9.2.4.4.1.2 Calculate response factors (RF) for each compound using the internal standard method.

9.2.4.4.1.3 The RFs must meet the minimum RF criteria listed in the table in Section 9.2.4.2.2.

9.2.4.4.1.4 Calculate the % Difference for each response factor in the calibration check standard vs. the response factors from the initial calibration.

9.2.4.4.1.5 If the percent difference/drift (%D) for the compounds listed in the table in Section 9.2.4.2.2 is ≤20%, the initial calibration is assumed to be valid. If the ≤20% D criteria is not met for more than 20% of the compounds in the initial calibration, corrective action/ investigation may be taken. After corrective action, another calibration verification standard may be injected. If the response for the analyte is still not ≤20%, a new initial calibration range must be generated.

9.2.4.4.1.6 For the poor performing compounds listed below that fail the 20%D or 50%D criteria adequate sensitivity may be demonstrated by including a low level standard (LLCCV) in the analytical batch.

Poor Performers	
Acetone	Acrolein
Carbon disulfide	1,4-Dioxane
2-Butanone	Cyclohexane
2-Hexanone	Methyl cyclohexane
4-Methyl-2-pentanone	Benzyl chloride
Chlorodibromomethane	Naphthalene
1,2-Dibromo-3-chloropropane	Cis-Dichloropropene
Bromomethane	Trans-Dichloropropene
Chloroethane	All Alcohols

When samples have non-detects for an analyte that fails the SOP criteria with low recovery a low level CCV must be analyzed in the batch as a demonstration of adequate sensitivity. The criterion for a passing LLCCV is detection only, and a passing LLCCV allows non-detects to be reported without flagging. Any sample detects for an analyte that fails the SOP criteria must be flagged as estimated, or detailed in the case narrative. In all cases every effort should be made to re-analyze on an instrument with a passing CCV.

9.2.4.4.1.7 Percent drift is used instead of percent difference in calibrations employing either the linear or second order regression modes.

9.2.4.4.1.8 For the compounds not listed in the table in Section 9.2.4.2.2: No one individual compound of interest may exceed 50%D. For SIM analysis the %D is 20%.

9.2.4.4.1.9 The retention times of the internal standards from the calibration check must be within ±30 seconds of the internal standards from the mid point standard of the original calibration. If the retention time for any internal standard changes by more than 30 seconds from the latest daily (12 hour) calibration standard, the chromatographic system is inspected for malfunctions, and corrections made as required. If corrective action does not result in the retention time criteria being achieved, the system must be re-calibrated using four additional standards.

9.2.4.4.1.10 Internal standard area response is also evaluated immediately after acquisition. The response (area count) of each internal standard in the calibration verification standard must be within 50% - 100% of its corresponding internal standard in the mid-level calibration standard of the initial calibration curve. If the EICP area for any internal standard changes by more than a factor of two (-50% to +100%), the mass spectrometer system must be inspected for malfunction and corrections made as appropriate. When corrections are made, re-analysis of samples analyzed while the system was malfunctioning is required.

10.0 Procedure

10.1. Gas Chromatograph/Mass Spectrometer Operation

10.1.1. The instrument operating parameters are set as follows at the beginning of a method of analysis and remain constant throughout the entire analytical procedure

10.1.1.1 Full Scan Operating Mode

Purge and trap unit

Purge Time:	11 minutes
Dry Purge:	1 Minutes
Purge Gas:	Nitrogen
Purge Flow:	40-45 ml/min
Purge Temp:	Water: Ambient; Solids: 40°C
Trapping Temp:	Ambient, <30°C

Desorb Time:	1 Minute
Desorb Temp:	VOCARB: 260°C, #10: 190°C

Gas chromatograph

Injector:	180°C
Carrier Gas:	Helium
Carrier Flow:	6 ml/min, 6890: 0.8 ml/min
Oven Program:	40°C for 1 min, 8°C/min to 90°C, 20°C/ min to 250°C for 3 min; 6890: 40°C for 1 min, 8°C/min to 100°C, 24°C/min to 220°C for 2 min
Run Time:	15 - 20 Minutes

Mass Spectrometer

Electron Energy:	70 volts (nominal)
Mass range:	35-260 AMU
Scan time:	0.9 sec./scan
Source Temp:	200°C
Separator Temp:	180°C

10.1.1.2 SIM Operating Mode

Purge and trap unit

Purge Time:	11 minutes
Dry Purge:	1 Minutes
Purge Gas:	Nitrogen
Purge Flow:	40-45 ml/min
Purge Temp:	Water: Ambient; Solids: 40°C
Trapping Temp:	Ambient, <30°C
Desorb Time:	1 Minute
Desorb Temp:	VOCARB: 260°C, #10: 190°C

Gas chromatograph

Injector:	180°C
Carrier Gas:	Helium
Carrier Flow:	6 ml/min, 6890: 0.8 ml/min
Oven Program:	40°C for 1 min, 8°C/min to 90°C, 20°C/ min to 250°C for 3 min; 6890: 40°C for 1 min, 8°C/min to 100°C, 24°C/min to 220°C for 2 min
Run Time:	15 - 20 Minutes

Mass Spectrometer

Electron Energy:	70 volts (nominal)
Mass range:	35-260 AMU
Scan time:	0.9 sec./scan
Source Temp:	200°C
Separator Temp:	180°C

SIM Parameters:

Group 1
Plot 1 Ion: 51.0/96

Ions/Dwell in Group	(Mass	Dwell)	(Mass	Dwell)	(Mass	Dwell)
	51.0	100	58.0	100	65.0	100
	67.0	100	70.0	100	88.0	100
	96.0	100	78.0	100	83.0	100
	85.0	100	62.0	100	64.0	100

Group 2
Group Start Time: 6.20
Plot 1 Ion: 82/117

Ions/Dwell in Group	(Mass	Dwell)	(Mass	Dwell)	(Mass	Dwell)
	82.0	100	107.0	100	109.0	100
	117.0	100				

Group 3
Group Start Time: 8.50
Plot 1 Ion: 75/157

Ions/Dwell in Group	(Mass	Dwell)	(Mass	Dwell)	(Mass	Dwell)
	75.0	100	95.0	100	150.0	100
	152.0	100	152.0	100	157.0	100
	174.0	100				

10.2. Sample Preparation

10.2.1. Screening: All samples extracts must be screened by GC/FID static headspace analysis to provide the analyst with appropriate initial dilution factors. For additional details see Eurofins Edison SOP No. ED-GCV-001, *Screening for Volatile Organics, Static Headspace with GC FID, SW846 Method 5021*, current revision.

10.2.2. Aqueous Samples: Unopened 40 mls vials with aqueous samples are placed in an Archon autosampler. 1 uL of Internal Standard/Surrogate Mix (see Section 7.2.4) is added by the Archon as the 5 mL of the sample passes through the sample loop.

10.2.3. Medium or high level soils: Medium or high level extracts that will be run on an Archon autosampler are prepared in 50mL volumetric flasks.

The Archon can be set up to add 1uL of 250ppm Internal Standard/Surrogate separately (see Section 7.2.3 and 7.2.2.2) to each sample as the 5mL portion passes through the sample loop.

10.2.4. **Low level soils:** Low level soils must be run on an Archon autosampler. 1uL of 250ppm Internal Standard/Surrogate separately (see Section 7.2.3 and 7.2.2.2) and 5mL reagent water is added to each sample vial by the Archon immediately before the sample is purged.

10.2.5. **SIM analysis:** Aliquot 10ml of sample and manually add 2ul of 2.5/50ppm of internal standard/surrogate mix. Load to soil section of the autosampler for heated purge.

10.3. Instrument Performance and Calibration Sequence

10.3.1. Once the GC/MS instrument has been setup and maintained as detailed in Section 10.1, the first operations to be performed are the performance checks and calibration standards.

10.3.2. Analyze the Instrument Performance Check Standard (BFB) as discussed in Section 9.2.1.

10.3.3. A unique initial calibration is then prepared for each sample introduction technique.:

10.3.3.1 **40 ml VOA Vial (Aqueous/Medium-High Level Soils):** Prepare aqueous calibration standards at six concentration levels for each parameter by adding the volumes of working standards listed in Table 3 to a 50mL volumetric flask of reagent water. Pour the calibration standards into 40mL VOA vials and load into the autosampler tray. If the internal standard is to be added by the Archon/OI autosamplers the addition of internal standard into the 50ml volumetric flaks may be omitted.

10.3.3.2 **40 ml VOA Vial (Low Level Soils):** If the calibration is for low-level soils prepared according to Method 5035AA, the calibration standards must be prepared by adding the volumes of working standards listed in Table 3 into a 5 mL syringe filled with reagent water and pouring the prepared standards into 40 mL VOA vials containing a magnetic stir bar.

10.3.4. Purge the standard for 11 minutes.

10.3.5. After purging is complete, desorb the sample onto the GC column by rapidly heating the trap to 260°C for VOCARB, 190°C for #10 and backflushing it with helium.

10.3.6. Begin the GC temperature program and data acquisition.

10.3.7. Re-condition the trap by baking for 12 minutes at 260°C for VOCARB, 210°C for #10.

10.3.8. Cool the trap to (<31°C). The trap is now ready for the next sample.

10.3.9. Transfer data to network, and process using CHROM software.

10.4. Sample Analysis Sequence

10.4.1. Once the initial calibration has been verified by successful analysis of an ICV and Method Blank, analysis of samples may begin.

10.4.2. Samples must be analyzed under the same instrument conditions and using the same injection volume as the calibration standards.

10.4.3. Equilibrate all samples to room temperature prior to analysis.

10.4.4. If the sample concentration exceeds that of the range, the sample must be diluted and re-analyzed.

10.4.5. The analytical run log is printed as a record of samples analyzed. The analyst will annotate the run log with any required information regarding anomalies or unusual events. The run log must be signed by the analyst and a reviewed and signed by a trained peer or manager

10.5. Data Processing

10.5.1. Prior to processing any standards or samples, target compound lists and sublists must be assembled in the Chrom system. These lists are required for processing of all data files including calibration files. The data includes compound names, retention time data, quantitation ions, qualitative identification ions, and the assigned internal standard for qualitative and quantitative identification.

10.5.2. Key data is manually entered the first time a compound list is used for data processing. Processing data using a compound list automatically generates response factor data and updates retention information.

10.5.3. Data is transferred from the acquisition PC to the network for auto-processing with CHROM software.

10.5.4. Each data file is checked for correct information including sample number, job number, QA batch, dilution factor, initial volume, final volume, and % moisture.

10.5.5. The data processing service from Chrom queries LIMS for the sample processing parameters.

10.5.6. Each data file is processed using calibration factors from the most recent initial calibration, quantitation from the daily calibration verification standard is not permitted.

10.5.7. The characteristic ions for target compounds, surrogate compounds, and internal standards which can be determined using SW8260D are listed in Table 7.

10.6. Interpretation and Qualitative Identification:

10.6.1 Target Analytes: Qualitative identification of target compounds is based on retention time and mass spectral comparison with characteristic ions in the target compound list. The reference mass spectrum is taken from a standard of the target compound analyzed by this method. The characteristic ions are the three ions of greatest relative intensity or any ions over 30% relative intensity if less than three such ions occur in the reference spectrum. Compounds are identified as present when the following criteria are met:

10.6.1.1. Once the GC/MS instrument has been setup and maintained as detailed in Section 10.1, the first operations to be performed are the performance checks and calibration standards.

10.6.1.2. The intensities of the characteristic ions of a compound maximize in the same scan or within one scan of each other.

10.6.1.3. The relative retention time (RRT) of the sample component is within ± 0.06 RRT units of the RRT of the standard component.

10.6.1.4. The most abundant ion in the standard target spectrum that equals 100% MUST also be present in the sample target spectrum.

10.6.1.5. All other ions that are greater than 10% in the standard target spectra should also be present in the sample.

10.6.1.6. The relative intensities of the characteristic ions agree within 30% of the relative intensities of these ions in the reference spectrum. (Example: For an ion with an abundance of 50% in the reference spectrum, the corresponding abundance in a sample spectrum can range between 20% and 80%).

10.6.1.7. Structural isomers that produce very similar mass spectra should be identified as individual isomers if they have sufficiently different GC retention times. Otherwise, structural isomers are identified as isomeric pairs.

10.6.1.8. If the compound does not meet all of the criteria listed above, but is deemed a match in the technical judgment of the mass spectral interpretation specialist, the compound

will be positively identified and reported with documentation of the identification noted in the raw data record.

10.6.2 Non-Target Analytes: Upon client request a library search to identify non-target Tentatively Identified Compounds (TIC) is performed. The NIST/EPA/NIH mass spectral library is used to identify non-target compounds (not including internal standard and surrogate compounds) of greatest apparent concentration by a forward search of the library. The following guidelines are used by the analyst when making TIC identifications:

10.6.2.1 Relative intensities of major ions in the reference spectrum (ions greater than 10% of the most abundant ion) should be present in the sample spectrum.

10.6.2.2 The relative intensities of the major ions should agree within ±20%. (Example: For an ion with an abundance of 50% in the standard spectrum, the corresponding sample ion abundance must be between 30 and 70%).

10.6.2.3 Molecular ions present in the reference spectrum should be present in the sample spectrum.

10.6.2.4 Ions present in the sample spectrum but not in the reference spectrum should be reviewed for possible background contamination or presence of co-eluting compounds.

10.6.2.5 Ions present in the reference spectrum but not in the sample spectrum should be reviewed for possible subtraction from the sample spectrum because of background contamination or co-eluting peaks. Data system library reduction programs can sometimes create these discrepancies.

10.6.2.6 If, in the technical judgement of the mass spectral interpretation specialist, no tentative identification can be made, the compound will be reported as 'Unknown'. If the compound can be further classified the analyst may do so (i.e, 'Unknown hydrocarbon', 'Unknown acid' , etc..).

10.7. Data Reporting

10.7.1. Final Report. LIMS TALS system automatically produces a data report consisting of key, hardcopy reports corresponding to specific data reporting requirements.

10.7.1.1. Total Ion Chromatogram. Full length chromatogram depicting the full length of the GC/MS acquisition.

10.7.1.2. Spectra of all detected target compounds. A page for each detected target compound spectra with a standard reference spectrum for comparison.

10.7.1.3. The calculations of the concentrations of each target compound in the sample, reported in units of ppb, ug/kg or ug/l.

10.7.1.4. Data summaries for each method blank indicating which samples were extracted with the indicated blank.

10.7.1.5. A copy of the initial calibration range together with the calibration verification report, and tune report.

10.7.1.6. Quality Control (QC) data report for each batch including surrogate recoveries, internal standard area summaries, LCS, MS/MSD and RPD summaries.

11.0. Calculations / Data Reduction

11.1. Target Compounds: are quantitated using the internal standard method.

11.1.1. Identified target compounds are quantitated using the integrated abundance from the EICP of the primary characteristic ion. The internal standard used shall be the one nearest the retention time of the analyte).

11.1.2. The average response factor (RRF) from the initial calibration is used to calculate the target analyte concentration in client samples using the formula found in Section 11.3.. See Section 9.2.4.2 for discussion of RRF.

11.1.3. Secondary ion quantitation is utilized only when there are sample interferences preventing use of the primary characteristic ion. If secondary ion quantitation is used an average relative response factor (RRF) must be calculated using that secondary ion.

11.1.4. Aqueous Samples

$$\text{Concentration } (\mu g/L) = \frac{(As)(Cis)(D)}{(Ais)(RRF)(Vs)}$$

Where:

As = Area of the characteristic ion for the target analyte in the sample

Cis = Concentration of the internal standard (ug/L)

D = Dilution factor, if the sample or extract was diluted prior to analysis. If no dilution is performed, D = 1.

Ais = Area of the characteristic for the associated internal standard

RRF = Average relative response factor from the initial calibration.

Vs = Volume of sample purged (ml)

11.1.5. Low Level Solid Samples

$$\text{Concentration (µg/Kg) (dry wt)} = \frac{(As)(Cis)}{(Ais)(RRF)(Ws)\,(DW)}$$

Where:

As = Area of the characteristic ion for the target analyte in the sample

Cis = Concentration of the internal standard (ug/L)

DW = Dry wt correction = $\frac{100 - \%\ \text{moisture}}{100}$

Ais = Area of the characteristic for the associated internal standard

RRF = Average relative response factor from the initial calibration.

Ws = Weight of sample purged (g)

11.1.6. Medium Level Solid Samples

$$\text{Concentration (µg/Kg) (dry wt)} = \frac{(As)(Cis)(Vt)(1000)(D)}{(Ais)(RRF)(Va)(Ws)(DW)}$$

Where:

As = Area of the characteristic ion for the target analyte in the sample

Cis = Concentration of the internal standard (ug/L)

D = Dilution factor, if the sample or extract was diluted prior to analysis. If no dilution is performed, D = 1

DW = Dry wt correction = $\frac{100 - \% \text{ moisture}}{100}$

Ais = Area of the characteristic for the associated internal standard

RRF = Average relative response factor from the initial calibration.

Va = Volume of the aliquot of sample methanol extract added to reagent water for purging in ul

Vt = Total volume of methanol extract in milliliters

Ws = Weight of sample purged (g)

11.2. Non-Target Compounds (Tentatively Identified Compounds): An estimated concentration for non-target (tentatively identified compounds) is calculated using the internal standard method . For quantiation, the nearest eluting internal standard free of interferences is used. The procedure used for calculating the concentration of non-target compounds is the same as that used for target compounds (see Section 11.1) with the following revisions:

11.2.1. The total area count of the non-target compound is used for As (instead of the area of a characteristic ion).

11.2.2. The total area count of the chosen internal standard is used as Ais (instead of the area of a characteristic ion).

11.2.3. A RF on 1.0 is assumed.

11.2.4. The resulting concentration is qualified as estimated ('J') indicating the quantitative uncertainties of the reported concentration.

11.3. Relative Response Factors

$$RRF = \frac{A_x}{A_{is}} \times \frac{C_{is}}{C_x}$$

Where:

A_x = Area characteristic ion for the compound (see Table 7)
Ais = Area characteristic ion of associated internal standard (See Table 7)
Cis = Concentration of internal standard
Cx = Concentration of compound in standard

11.4. Percent Relative Standard Deviation (% RSD) : as discussed in Section 9.2.4.2. (Initial calibration):

$$\% \text{ RSD} = \frac{\text{Standard Deviation of RRFs}}{\text{Mean RRF}}$$

11.5. Percent Difference (% D):as discussed in Section 9.2.4.4 (Continuing calibration):

$$\% \text{ D} = \frac{RRF_c - \overline{RRF_i}}{\overline{RRF_i}} \times 100$$

Where: RRFc = RRF from continuing calibration

$\overline{RRF_i}$ = Mean RRF from current initial calibration

11.6. Percent Recovery (% R): Surrogates and Spikes

$$\text{Recovery (\%)} = \frac{\text{Concentration (or amount) found}}{\text{Concentration (or amount) added}} \times 100$$

11.7. Dry Weight Correction: All solid samples must be corrected for dry weight using the following formula for dry weight determination.

$$DW = \frac{Gd}{Gw} \times 100$$

Where:

DW	=	Percent % Dry Weight
Gd	=	Dry weight of selected sample aliquot
Gw	=	Wet weight of selected sample aliquot

Multiply the DW value times the wet weight of the sample extracted. NOTE: This calculation can also be performed automatically by the target system provided the DW value is available and entered into the system.

11.8. Accuracy:

$$\text{ICV , CCV and LCS \% Recovery} = \frac{\text{observed concentration}}{\text{known concentration}} \times 100$$

$$\text{MS \% Recovery} = \frac{\text{(spiked sample) - (unspiked sample)}}{\text{spiked concentration}} \times 100$$

11.9. Precision (RPD):

$$\text{Matrix Duplicate (MD)} = \frac{|\text{orig. sample value - dup. sample value}|}{[(\text{orig. sample value + dup. sample value})/2]} \times 100$$

11.10. Calculation of Percent (%) Error:

$$\%Error = \frac{x_i - x_i'}{x_i} x\,100$$

Where:

x_i ' = Measured amount of analyte at calibration level *i*, in mass or concentration units
x_i = True amount

11.11. Relative Standard Error (RSE):

$$\%\,RSE = 100\% \times \sqrt{\frac{\sum_{i=1}^{N}\left[\frac{C_i - PC_i}{C_i}\right]^2}{(N-P)}}$$

Where:

N = Number of points in the curve
P = Number of parameters in the curve (= 1 for average response factor, 2 for linear, 3 for quadratic)
CI = True concentration for level i
PCi = Predicted concentration for level

12.0 Method Performance

12.1. Method Detection Limit Study (MDL)

A Method Detection Limit (MDL) study, as described in the Eurofins corporate Detection and Quantitation Limits SOP, CA-Q-S-006, must be performed initially and whenever a significant change affecting sensitivity is made to the analytical system. The MDL must be re-evaluated from quarterly MDL points at least every 12 months.

12.2. Demonstration of Capabilities

For DOC procedure refer to Section 19 in the most current revision of Eurofins Edison's Quality Assurance Manual (ED-QA-LQM).

12.3. Lower Limit of Quantitation Verification

The lowest calibration standard analyzed establishes the LLOQ or Reporting Limit. The capability to reliably detect this concentration through the preparation, clean-up and analytical procedure is verified through the annual analysis of a standard at the LLOQ/RL. The LLOQ verification shall also be performed whenever significant changes are made to the preparation and/or analytical procedure. The annual LLOQ verification is completed and documented with the required annual MDL evaluation

12.3.1 The LLOQ verification standard shall be prepared at a concentration 0.5-2 times the LLOQ/RL, and be taken through all of the same preparation and clean-up methods as client samples.

12.3.2 The LLOQ verification standard for aqueous matrix shall be prepared using laboratory deionized water and for the solid matrix using clean Ottawa sand. Other clean matrices may be used in addition, for project specific requirements.

12.3.3 The LLOQ shall be verified annually on each instrument used for client sample analysis.

12.3.4 Recovery of each analyte must meet the laboratory established LCS recovery limits + 20%. (For example, if the LCS recovery limits are 70-130%, the LLOQ verification must meet recovery limits of 50-150%.) Once sufficient points have been generated, LLOQ based statistical limits may be used in place of limits based on LCS recovery.

NOTE: The lower recovery limit for the LLOQ can be no lower than 10%.

12.4. Training Requirements

Refer to Eurofins SOP No. ED-GEN-022, (*Training*), for the laboratory's training program.

13.0 Pollution Control

13.1. It is Eurofins's policy to evaluate each method and look for opportunities to minimize waste generated (i.e., examine recycling options, ordering chemicals based on quantity needed, preparation of reagents based on anticipated usage and reagent stability). Employees must abide by the policies in Section 13 of the Corporate Environmental Health and Safety Manual (CW-E-M-001) for "Waste Management and Pollution Prevention."

14.0 Waste Management

14.1. Waste management practices are conducted consistent with all applicable rules and regulations. Excess reagents, samples and method process wastes are disposed of in an accepted manner. Waste description rules and land disposal restrictions are followed. Waste disposal procedures are incorporated by reference to Eurofins Edison SOP No. ED-SPM-008, *Laboratory Waste Disposal*

Practices, current revision. The following waste streams are produced when this method is carried out.

- Laboratory Generated Aqueous Waste (aqueous VOA vials – used and unused). This waste may have a pH of less than 2.0. These vials are collected in satellite accumulation. The vials are then transferred to the waste room. These vials are passed through a vial crusher and the liquid portion is separated from the solid portion. The solid is dumped into the municipal garbage. The liquid is pumped into the neutralization system where it is neutralized to a pH of 6 to 9 with sodium bicarbonate (Seidler Chemical SC-0219-25). When neutralization is complete, the material is transferred to the municipal sewer system**.**

- Expired Standards – The vials are collected in a 1 gallon polyethylene bucket. These vials are then transferred to an open top 55 gallon steel or polyethylene waste drum. These drums are transported to a waste facility for proper disposal.

- Soil Retain Samples - These samples if not flagged in the system for any hazardous constituents are transferred to poly-lined cubic yard boxes. These boxes when full are sent to stabilization or incineration. These materials are sent out as hazardous for lead and chromium

  Teris Profile Number (incineration): 50016710
  Onyx Profile Number: (stabilization) 402535

- Methanol Preserved Samples/Returned Methanol Preservative - Methanol preserved sample vials are collected in satellite accumulation and then transferred to a 55 gallon open top steel waste drum in the waste room. This drum is then removed by a waste vendor for incineration.

  Teris Profile Number: 50016652
  Onyx Profile Number: 282493

15.0 References / Cross-References

15.2. .United States Environmental Protection Agency, “Method 8260D, Volatile Organic Compounds by Gas Chromatography/Mass Spectrometry (GC/MS)”, Test Methods for Evaluating Solid Waste, SW846, Update VI, Revision 4, June 2018.

15.3. United States Environmental Protection Agency, “Method SW8000D: Determinative Chromatographic Separations”, Test Methods for Evaluating Solid Wastes, SW846, Laboratory Manual, Physical/Chemical Methods, Update V, Revision 4, October 2012.

15.4. U.S. EPA. 2003. "Method 5030C (SW-846): Purge-and-Trap for Aqueous Samples," Revision 3. Washington, DC.

15.5. U.S. EPA. 2002. "Method 5035A (SW-846): Closed-System Purge-and-Trap and Extraction for Volatile Organics in Soil and Waste Samples," Draft Revision 1. Washington, DC.

15.6. Eurofins Edison Document No. ED-QA-LQM, *Laboratory Quality Manual*, most current revision.

15.7. Eurofins Document No. CW-E-M-001, Corporate Environmental Health and *Safety Manual,* current revision.

15.8. Eurofins Edison SOP Nos. ED-MSV-001*, Purge and Trap for Aqueous Samples, SW846 Method 5030*, current revision.

15.9. Eurofins Edison ED-MSV-002, *Closed-System Purge-and-Trap and Extraction for Volatile Organics in Soil and Waste Samples, SW846 Method 5035A,* current revision.

15.10. Eurofins Edison SOP No. ED-GCV-001, *Screening for Volatile Organics, Static Headspace with GC FID, SW846 Method 5021*, current revision.

15.11. Eurofins Corporate Quality SOP No. CA-Q-S-001, *Solvent & Acid Lot Testing & Approval*, current revision.

15.12. Eurofins Edison SOP No. ED-GEN-023, *Bulk Solvent Testing and Approval*, current revision.

15.13. Eurofins Edison SOP No. ED-GEN-008, *Standard Operating Procedure for Preparation, Purity and Storage of Reagents and Standards*, current revision

15.14. Eurofins Edison SOP No. ED-SPM-004, *Sample Storage & Handling Procedures for Mitigation of Sample and Laboratory Contamination,* current revision

15.15. Eurofins Edison Work Instruction No. EDS-WI-096, *8260C ICAL Procedure*, current revision.

15.16. Eurofins Edison SOP No. ED-GEN-022, *Training*, current revision.

15.17. Eurofins Edison SOP No. ED-SPM-008, *Laboratory Waste Disposal Practices*, current revision

15.18. Eurofins Edison Work Instruction Document No. EDS-WI-012, *Client Complaint/Corrective Action Form,* current revision.

15.19. Eurofins Corporate Quality Memorandum, CA-Q-QM-002, *GC/MS Tuning Policy*, current revision.

15.20. Eurofins Edison SOP No. ED-GEN-003, *Standard Operating Procedure for Control of Non-Conformances and Corrective Action,* current revision

16.0 Method Modifications:

16.1 Method 8260D requires the BFB tune standard to be analyzed once prior to an ICAL and not daily after that prior to sample analysis. The laboratory will analyze the BFB tune daily, prior to QC and sample analysis. The laboratory will use the tighter criteria from Methods 8260B/8260C for tune evaluation, rather than the criteria suggested in Table 3 of Method 8260D.

17.0 Attachments

N/A

18.0 Revision History

- Revision 9, dated 10/17/2022
    - Updated throughout to Eurofins branding throughout the document.
    - Removed all references to 8260C
    - Section 8.3.1: added requirements for handing aqueous samples from chlorinated sources.
    - Section 9.2.2: updated throughout to detail requirements for Relative Standard Error (RSE) evaluation if ICAL.
    - Section 11.11: added formula for calculation of RSE.
- Revision 8, dated 07/16//2020
    - Updated throughout to include requirements of SW 8260D and 8000D.
    - Updated Table 1 with full current analyte list.
    - Add text to Section 1.1.6 detailing procedures for documenting method variations via NCMs.
    - Section 2.6: clarified text regrading lower than standard RLs.
    - Section 4.4: clarified text regarding trip blanks.
    - Section 7.1.1: revised source and details of organic free water.
    - Section 8.0: added handling and preservation details for various soil and aqueous sample types.
    - Section 9.1.1.4: Added that concentrations allowed in blanks (one half of RL), how blank concentration relates to sample concentration (<1/10) and some guidance on re-analysis when concentration exceeds criteria.
    - Section 9.1.3: added that CCV/LCS can be the same run.
    - Tune verifications as not required for daily CCV updated throughout.
    - Section 9.2.2.1.: Allowance for last calibration standard to be the start of 12-hour clock for samples analyzed after initial calibration. Calculations for verifying peak resolution
    - New Section 9.2.4.2 added: Calibration Point Read Back Criteria
    - Section 11.10: added formula for calculation of Percent Error.
    - Section 12 (Method Performance) updated to include new MDL procedure and annual LLOQ procedure.
    - Updated references in Section 15 as necessary.

- Revision 7, dated 04/01/2020:
    - Updated formatting and branding to Eurofins
    - Sec 7.2.8: Revised Expiration dates based on concentration level and corrected storage requirements.
    - Sec 8.1: Changed Storage Blanks storage period from 1 week to 1-2 weeks.
- Revision 6, dated 01/17/2018:
    - Revised Table 5 and 6: revised to add additional levels plus Benzene and chloroform and to updated concentration level of ICV to current level.
    - 7.2.1 and Table 2: Table in Section 7.2.1 and Table 2 updated to include complete list of standards currently in use as well as to update vendor catalog number for several items.
    - Section 7.2.5: SIM IS/SS mix corrected to reflect lower concentration of IS mix .
    - Section 9.1.2.1.4: SIM MS/MSD preparation revised.
    - Section 9.1.3.2: SIM LCS/LCSD preparation revised.
    - Section 9.2.2.2: additional SIM levels added.
    - Section 9.2.3: SIM CCV level concentration revised to reflect lower concentration.
    - Section 10.2.5: New SIM analysis preparation narrative added
    - Section: 6.1: New instrumentation added.
    - Section: Section: 9.1.4.3: Revised to have any one surrogate out without the need for corrective action. This corrects previous narrative of one surrogate out of two.
- Revision 5, dated 12/11//2015:
    - Revised Table 5: new concentration of low standard (1,4-dioxane only).
- Revision 4, dated 12/08//2014:
    - Section 9.2.4.2.2: Table revised to reflect minimum RF of 0.050 for following compounds: acetone, 2-butanone, 4-methyl-2-pentanone, 2-hexanone.
    - Section 9.2.4.3: added statement ‘for poor performers the range is 50-150%’.
- Revision 3, dated 11/10/2014:
    - Tables 1 and 7: added 1,2,4,5-Trimethylbenzene, 1,4-Diethylbenzene, Butadiene, 1,4-Difluorobenzene, 1-Chlorohexane, Freon 114, Freon 123a, Isooctane, 4-Ethyltoluene, t-Amyl Alcohol, Chlorofluoroethylene to list of target compounds and list of standard sources.

- Section 2.5: added chloroform, vinyl chloride and benzene to the list of SIM analytes addressed in this section.
- Section 2.6: revised the concentration of the low ketone standard to 2.5 ug/l.
- 7.2.1 and Table 2: Table in Section 7.2.1 and Table 2 updated to include complete list of standards currently in use as well as to update vendor catalog number for several items. All standards prep tables revised to reflect current standard prep instructions.
- Section 8. Preservation by TSP and holding time is added.
- Section 9.1.2.1: updated source of standards used in various spiking solutions.
- Section 9.1.3: LCS/MS/MSD. Preparation tables now indicate using calibration mix and not the second source mix.
- Sections 9.1.4.3 and 9.1.1 : Revised to indicate that we are now spiking with 4 surrogates instead of the method required 3. One surrogate is now allowed to be out of limit criteria for either 1,2-Dichloroethane-d4 and Dibromofluoromethane.
- Section 9.2.2: Chloroform, Vinyl Chloride and Benzene added as SIM compounds.
- Section 9.2.4.2.3.1. A list of 'poor performing compounds' is added with a ICAL RSD criteria of 50%.
- Section 9.2.4.3: now specifies that up to 10% of the compounds are allowed to exceed the 70-130% ICV recovery criteria as long as their recoveries are within 65-135%..
- Section 9.2.4.4.1.6: Added the following to the first sentence: '…or 50%D for the poor performing compounds'.
- Section 10.1.1.2: updated masses/dwell time for Group 1 under SIM Parameters.
- Throughout document as appropriate: Replaced references to Target with references to CHROM
- Added Section 10.5.5: "The data processing service from Chrom queries LIMS for the sample processing parameters."

- Revision 2, dated 11/04/2013:
  - Tables 1 and 7: added methyl acrylate, 1-methylnaphthalene and 2-methylnapthalene.
- Revision 1, dated 09/16/2011:

- Tables 1 and 7: added cyclopentene, 2-chloro-1,3-butadiene, methacrylonitrile, propionitrile, ethyl methacrylate, 2-nitropropane, indan and isobutyl alcohol to list of target compounds and list of standards sources.

- Section 7.2.1 and Table 2: Table in Section 7.2.1 and Table 2 updated to include complete list of standards currently in use as well as to update vendor catalog number for several items.

- Table 3: Initial Calibration Standards Preparation: is now split into three tables to include aqueous low level analysis.

- Table 5: added following footnote:

  Levels 1 and 2 respectively are prepared in 500ml and 100ml final volumes
  [1]This level is also used as the Continuing Calibration Verification.

- Revision 0, dated 02/15/2011: New

Table 2: Working Standards Preparation

Target Compound Standard Name	Lab Name	Vendor	Cat. #	Vol. Std. Added	Conc. of Stock Std.	Concentration of Standard	Final Vol/ Total vol of MeOH
Gas Mix Hi	Gas (Hi)	Restek	567645	5ml mL	2000 ppm	500 ppm	20mL 15mL TV/M
Gas Mix Li	Gas (Li)	Restek	567645	500 uL	2000 ppm	50 ppm	20mL 19.5mL TV/M
8260Mix 1	Mix 1 (Hi)	Restek	567641 567646 567642 568022	2.5ml 2.5 ml 2.5 ml 2.5 ml	2000 ppm	500 ppm	10ml
8260 combined	Mix 1 (Li)	Restek	567641 567646 567642 568022 567643 568018 568713 568722 568723	1.0ml 1.0ml 1.0ml 1.0ml 1.0ml 1.0ml 1.0ml 1.0ml 1.0ml	2000 ppm	50 ppm	40ml 31ml TV/M
Acrolein	AC	Restek	82402	1.0ml	20000 ppm	500 ppm	40ml 39ml TV/M
8260 Mix 2	Mix 2 (Hi)	Restek	567643 568722 568019-fl 568713-fl	2.5ml 2.5 ml 2.5 ml 2.5 ml	2000 ppm	500 ppm	10mL
8260 Mix 3	Mix 3 (Hi)	Restek	568723 568021-fl	2.5ml 2.5ml	2000 ppm	500 ppm	10ml 5ml TV/M
1,4-Dioxane	1,4-Dioxane	Supelco	360481	483.6ul	Neat	50000 ppm	10ml/9.52TVM
1,4-Dioxane	1,4-Dioxane	Supelco	NA	100ul	50000 ppm	500 ppm	10ml/9.90TVM
Propenes*	Propenes	Supelco	21240202	NA	1000/2000 ppm	NA	NA
Propenes*	Propenes	Supelco	21240202	1ml	1000/2000 ppm	50 ppm (varied)	20ml/ 19ml
Gas SS	Gas SS	Restek	567645.sec	1ml	2000ppm	50 ppm	40ml 39ml/TV/M
8260 Mix 1 SIM	8260 Mix 1 SIM	Supelco	5-02111	50 ul	2000ppm	10 ppm	10ml 9.95 TV/M
1,4-Dioxane SIM	1,4-Dioxane	Supelco	NA	100 ul	50000 ppm	500 ppm	10ml/9.90TVM

Table 2: Working Standards Preparation

Target Compound Standard Name	Lab Name	Vendor	Cat. #	Vol. Std. Added	Conc. of Stock Std.	Concentration of Standard	Final Vol/ Total vol of MeOH
8260 SS	8260 SS	Restek	567641.sec 567646.sec 567642.sec 568022- sl 567643.sec 568019- sl 568713- sl 568722.sec 568723.sec 568021- sl	1 ml 1 ml 1 ml 1 ml 1 ml 1 ml 1 ml 1 ml 1 ml 1 ml	2000 ppm	50 ppm	40 ml 30 ml TV/M
Acrolein SS	AC SS	Restek	568720.sec	1 ml	20000 ppm	500 ppm	40 ml 39 ml TV/M
Propenes SS	Propenes SS	Supelco		1 ml	1000/2000 ppm	50/100 ppm	40 ml 39 ml TV/M
8260Mix 1 SIM SS	SIM MIX1 SS	Supelco	5S-02111	50ul	2000 ppm	10 ppm	10ml 9.95 TV/M
Benzene/ Chloroform	Ben/chl	Absolute	70025/ 70076	100ul each	1000ppm	10ppm	10ml 9.90 TV/M
1,4-Dioxane (SS)	1,4-Dioxane	Absolute	70373	1ml	1000 ppm	500 ppm	2ml/1ml TV/M

Asterisk (*) indicates a custom standard mix.

Table 3: Initial Calibration Standards Preparation, Low Level Soil

Standard Solution	Final Volume Reagent Water (ml)	Volume of Standard Added to Reagent Water (ul)					
		1ppb *	5ppb*	20ppb[1]	50ppb	200ppb	500ppb
Gas Mix (50ppm)	5	-	-	2.0	5	-	-
	50	1.0	5.0			-	-
Gas Mix (500ppm)	5	-	-	-		2.0	5.0
		-	-	-			
Mix 1 (combined) (50ppm)	5	-	-	2.0	5	-	-
	50	1.0	5.0			-	-
Mix 1 (Hi) (500ppm)	5	-	-	-	-	2.0	5.0
		-	-	-	-	-	-
Freon Mix							
AC (500ppm)	5	-	-	3.0	4.0	5.0	6.0
	50	10	20			-	-
Mix 2 (Hi) (500ppm)	5	-	-	-	-	2.0	5.0
		-	-	-	-		
Mix 3 (500ppm)	5					2.0	5
Propenes (50ppm)	-	-	-	-	-	-	-
	50	10.0	20.0		-	-	-
Propenes (Hi)(500ppm)	5	-	-	2.0	5.0	20	50
	-	-	-	-	-	-	-

[1]**This level is also used as the Continuing Calibration Verification.**

Table 3a: Initial Calibration Standards Preparation, Aqueous (LOW LEVEL)

Standard Solution	Volume of Standard Added to Reagent Water (ul)						
	0.5ppb*	1ppb*	5ppb*	20ppb[1]	50ppb	200ppb	500ppb
Gas Mix (500ppm)	0.5	1	1	2	5	20	50
Mix 1 (Hi) (500ppm)	0.5	1	1	2	5	20	50
Mix 2 (Hi) (500ppm)	0.5	1	1	2	5	20	50
Mix 3 (varied)	0.5	1	1	2	5	20	50
AC (500ppm)	2	4	4	4	10	20	40
1,4-Dioxane (500ppm)	15	30	-	-	-	-	-
Freons mix	0.5	1	1	2	5	20	50
Propenes (1000/2000ppm)	0.5	0.5	0.5	1	2.5	10	25
Methanol Compensate	3000	2800	610	300	280	190	0
Final vol. (reagent water)	500ml	500 ml	100ml	50 ml	50ml	50ml	50ml

[1]This level is also used as the Continuing Calibration Verification.

Table 3b: Initial Calibration Standards Preparation, Aqueous

Standard Solution	Volume of Standard Added to Reagent Water (ul)					
	1.0ppb*	5ppb*	20ppb[1]	50ppb	200ppb	500ppb
Gas Mix (500ppm)	1	1	2	5	20	50
Mix 1 (Hi) (500ppm)	1	1	2	5	20	50
Mix 2 (Hi) (500ppm)	1	1	2	5	20	50
Mix 3 (varied)	1	1	2	5	20	50
AC (500ppm)	4	4	4	10	20	40
1,4-Dioxane (500ppm)	30	-	-	-	-	-
Freons Mix	1	1	2	5	20	50
Propenes (1000/2000ppm)	0.25	0.5	1	2.5	10	25
Methanol Compensate	2800	610	300	280	190	0
Final vol. (reagent water)	500 ml	100ml	50 ml	50ml	50ml	50ml

[1]This level is also used as the Continuing Calibration Verification.

Table 4 : ICV Standard Preparation, Low Level Soil

Standard Solution	Concentration	Volume of Standard Added to 5.0 ml of Reagent Water (ul)	Final Concentration (ug/L)
GAS SS (Separate lot)	50ppm	2	20
8260 SS (Separate lot)	50ppm (+varied)	2	20
AC SS (separate lot)	500ppm	3	300
Freon SS (Separate lot)	50ppm	2	20
Propenes SS(separate lot)	50ppm (varied)	2	20 (varied)

Table 4a: ICV Standard Preparation, Aqueous

Standard Solution	Concentration	Volume of Standard Added to 50 ml of Reagent Water (ul)	Final Concentration (ug/L)
GAS SS (Separate lot)	50ppm	20	20
8260 SS (Separate lot)	5000ppm (varied)	20	20
AC SS (separate lot)	500ppm	4	400
Freons SS (Separate lot)	50ppm	20	20
Propenes (second source)	50ppm (varied)	20	20 (varied)

Table 5: SIM Initial Calibration Standards Preparation

Standard Solutions	Volume Standard Solution Added to Reagent Water (Final Concentration)							
8260 Mix 1 SIM (10ppm)	1 ul (0.02 ppb)	2 ul (0.04 ppb)	1 ul (0.05 ppb)	1 ul (0.1 ppb)	1 ul (0.2 ppb)	2.5 ul (0.5 ppb)	5 ul (1 .0 ppb)	10 ul (2.0 ppb)
1,4-Dioxane (500ppm)	4 ul (0.4 ppb)	2 ul (1.0 ppb)	1 ul (5.0 ppb)	1 ul (10 ppb)	1 ul (20 ppb)	2.5 ul (30 ppb)	5 ul (40 ppb)	10 ul (50 ppb)
SIM (ben/chl) 10ppm	1 ul (0.02 ppb)	1 ul (0.02 ppb)	1 ul (0.05 ppb	1 ul (0.1 ppb)	1 ul (0.2 ppb)	2.5 ul (0.5 ppb)	5 ul (1.0 ppb)	10 ul (2.0 ppb)
Final Vol. (reagent water)	**500ml**	**500ml**	**200ml**	**100ml**	**50ml**	**50ml**	**50ml**	**50ml**

levels 1 and 2 are respectively prepared in 500ml and 100ml final volumes
[1]**This level is also used as the Continuing Calibration Verification.**

Table 6 : SIM ICV Standard Preparation

Standard Solution	Concentration	Volume of Standard Added to 200 ml of Reagent Water (ul)	Final Concentration (ug/L)
SIM MIX1 SS (Second source)	10ppm	1	0.05
1,4-Dioxane SS	50ppm	20	5

TABLE 7
Characteristic Ions of Volatile Organic Compounds

Parameter	Primary ion	Secondary ion
1,1,1-Trichloroethane	97	99,117,119
1,1,2,2-Tetrachloroethane	83	85,131,133,166
1,1,2-Trichloroethane	97	83,85,99,132,134
1,1-Dichloroethane	63	65,83,85,98,100
1,1-Dichloroethene	96	61,98
1,1-Dichloropropene	75	110. 77
1,2,3-Trichlorobenzene	180	182
1,2,3-Trichloropropane	110	75
1,2,4-Trichlorobenzene	180	182, 145
1,2,4-Trimethylbenzene	105	120
1,2-Dibromo-3-Chloropropane	75	155, 157
1,2-Dibromomethane	107	109
1,2-Dichloroethane	62	64,100,98
1,2-Dichloroethene	96	61,98
1,2-Dichloropropane	63	65,114
1,2-Dichlorotrifluoroethene	67	117
1,2-Difluorotetrachloroethene	101	103, 167
1,3,5-Trimethylbenzene	105	120
1,3-Dichlorobenzene	146	148, 111
1,4-Dichlorobenzene	146	148, 111
1,4-Dioxane	88	58
1-Chloropropane	63	78
1-Methylnaphthalene	142	141
1-Propene	41	42
2,2-Dichloropropane	77	97

TABLE 7 Characteristic Ions of Volatile Organic Compounds		
2,4,4-trimethyl-1-pentene	41	57, 97
2-Butanone	72	57
2-Chloroethyl vinyl ether	63	65, 106
2-Chloropropane	78	63
2-Chlorotoluene	91	126
2-Chloro-1,3-butadiene	88	53
2-Hexanone	43	58,100
2-Methylnaphthalene	142	141, 115
2-Nitropropane	39	42, 44
2-Octane	43	58
2-Octanol	45	55
4-Chlorotoluene	91	126
4-Methyl-2-Pentanone	43	58,100
Methacrylonitrile	67	41
Acetone	43	58
Acetonitrile	39	40, 41
Acrolein	56	55
Acrylonitrile	53	52
Allyl Alcohol	57	40, 39
Allyl Chloride	76	41
Amyl Acetate	43	70, 61
Benzene	78	--
Benzyl Chloride	91	126, 65
Bromobenzene	156	77, 158
Bromochloromethane	129	49, 130
Bromodichloromethane	83	85
Bromoform	173	171,175,
Bromomethane	94	96
Butyl Acetate	73	56, 43
Butyl Acrylate	73	56, 55
Butyl methacrylate	87	69
Camphene	93	121
Camphor	95	81
Carbon disulfide	76	78
Carbon tetrachloride	117	119,121
Chlorobenzene	112	114
Chloroethane	64	66
Chloroform	83	85
Chloromethane	50	52
Chlortrifluoroethene	116	118
cis-1,3-Dichloropropene	75	77

TABLE 7 Characteristic Ions of Volatile Organic Compounds		
Cyclohexane	56	84, 69
Cyclopentene	67	68, 68, 53
Dibromochloromethane	129	208,206
Dibromomethane	93	95, 174
Dichlorodifluoromethane	85	87
Dimethylnaphthalene (total)	141	156, 155
Epichlorohydrin	57	62, 49
Ethanol	46	45
Ethyl Acetate	70	61, 43
Ethyl Acrylate	55	56
Ethyl Ether	59	74, 75
Ethylbenzene	106	91,
Ethyl methacrylate	69	41, 99
Freon TF	101	103, 151, 85
Hexachlorobutadiene	225	223
Hexane	56	57, 86
Indan	117	118, 58
Iodomethane (methyl iodide)	142	127
Isobutyl Alcohol (Isobutanol)	43	41, 42
Isoprene	67	53, 59
Isopropanol	45	59
Isopropyl Acetate	43	61, 87
Isopropyl Ether (DIPE)	45	87
Isopropylbenzene	105	120
Methyl Acetate	43	74
Methyl Acrylate	55	85, 42
Methyl cyclohexane	83	55, 98
Methyl Methacrylate	100	69
Methyl tert-butyl ether (MTBE)	73	57
Methylene chloride	84	49,51,86
Methylnaphthalene (total)	142	141, 115
Naphthalene	128	--
n-Butanol	56	41, 43
n-Butylbenzene	91	92, 134
n-Heptane	57	43, 71
n-Pentane	72	57
N-Propanol	60	59
n-Propylbenzene	91	120
P-Isopropyltoluene`	119	134, 91
Propyl Acetate	43	61, 73
Propionitrile	54	52, 54

TABLE 7 Characteristic Ions of Volatile Organic Compounds		
sec-Butylbenzene	105	134
Styrene	104	78,103
Tert-Amyl Methyl Ether	73	55, 87
Tert-butyl Alcohol	59	--
Tert-Butyl Ethyl Ether	59	87
Tert-Butylbenzene	119	91, 134
Tetrachloroethene	164	129,131,166
Tetrahydrofuran	42	72, 71
Toluene	92	91
Total Xylenes	106	91
trans,-1,3-Dichloropropene	75	77
Trans-1,4-dichloro-2-butene	53	75
Trichloroethene	130	95,97,132
Trichlorofluoromethane	101	103
Vinyl acetate	43	86
Dichlorofluoromethane	67	69
Chlorotrifluoroethene	116	118
1,2-tetrachlorodifluoroethane	101	103,167
1,2-Dichlorotrifluoroethane	67	117
Vinyl chloride	62	64
Isooctane	57	41, 56
1- Chlorohexane	91	93, 55, 56
1,2,4,5-Tetramethylbenzene	119	134, 91
4-EthylToluene	105	120, 77
Chlorotrifluoroethylene	66	116,118,85
Freon 114	85	87,135,137
t-Amyl Alcohol	59	55, 73, 43
1,4-Difluorobenzene	114	63
1,4-Diethylbenzene	119	105,134
Freon 123a	67	69, 117, 119
Butadiene	54	53, 39
4-Bromofluorobenzene (sur)	95	174,176
1,2-Dichloroethane-d4 (sur)	65	102, 104
Toluene-d8 (sur)	98	70,100
Fluorobenzene (istd)	96	77
Chlorobenzene-d5 (istd)	117	82,119
1,4-Dichlorobenzene-d4 (istd)	152	115,150

Title: Mercury Analysis for Water and Wastewater using EPA 245.1 and SW846 7470A; Mercury in Drinking Water using EPA 245.1; Leeman Mercury Analyzer (Cold Vapor Technique)

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Approvals (Signature/Date):

Laura Demone	08/11/22	Donald Evans	08/11/22
Laura Demone Department Manager	Date	Donald Evans Operations Manager	Date
	08/11/22		08/11/22
Carl Armbruster Quality Assurance Manager	Date	Mark Acierno Laboratory Director	Date
Dan Helfrich Healthy & Safety Manager Coordinator	08/11/22 Date		

1.0 Scope and Application

1.1. Analytes, Matrix(s), and Reporting Limits

EPA Method 245.1 and SW846 Method 7470A are applicable to the determination of mercury in water matrices. Mercury may be found in water in both inorganic and organic forms. Organomercury compounds must first be broken down to respond to the cold vapor atomic absorption technique.

The typical detection limit using a 30 ml sample size is 0.2ug/L Hg.

On occasion clients may request modifications to this SOP. These modifications are handled following the procedures outlined in Sections 7 (Review of Work Request) and 19 (Test Methods and Method Validation) of TestAmerica Edison's Quality Assurance Manual (ED-QA-LQM).

2.0 Summary of Method

A digested sample is analyzed using cold vapor atomic absorption. The mercury is reduced to the elemental state and aerated from solution in a closed system. The mercury vapor passes through a cell positioned in the light path of an atomic absorption spectrophotometer. Absorbance is measured as a function of mercury concentration.

3.0 Definitions

For a complete list of definitions refer to Appendix 2 in the most current revision of TestAmerica Edison's Quality Assurance Manual (ED-QA-LQM).

4.0 Interferences

4.1 The addition of potassium persulfate during the digestion step can eliminate the possible interference from sulfide in the sample without affecting the recovery of inorganic mercury.

4.2 Copper may also be a potential interference although no effect has been observed for samples containing up to 10 mg/l total copper.

4.3 Samples that contain high levels of chloride have a potential to interfere due to a reaction that takes place during the oxidation step. During this step chloride is converted to free chlorine which absorbs light at 253.7 nm. The analyst must not allow the chlorine to be swept into the optical cell. The possibility of the chlorine interfering with the analysis can be minimized by using an excess of up to 7.5 ml hydroxylamine sulfate.

5.0 Safety

Employees must abide by the policies and procedures in the Corporate Environmental Health and Safety Manual (CW-E-M-001) and this document. This procedure may involve hazardous material, operations and equipment. This SOP does not purport to address all of the safety problems associated with its use. It is the responsibility of the user of the method to follow appropriate safety, waste disposal and health practices under the assumption that all samples and reagents are potentially hazardous. Safety glasses, gloves, lab coats and closed-toe, nonabsorbent shoes are a minimum.

5.1 Specific Safety Concerns or Requirements

Samples that contain high concentrations of carbonates or organic material or samples that are at elevated pH can react violently when acids are added.

5.2 Primary Materials Used

The following is a list of the materials used in this method, which have a serious or significant hazard rating. **Note: This list does not include all materials used in the method. The table contains a summary of the primary hazards listed in the MSDS for each of the materials listed in the table.** A complete list of materials used in the method can be found in the reagents and materials section. Employees must review the information in the MSDS for each material before using it for the first time or when there are major changes to the MSDS.

Material (1)	Hazards	Exposure Limit (2)	Signs and symptoms of exposure
a)Mercury b)(1,000 PPM in Reagent)	Oxidizer Corrosive Poison	0.1 mg/M^3 Ceiling (Mercury Compounds)	Extremely toxic. Causes irritation to the respiratory tract. Causes irritation. Symptoms include redness and pain. May cause burns. May cause sensitization. Can be absorbed through the skin with symptoms to parallel ingestion. May affect the central nervous system. Causes irritation and burns to eyes. Symptoms include redness, pain, and blurred vision; may cause serious and permanent eye damage.
Sulfuric Acid	Corrosive Oxidizer Dehydrator Poison	1 mg/M^3-TWA	Inhalation produces damaging effects on the mucous membranes and upper respiratory tract. Symptoms may include irritation of the nose and throat, and labored breathing. Symptoms of redness, pain, and severe burn can occur. Contact can cause blurred vision, redness, pain and severe tissue burns. Can cause blindness.

Material (1)	Hazards	Exposure Limit (2)	Signs and symptoms of exposure
Nitric Acid	Corrosive Oxidizer Poison	2 ppm-TWA 4 ppm-STEL	Nitric acid is extremely hazardous; it is corrosive, reactive, an oxidizer, and a poison. Inhalation of vapors can cause breathing difficulties and lead to pneumonia and pulmonary edema, which may be fatal. Other symptoms may include coughing, choking, and irritation of the nose, throat, and respiratory tract. Can cause redness, pain, and severe skin burns. Concentrated solutions cause deep ulcers and stain skin a yellow or yellow-brown color. Vapors are irritating and may cause damage to the eyes. Contact may cause severe burns and permanent eye damage.
Hydrochloric Acid	Corrosive Poison	5 PPM-Ceiling	Inhalation of vapors can cause coughing, choking, inflammation of the nose, throat, and upper respiratory tract, and in severe cases, pulmonary edema, circulatory failure, and death. Can cause redness, pain, and severe skin burns. Vapors are irritating and may cause damage to the eyes. Contact may cause severe burns and permanent eye damage.
Potassium Permanganate	Oxidizer	5 Mg/M^3 for Mn Compounds	Causes irritation to the respiratory tract. Symptoms may include coughing, shortness of breath. Dry crystals and concentrated solutions are caustic causing redness, pain, severe burns, brown stains in the contact area and possible hardening of outer skin layer. Diluted solutions are only mildly irritating to the skin. Eye contact with crystals (dusts) and concentrated solutions causes severe irritation, redness, and blurred vision and can cause severe damage, possibly permanent.
1 – Always add acid to water to prevent violent reactions.			
2 – Exposure limit refers to the OSHA regulatory exposure limit.			

6.0 Equipment and Supplies

6.1 Instrumentation

6.1.1. Leeman Laboratories Inc. Hydra II AA Automated Hg Analyzer

6.1.2. Computer and Monitor with Leeman Envoy software

6.1.3. Block digestor (Environmental Express or SCP Science): Adjustable and capable of maintaining a temperature of 90 -95°C.

6.2 Supplies

6.2.1. 50 ml Hot Block Digestion Cups

6.2.2. 100 ml graduated cylinder

6.2.3. Eppendorf Pipettes and tips in various sizes

6.2.4. 100 ml volumetric flasks

6.2.5. 15 ml sample cups

6.2.6. 10 liter carboy container

6.2.7. Pump tubing:

- Sample, viton, blue tab
- Reductant, red tab
- Drain, blue tab
- Rinse, black tab

6.2.8. Drying Tube – Purchased pre-packed with Magnesium Perchlorate from Leeman Labs. Located prior to the optical cell.

6.2.9. Nitrogen or Argon supply - capable of producing 80 PSI.

7. Reagents and Standards

7.1. Reagents

Storage requirements: store at room temperature

Life of Reagent: - Concentrated acids: refer to manufacturer's instructions
- Laboratory prepared reagents and diluted acids: one year

Document prepared reagents in the Reagent module located in the TestAmerica Laboratory System (TALS).

7.1.1 Sulfuric acid - Concentrated (Trace Grade or Equivalent); store at room temperature; for stability information, refer to manufacturer's instructions.

7.1.2 Nitric acid - Concentrated (Trace Grade or Equivalent)

7.1.3 Hydrochloric acid-Concentrated (Trace Grade or Equivalent)

7.1.4 Potassium Permanganate (ACS Grade); for stability information, refer to manufacturer's instructions.

7.1.5 Sodium Chloride (analytical reagent grade); for stability information, refer to manufacturer's instructions.

7.1.6 Hydroxylamine Hydrochloride (ACS Grade); for stability information, refer to manufacturer's instructions.

7.1.7 Stannous Chloride (ACS Grade); for stability information, refer to manufacturer's instructions.

7.1.8 Potassium Persulfate (ACS Grade); for stability information, refer to manufacturer's instructions.

7.1.9 Deionized water - 18 megohm minimum

7.1.10 10% Hydrochloric Acid- Add approximately 5 liters of deionized water into an 8 liter carboy container. Cautiously add 800 ml of concentrated HCl and bring the final volume up to 8 liters with deionized water.

7.1.11 10% Stannous chloride solution - Add 50 g of $SnCl_2$ to 500 ml 10% HCl solution.

7.1.12 Sodium chloride/Hydroxylamine Hydrochloride solution - Dissolve 120 g of NaCl and 120 g of hydroxylamine hydrochloride in deionized water and dilute to 1 liter using deionized water.

7.1.13 Potassium Permanganate ($KMnO_4$) 5% solution w/v - Dissolve 100 g of $KMnO_4$ in deionized water and dilute to 2 liters using deionized water.

7.1.14 0.15% Nitric Acid- Add approximately 5 liters of deionized water into an 8 liter carboy container. Cautiously add 12mL of concentrated HNO_3 and bring the final volume up to 8 liters with deionized water.

7.1.15 Potassium Persulfate ($K_2O_8S_2$) 5% solution w/v: Dissolve 50 g of potassium persulfate in 1 liter of deionized water.

7.2 Standards

Storage requirements: all standards are stored at room temperature
Shelf-life: Stock standards – refer to manufacturer's instructions
Intermediate standards – made fresh daily
Working standards – made fresh daily

(Note: expiration date must not go beyond the expiration date of the source stock).

Concentration: see Attachment 1 for example certificates of analysis (COA) for all of the standards mixes listed below. The COA lists the manufacturer's part number, certified concentration and shelf life.

Document standard preparation in TALS (see Section 11.5.2).

7.2.1 Stock Mercury Calibration (10 ppm Hg) - Purchase from SCP Science; store at room temperature; for stability information, refer to manufacturer's instructions.

7.2.2. Stock Mercury Quality Control Standard (10 ppm Hg) - Purchase from Inorganic Ventures; store at room temperature; for stability information, refer to manufacturer's instructions. This stock standard must be purchased from a second source vendor.

7.2.3. Intermediate Calibration Standard (DCAL-Int): Dilute 1 ml of Stock Mercury Calibration (Sec 7.2.1) to 100 ml with 0.15% HNO_3. The resulting solution will contain 100ppb Hg.

7.2.4. Intermediate Quality Control Standard (DQCS-Int): Dilute 1 ml of Stock Mercury Calibration Verification Standard (Sec 7.2.2) solution to 100 ml with 0.15% HNO_3. The resulting solution will contain 100ppb Hg.

7.2.5. Calibration Standard Preparation: Use six 50 ml hot block digestion cups to prepare the standards. Add small portion of 0.15% HNO_3 to each cup. Working in increasing order, spike the appropriate cup with 0.0, 0.06, 0.3, 0.6, 1.5, and 3.0 ml of working solution DCAL-Int (Sec 7.2.3). Bring to final volume of 30 ml and mix thoroughly. The corresponding concentrations are 0.0ppb, 0.2ppb, 1.0ppb, 2.0ppb, 5.0ppb, and 10.0ppb mercury respectively. For drinking water analysis, the 2.0ppb standard is also analyzed as the Maximum Contaminant Level (MCL) standard

7.2.6. Quality Control Standard (QCS) Preparation: Add a small portion of 0.15% HNO_3 to a 30 ml hot block digestion cup and spike 1.5 ml of DQCS-Int (Sec 7.2.4). Bring up to final volume and mix thoroughly. The resulting solution will contain 5.0 ppb of Hg.

7.2.7. Initial Calibration Verification (ICV) standard: Add a small portion of 0.15% HNO3 to a 30 ml hot block digestion cup and spike 1.5 ml of DQCS-Int (Sec 7.2.4). Bring up to final volume and mix thoroughly. The resulting solution will contain 5.0 ppb of Hg.

7.2.8. Continuing Calibration Verification (CCV) standard 5.0ppb: The 5.0 ppb

prepared from the calibration standards (Sec 7.2.5) is used as the Continuing Calibration Verification standard (CCV).

7.2.9. Maximum Contaminant Level (MCL) standard 2.0 ppb *for drinking water analysis:* The 2.0ppb standard prepared from the calibration standards (Sec 7.2.5) is analyzed as the Maximum Contaminant Level (MCL) standard.

7.2.10. Reporting limit check standard (CRI) 0.2 ppb: The 0.2ppb standard prepared from the calibration standards (Sec 7.2.5) is analyzed as the CRI standard. This standard is being analyzed as part of some client's special projects.

8.0 Sample Collection, Preservation, Shipment and Storage

Sample container, preservation techniques and holding times may vary and are dependent on sample matrix, method of choice, regulatory compliance, and/or specific contract or client requests. Listed below are the holding times and the references that include preservation requirements.

Matrix	Sample Container	Min. Sample Size	Preservation	Holding Time [2]	Reference
Waters	P. FP, G[1]	250 ml	HNO_3, pH < 2 prior to shipment; if not, acidify upon receipt in lab; Cool 4±2°C	28 Days	40 CFR Part 136.3

[1] Polyethylene, fluoropolymer, glass
[2] Inclusive of digestion and analysis
[3] Acid preservation may be omitted for shipping; however, acid must be added upon receipt in the lab. Following acidification, mix the sample and hold for at least 16 hours for method 245.1 drinking water and 24 hours for non-potable water. Just prior to digestion or direct analysis, verify pH<2. If pH≥2, repeat steps (i.e., add acid, hold for 24hrs, verify ph<2).

9.0 Quality Control

9.1. Sample QC - The following quality control samples are prepared with each batch of samples.

Quality Controls	Frequency	Control Limit
Method Blank (MB)	1 in 20 or fewer samples	**245.1**: < RL or; <10%sample concentration (whichever is greater) **7470A**: < RL; **or;** < 5% of the reg limit; **or,** < 5% of the measured sample concentration (whichever is greater)
Laboratory Control Sample (LCS)	1 in 20 or fewer samples	**245.1**: 85-115% **7470A**: 80-120%
Matrix Duplicate (DUP)[1]	1 in 20 or fewer samples	20% RPD
Matrix Spike (MS)[1]	**245.1**: 1, if 10 samples or less; 2 if 11-20 samples **7470A**: 1 in 20 or fewer samples	**245.1**: 70-130% **7470A**: 75-125%
Matrix Spike Duplicate (MSD)	When requested by client	**245.1**: 70-130% **7470A**: 75-125% **For both 245.1** & **7470A**: If MS and MSD are both ≥ 5X CRDL, then 20% RPD. If MS and MSD are less than the CRDL, the RPD is not calculated; otherwise ±CRDL.
Serial Dilution (SD) for 7470A	1 in 20 or fewer samples	±10%

[1] The sample for DUP and MS are randomly selected, unless specifically requested by a client. Use the same environmental sample for the matrix spike and matrix duplicate sample whenever possible. If insufficient sample amount is available, another environmental sample may be used for the duplicate sample.

9.1.1 **Method Blank:** One laboratory method/preparation blank will be analyzed with each batch of samples prepared together (not to exceed 20 samples). Preparation blank is used to identify possible contamination during acid digestion. If the Mercury concentration in the MB is above the specified control limit, the batch must be prepared again and the samples reanalyzed.

- For 7470A, results must be less than RL, 5% of the regulatory limit for that analyte, or 5% of the measured concentration in the sample, whichever is greater.
- For 245.1, results must be less than RL or less than 10% of the determined Mercury concentration in the sample, whichever is greater.

9.1.2 **Laboratory Control Sample (LCS):** A laboratory control sample must be analyzed with each batch of samples digested. A blank is spiked with 0.1

ug of mercury (0.30 ml of standard DCAL-Int). This is equivalent to 1.0 ppb Hg if a 30 ml portion of sample is digested. Results of the aqueous LCS must fall within ±15% of the true value for Method 245.1 and ±20% for Method 7470A.

9.1.3 **Matrix Duplicate (DUP):** A duplicate is analyzed for each batch of samples digested. If original sample and duplicate are both ≥ CRDL, then 20% RPD. If original sample and duplicate are less than the CRDL, the RPD is not calculated; otherwise, ±CRDL.

9.1.4 **Matrix Spike (MS):** A matrix spike is prepared and analyzed for each batch of samples. A portion of sample is spiked with 0.1 ug of mercury (0.30 ml of standard DCAL-Int). This is equivalent to 1.0 ppb Hg if a 30 ml portion of sample is digested. A recovery of 70-130% for Method 245.1 and 75-125% for Method 7470A is required.

9.1.5 **Matrix Spike Duplicate (MSD):** When requested by the client, a matrix spike duplicate is prepared and analyzed for each batch of samples. A portion of sample is spiked with 0.1 ug of mercury (0.30 ml of standard DCAL-Int). This is equivalent to 1.0 ppb Hg if a 30 ml portion of sample is digested. A recovery of 70-130% for Method 245.1 and 75-125% for Method 7470A is required. For both 245.1 & 7470A: if original sample and duplicate are both ≥ CRDL, then 20% RPD. If original sample and duplicate are less than the CRDL, the RPD is not calculated; otherwise, ±CRDL.

9.1.6 **Serial Dilution (SD):** For method 7470A, a five fold serial dilution must be performed on one sample per batch. The sample should contain a sufficiently high concentration; minimally a factor of 25 times above the estimated detection limit. Dilute the sample by a minimum of five fold (1+4) and reanalyze. Results must agree within 10% of the original determination. If not, a chemical or physical effect should be suspected.

9.2 Instrument QC

9.2.1. **Initial Calibration Verification (ICV):** Initial calibration is verified after calibration. The ICV solution should be prepared using a second source vendor; see section 7.2.7 for preparation instructions. Use a concentration of mercury at the midpoint of the calibration range (5.0ppb). The Cal standard containing 5ppb is analyzed as the ICV; see Sec 7.2.7 for preparation instructions. For 245.1, the results must not differ from the true value by more than 5%. For 7470A, the results must be within 10% of the true value. If it's outside of the acceptable limits, terminate the analysis, correct the problem and recalibrate the instrument.

9.2.2. **Continuing Calibration Verification (CCV):** Calibration verification is performed after the calibration, after every 10 samples, and at the end of the run. The CCV solution should be prepared from the same CAL

standard as used to prepare the calibration solutions. Use a concentration of mercury at the midpoint of the calibration range (5.0ppb). The Cal standard containing 5ppb is analyzed as the CCV.

For 245.1 and 7470A, CCV must not differ from the true value by more than 10%. If it does, stop the analysis and recalibrate. Re-analyze the previous ten samples following the last good calibration verification.

9.2.3. **Initial and Continuing Calibration Blank (ICB/CCB):** ICB and CCB must be analyzed after the calibration curve, every 10 samples and at the end of the analytical run. For methods 245.1 and 7470A, the absolute value of the calibration verification blank must not exceed the reporting limit. If it does, terminate the analysis, correct the problem, recalibrate and reanalyze the samples following the last good CCB. The calibration verification blank is the same blank solution as used for the calibration blank.

9.2.4. **Maximum Contaminant Level (MCL):** For drinking water analysis, one MCL standard shall be analyzed per calibration. The 2.0ppb standard prepared from the calibration standards (Sec 7.2.5) is analyzed as the Maximum Contaminant Level (MCL) standard. The result must be within 50% of the true value. If it's outside of the acceptable limit, terminate the analysis, correct the problem, and recalibrate the instrument.

9.2.5. **Quality Control Standard (QCS):** The calibration is verified after calibration using a second source vendor; see Sec 7.2.6 for preparation instructions. For 245.1 and 7470A, the results must not differ from the true value by more than 10%. If it's outside of the acceptable limits, terminate the analysis, correct the problem and recalibrate the instrument.

9.2.6. **Reporting limit check standard (CRI):** At the beginning of the analysis, verify the accuracy at the reporting limit (RL) by analyzing a solution at the RL level. RL check solution is analyzed to demonstrate that the mercury analyzer is capable of detecting the target analyte at the reporting limit (RL). Laboratory limits are 50-150% of the true value. This standard is being analyzed as part of some client's special projects.

10.0 Procedure

10.1 Sample Preparation

10.1.1 Filtration Procedure for Dissolved Mercury not filtered in the Field

10.1.1.1 The unpreserved sample must be filtered through a 0.45um filter unit as soon as practical after collection.
10.1.1.2 Collect the required volume of filtrate by using a 0.45um filter unit and a vacuum pump.
10.1.1.3 Acidify the filtrate with 1:1 HNO_3 to a pH of <2.
10.1.1.4 The method blank (MB) must be filtered and digested under the

same conditions as the lab filtered samples.

10.1.2 If sample is preserved in laboratory, hold sample for 16 hours for method 245.1 drinking water and 24 hours for wastewater following acidification. Verify and record if sample pH is < 2.0 after 16 or 24 hours (depending on the analysis- drinking water or wastewater) and prior to digestion/analysis. If pH is > 2.0, add HNO3, hold for 24 hours and verify pH.

10.1.3 Transfer 30 ml sample (DI water for MB and LCS) or standard, or an aliquot diluted to 30 ml, to an appropriately identified 50 ml hot block digestion cup. For QA samples, transfer 3 aliquots of 30 ml sample to three digestion cups labeled as SAMPLE, DUP and MS. Spike LCSW and MS with 0.1 µg of mercury (0.3 ml of DCAL-INT standard).

10.1.3.1. Due to insufficient sample volume or an unusual matrix (e.g., samples with potentially high interferences), it may be necessary to perform a pre-digestion dilution. For example, a 10X pre-digestion dilution can be performed by transferring 3 mL of sample to 50 mL hotblock cup. Add 27 ml of deionized water. Record 3 mL initial volume and 30 mL final volume in TALS (see Sec 11.5.2). Continue to section 10.1.3.

10.1.4 Add 1.5 ml concentrated H_2SO_4 and 0.75 ml concentrated HNO_3 mixing well after each addition.

10.1.5 Add 4.5 ml of potassium permanganate solution to each bottle. Mix well and let stand for 15 minutes (minimum); if the color has disappeared, add additional $KMnO_4$ until the purple color persists for at least 15 minutes (document in the Sample preparation log any additional amount of $KMnO_4$ added). The same amount of $KMnO_4$ must be added to the standards and samples.

10.1.6 Add 2.4 ml potassium persulfate solution to each bottle.

10.1.7 Heat for 2 hours in a 95^{o} C hot block digestor. Remove from block digestor and cool.

10.1.8 Add 1.8 ml Sodium chloride - Hydroxylamine hydrochloride solution to reduce the excess permanganate. Mix well; solution should become colorless. If necessary additional Sodium chloride - Hydroxylamine HCl solution may be added.

10.1.9 Due to differing rates of evaporation, the final volume of the standards/samples maybe different. <u>The standards and samples, including all QC, must be at the same final volume before analysis;</u> if not, find the sample or standard with the highest final volume (e.g., 41 mL) and add deionized water to any sample or standard to bring them to the same final

volume . Mix well. Wait at least 30 seconds after decolorization before analyzing.

10.2 Calibration

10.2.1. The instrument must be calibrated daily or once every 24 hours and each time the instrument is set up. The instrument is calibrated according to the manufacturer's specifications and must contain at least four standards and a blank. The laboratory currently uses five standards and a blank. The correlation coefficient of the calibration curve must be ≥0.995. If it does not, the problem must be corrected, and the instrument must be recalibrated. Standard preparations must be documented in TALS (see Sec 11.5.2).

10.2.2. Prepare five calibration standards, blank and calibration verification standard as detailed in sections 7.2.5 and 7.2.6.

10.2.3. Calibration Curve Read-Back:

10.2.1.1 Low-Level Readback (at the RL) – evaluate the 0.2 ug/L calibration standard. The %RE (relative error) must be +/- 20% of the true value (see Sec 11.4). If %RE is outside of criteria limits, stop the analysis and recalibrate.

10.2.1.2 Mid- Level Readback – evaluate the 5.0 ug/L calibration standard (the mid-level calibration standard). The %RE (relative error) must be +/- 10% of the true value (see Sec 11.4). If %RE is outside of criteria limits, stop the analysis and recalibrate.

10.3 Sample Analysis

10.3.1 Following a sample digestion procedure, the samples are ready for instrumental analysis. It is advisable to investigate each matrix for any complexities, which might adversely affect the acquisition of valid data.

10.3.2 The following analytical run sequence is currently used:

Instrument Calibration (Blank and five standards)

ICV
ICB
CRI
QCS
MCL (for drinking water analysis)
CCV
CCB
10 Samples

CCV
CCB
Repeat until run is completed
CCV
CCB

10.3.3 Instrument Operation:

10.3.3.1. Turn on Computer, Monitor and the Hg analyzer (Hydra IIAA).

10.3.3.2. Plumbing the Reagent Lines:

10.3.3.2.1. One at a time, feed each of the pump tubes into a pump cassette, sliding the tube through the plastic clips at the bottom until the plastic tab is secure. Then, holding the tube taut, slide the loaded cassette onto the pump head and click the clamp, lever up. The tab end of the tube should be located at the front of the pump head.

10.3.3.2.2. Reductant (Red): Connect tab end of tube to the reductant tubing that is connected to the reductant bottle and the other end to the mixing tee.

10.3.3.2.3. Sample (Blue): Connect tab end of tube to the autosampler probe and the other end to the mixing tee.

10.3.3.2.4. Drain (Blue): Connect the tab end of tube to the sample discharge tube connected on the Liquid/Gas separator and the other end to the waste line.

10.3.3.2.5. Rinse (Black): Connect tab end of tube to rinse tubing that is connected to the rinse bottle. Connect the other end to the rinse tubing leading to the rinse cup.

10.3.3.3. Preparation of Reagents:

10.3.3.3.1. Pour the 10% $SnCl_2$ solution into the reductant bottle.

10.3.3.3.2. Pour the 10% HCl solution into the rinse reservoir bottle.

10.3.3.4. Start the Program:

10.3.3.4.1. Click the Envoy icon on the computer desktop
10.3.3.4.2. Click Method, select 245.1_7470A, and select OK
10.3.3.4.3. On the main screen, click the StartUp icon. Wait 15

minutes before analyzing calibrating.

10.3.3.4.4. Click Sequence Tab on bottom of screen

10.3.3.4.5. Click Sequence on top of screen

10.3.3.4.6. Select Open, then select New

10.3.3.4.7. Using the Prep Batch Sheet and hand scanner, enter the sample barcodes into the Sample ID column. Include the Serial Dilution and all needed sample dilutions

10.3.3.4.8. Pour out the digested calibration standards and samples into the proper locations on the autosampler.

10.3.3.4.9. Click the Run Sequence icon to begin calibration and to run samples.

10.3.3.4.10. To shut down the instrument, click the Stop icon on the main screen.

11.0 Calculations / Data Reduction

11.1 Accuracy:

$$\text{ICV / CCV, LCS \% Recovery} = \frac{\text{observed concentration}}{\text{known concentration}} \times 100$$

$$\text{MS \% Recovery} = \frac{\text{(spiked sample) - (unspiked sample)}}{\text{spiked concentration}} \times 100$$

11.2 Precision (RPD):

$$\text{Matrix Duplicate (MD)} = \frac{|\text{orig. sample value - dup. sample value}|}{[(\text{orig. sample value + dup. sample value})/2]} \times 100$$

11.3. Final results calculation in aqueous samples :

$$\text{Concentration = mg/ L} = \frac{C \times V1 \times D}{V2}$$

Where:

C= Element concentration from instrument (ppb)

V1= Final volume of sample digested (in liters)

D= Dilution performed on sample

V2= Initial volume of sample digested (in liters).

11.4. Relative Error (%RE):

% Relative Error = (100) (MC-TC)/TC

MC = Measured Concentration of the calibration standard
TC = True Concentration for the calibration standard

11.5. Data Processing:

11.5.1. All data is recorded directly in TALS' Analyst Desktop II program.

11.5.2. Record standard/sample preparations in the Analyst Desktop II program located in the TestAmerica Laboratory System (TALS) Reagent module. The analyst must enter the following information: Source standard, Initial and final sample volume, spike name and amount used, all reagents and their corresponding lot numbers, creation and expiration dates.

11.5.3. Record the following reagents and the volume used for sample preparation in the batch information page under "batch comments" in TALS Analyst Desktop II: concentrated sulfuric acid, concentrated nitric acid, potassium permanganate, potassium persulfate and sodium chloride-hydroxylamine hydrochloride. Record reagents in TALS by opening the prep batch, click on "edit" and then right click to choose "view batch information." Enter the information in the "batch comments" section.

11.5.4. Import Data to TALS

Click the Analysis tab

11.5.4.1. Select the analytical run that needs to be imported
11.5.4.2. Select Statistics
11.5.4.3. Click Load and select TALS Import, click OK
11.5.4.4. Click Report, click CSV File
11.5.4.5. Name the import file (e.g., batch name, today's date)
11.5.4.6. The newly created import file is in the Import Folder on the desktop Send to TALS Import Folder

11.5.5. Creating the Raw Data PDF:

11.5.5.1. Click the Analysis Tab, select Detailed

11.5.5.2. Click Load, select Use This To Make PDF

11.5.5.3. Click Report, Click Printer

11.5.5.4. Include the calibration curve graph:

11.5.5.4.1. Go to Methods Tab and click Calibration, click Print

11.5.5.5. Combine both documents using the PDF Creator program. The new document is located in the Documents folder on the C:\ drive. Add this document to the "Doc's" location in the analytical batch.

11.5.6. Complete the Data Review Checker (DRC) in TALS: Prior to data submission (i.e., prior to 1st leveling data in the Analyst Desktop II module in TALS), the analyst must execute the DRC (Data Review Checker) program in the applicable analytical batch.

11.5.6.1. Open the analytical batch and click on the Edit tab above to enter the Edit Mode.

11.5.6.2. Press F8 command key on the computer keyboard or right click anywhere on the batch worksheet then click on 'Run checklist.'

11.5.6.3. Acknowledge by filling in responses to all unacknowledged findings.

11.5.6.3.1. Highlight the checklist with findings, then highlight the associated list of findings; right click and choose 'Acknowledge Item.'

11.5.6.3.2. Fill in appropriate comments in the response box, then hit 'OK.'

11.5.6.3.3. Acknowledge all Finding items in the 'Manual Batch Checklist' except for the *"2nd Level review complete?"* this is to be completed by the 2nd level reviewer

12. Method Performance

12.1. Method Detection Limit Study (MDL)

The method detection limit (MDL) is the lowest concentration that can be detected for a given analytical method and sample matrix with 99% confidence that the analyte is present. The MDL is determined according to the laboratory's MDL procedure in Section 19 (Test Methods and Method Validation) of TestAmerica Edison's Quality Assurance Manual (ED-QA-LQM). MDLs reflect a calculated (statistical) value determined under ideal laboratory conditions in a clean matrix, and may not be achievable in all environmental matrices. The laboratory maintains MDL studies for analyses performed; these are verified at least annually unless method requirements require a greater frequency.

12.2 Instrument Detection Limit:

The IDL for each analyte must be determined for each wavelength used on each instrument. The IDL must be determined annually or if the instrument is adjusted in any way that may affect the IDL. For 245.1, the IDL is determined by multiplying

the average of the standard deviations obtained from the analysis of 10 reagent blanks by 3. For 7470A, the IDL is determined by multiplying the average of the standard deviations obtained from the analysis of 7 reagent blanks by 3.14

12.3 Linear Dynamic Range (LDR)

The upper limit of the LDR must be established. It must be determined from a linear calibration prepared from a minimum of three different concentration standards, one of which is close to the upper limit of the linear range. The LDR should be determined by analyzing succeedingly higher standard concentrations of mercury until the observed analyte concentration is no more than 10% below the stated concentration of the standard. The determined LDR must be documented and kept on file. The LDR which may be used for the analysis of samples should be judged by the analyst from the resulting data. Determined sample analyte concentrations that are greater than 90% of the determined upper LDR limit must be diluted and reanalyzed. The LDR should be verified annually or whenever, in the judgement of the analyst, a change in analytical performance caused by either a change in instrument hardware or operating conditions would dictate they be redetermined.

12.4 Demonstration of Capabilities

For DOC procedure refer to Section 19 in the most current revision of TestAmerica Edison's Quality Assurance Manual (ED-QA-LQM).

12.5 Training Requirements

Refer to TestAmerica SOP No. ED-GEN-022, Training, for the laboratory's training program.

12.6 Lower Limit of Quantitation (LLOQ)

12.6.1. LLOQ verification: prior to analyzing samples under method 7470A, after any change that may affect the LLOQ, and quarterly, the LLOQ must be verified. The LLOQ is initially verified by the analysis of 7 replicate samples spiked at the LLOQ (0.2 ppb). The seven replicates must be digested prior to analysis. The LLOQ is verified when the mean recovery of the 7 replicates is +/-30% of the true value and the RSD < 20%.

12.6.2. LLOQ on-going verification: the LLOQ is re-verified quarterly by the analysis of a method blank (MB) and 1 blank sample (deionized water spiked at the LLOQ (0.2 ppb). The MB and LLOQ samples must be digested prior to analysis. The LLOQ is re-verified if the MB result is +/- 0.5 times the reporting limit for each analyte and when the recovery of the LLOQ sample is +/-30% of the .true value.

13. Pollution Control

It is TestAmerica's policy to evaluate each method and look for opportunities to minimize waste generated (i.e., examine recycling options, ordering chemicals based on quantity needed, preparation of reagents based on anticipated usage and reagent stability). Employees must abide by the policies in Section 13 of the Corporate Environmental Health and Safety Manual (CW-E-M-001) for "Waste Management and Pollution Prevention."

14. Waste Management

14.1. Waste management practices are conducted consistent with all applicable rules and regulations. Excess reagents, samples and method process wastes are disposed of in an accepted manner. Waste description rules and land disposal restrictions are followed. Waste disposal procedures are incorporated by reference to TestAmerica SOP No. ED-SPM-008 (Laboratory Waste Disposal Procedures).

14.2. The following waste streams are produced when this method is carried out.

- Digested Samples: Corrosive Acid- Materials that are not above regulatory limits will be submitted for elementary neutralization with 50% sodium hydroxide solution (Siedler Chemical SC-1824-03). Major concern is heat generated from the neutralization process. Initial volume of acid waste to be neutralized should be no more than 15 gallons. Finished neutralization with sodium bicarbonate (Siedler Chemical SC-0219-25) to a pH of 6 – 9 in the primary tank. Once pH has been established the primary tank is transferred through filter housing to a secondary tank. The pH is rechecked. If the pH is within specifications, the secondary tank is released to the municipal sewer system.

- Samples above regulatory limits and expired RCRA metals standards (Waste Corrosive Liquid, Acidic, Inorganic, n.o.s.) are collected in satellite accumulation and sent off site through a Waste disposal vendor.
  Onyx Profile WIP Number: 590598
  Teris Profile Number 50016653

15. References / Cross-References

15.1. Determination of Mercury in Water by Cold Atomic Absorption Spectrometry, EMSL-Cincinnati, EPA/600/R-94/11, May 1994; Method 245.1 Revision 3.0.

15.2. Test Methods for Evaluating Solid Waste Physical/Chemical Methods (SW846), Third Edition, September 1986, Final Update I, July 1992, Final Update IIA, August 1993, Final Update II, September 1994; Final Update IIB, January 1995; Final Update III, December 1996.

15.3. TestAmerica Edison Document No. ED-QA-LQM, *Laboratory Quality Manual*, most current revision.

15.4. TestAmerica Edison SOP ED-GEN-022, *Training*, most current revision.

15.5. TestAmerica Edison SOP No. ED-SPM-008, *Laboratory Waste Disposal Procedures*, current revision.

15.6. Corporate Environmental Health and Safety Manual CW-E-M-001, most current revision.

15.7. Leeman Hydra II AA Operating Manual

16. Method Modifications:

Item	Method No.	Modification
Sample Preparation	SW 7470A	Stannous Chloride is automatically added via the instrument versus the manual addition of Stannous Chloride as stated in the method. This is an instrument manufacturer's improvement that will reduce error due to loss of Mercury.
Sample Preparation	SW 7470A EPA 245.1	The hotblock has replaced the hot-water bath for digestion. This modification has been made to reduce cross-contamination (the hotblock tubes are disposable).
Sample Preparation	SW 7470A EPA 245.1	The typical prep sample size is reduced to 30ml (previously 100ml). This modification was made to allow for limited available sample volumes. Reagent volumes were adjusted to maintain sample to reagent volume ratio
Sample Preparation	EPA 245.1	The Initial Calibration Verification (ICV) will be prepared from a secondary source (as required in Method SW7470A), rather than the calibration standard source. Method 7470A and 245.1 will be prepared and analyzed in the same batch which improves efficiency and reduces waste. CCV will be prepared using the same source as the calibration standards.

17. Attachments

Attachment 1: Example Certificate of analysis (10 ppm Hg)

18. Revision History

- Revision 14, dated 11 August 2022
  - Sec 12.6: Added the lower limit of quantitation (LLOQ), associated instructions and recovery limits.
- Revision 13, dated 13 September 2021
  - Sec 7.2.10 & 9.2.6: Revised to clarify use of CRI standard.
- Revision 12, dated 03 August 2020
  - Updated header with Eurofins logo.
  - Sec 6.1.1: Replaced Hydra AA with Hydra IIAA

- Sec 6.1.2: Updated Hg software from WinHg to Envoy.
- Sec 8.0: Revised the required sample volume from 500ml to 250ml.
- Sec 9.1: Revised MB control limits for 245.1 and 7470A to <RL; added MSD to the QC table.
- Sec 9.1.1: Updated the control criteria for 245.1 and 7470A from <MDL to <RL.
- Sec 9.1.3: Updated the criteria for the matrix duplicate to reflect current laboratory practices.
- Sec 9.1.5: Added MSD to the list of Sample QC.
- Sec 9.2.2: Revised the CCV recovery limit for method 7470A to 10%.
- Sec 10.2.3: Added calibration curve read-back criteria.
- Sec 10.3.3.4: Updated instructions for the instrument/software operation.
- Sec 11.4: Added Relative % error calculation.
- Sec 11.5.2: Added reagent/standard information to document in TALS ADII.
- Sec 11.5.3.: Added requirements to record reagent volumes in the batch information page of TALS ADII; subsequent sections adjusted accordingly.
- Sec 11.5.4 & 11.5.5: Updated instructions for data import and raw data pdf generation.
- Sec 11.5.6: Added Data Review Checker (DRC) instructions.
- Sec 15.7 & 15.8: Deleted work instructions EDS-WI-007 (TestAmerica Edison Metals Data Review Checklist) and WI EDS-WI-125 (TestAmerica Edison Metals Initial-Calibration Data Review Mercury checklist), not applicable.

- Revision 11, dated 25 April 2018
  - Sec 8: Added footnote 3 to clarify instructions for metals sample preservation (verify pH <2.0 prior to digestion).
  - Sec 10.1.2: Added procedure for samples which pH are >2.0 and acid preservation is performed in laboratory.
  - Sec 12.3: Added procedure for the determination of linear dynamic range.

- Revision 10, dated 05 February 2018
  - Sec 7.2.10: Added CRI to the list of standards.
  - Sec 9.2.6: Added CRI to the list of instrument QC.
  - Sec 10.3.2: Revised the instrument sequence list to include CRI.

- Revision 9, dated 09 December 2015
  - Sec 7.2.7: Revised preparation procedure for ICV, standard solution should be obtained from a source different from the calibration standard. Removed reference to CCV.
  - Sec 7.2.8: Added CCV preparation procedure; standard solution same source as the calibration standard. Subsequent sections adjusted accordingly.
  - Sect 9.2.1: Revised - ICV solution should be prepared from a separate source.
  - Section 16: Added Method Modifications for using the secondary source when preparing the ICV standard for method EPA 245.1.

- Revision 8, dated 13 January 2014
  - Sec 7.1.8: Added the stock standard Potassium Persulfate to the list of reagents; subsequent sections adjusted accordingly.
  - Sect 7.1.15: Added preparation instructions for the 5% Potassium Persulfate

solution.
- Sec 7.1: Revised to include recording of reagent prep information in TALS and discontinue the use of Mercury Prep Logbook.
- Sec 7.2.5 & 7.2.6: Revised the spiking amount to add when preparing calibration standards in 30 ml final volume; also replaced the 100 ml flasks with 50 ml digestion cups.
- Sec 9.0: The matrix spike (MS) frequency for Method 245.1 has been revised to 1 per 10 samples to comply with the method.
- Sec 11.4.4: Added the Mercury calibration checklist (EDS-WI-125).
- Sec 15.8: Added WI# ED-WI-125 on the list of references; subsequent sections adjusted accordingly.
- Sec 17: Updated the attached COA for the Hg standards.

- Revision 7, dated 02 December 2011
  - Sec 1.1: Revised detection limit to 0.2 ug/L Hg to reflect actual laboratory limits.
  - Sec 1 & 12: Revised the LQM reference for DOC and Test Methods and Method Validation to Section 19.
  - Sec 3: Revised LQM reference for the definitions.
  - Sec 5: Revised in accordance with TestAmerica Corporate Quality SOP No. CW-QS-002, Writing a Standard Operating Procedure (SOP), most current revision.
  - Sec 7.1: Added requirements that all prepared reagents are recorded in TestAmerica Edison Mercury Std Prep Log.
  - Sec 7.2 & 10.2.1: Revised the documentation of standard preparation from logbook to TALS.
  - Sec 7.2.5: Replaced cal std 0.1 ug/L (Hg) with 0.2 ug/L.
  - Sec 9.1 and 9.1.1: Expanded MB control limit for 245.1 to reflect method's criteria.
  - Replaced PB (preparation blank) with MB (method blank) in various sections where applicable.
  - Sec 10.1.1: Added procedure for filtering dissolved samples in the lab.
  - Sec 10.1.2.1: Added procedure for pre-digestion dilutions.
  - Sec 10.1.8: Added procedure for visually checking and adjusting final volumes for all standards and samples.
  - Sec 10.3.3.11.7: Added procedure for post-digestion dilutions.

- Revision 6, dated 03 September 2009

  - Revised SOP format in accordance with TestAmerica Corporate Quality SOP No. CW-QS-002 (Rev 0) Writing a Standard Operating Procedure (SOP).
  - Combined SOP ED-MT-014 and ED-MT-015 with SOP ED-MT-017; retired SOP ED-MT-014 and ED-MT-015 at the effective date of this SOP.
  - Sec 6.1.3: Replaced water bath with block digestor.
  - Sec 6.2.1: Replaced 300 ml BOD bottles with 50 ml hot block digestion cups
  - Sec 6.2.3. Added 'Rinse Black' and changed Drain 'Black' to Drain 'Blue' in the list of Reagent lines
  - Sec 7.1: Added 0.15% HNO3 to list of reagents. Deleted Potassium Persulfate, Magnesium Perchlorate, 0.5N H2SO4 & 5% Potassium Persulfate solution to list of reagents; Reagents deleted are not applicable to this method.
  - Sec 7.2 Standards: Revised the Hg stock standard concentrations and preparation

of standards.

- Sec 7.1.9 & 7.1.13: Revised preparation procedure to reflect actual lab practices.
- Sec 7.2.6: Renamed the second source standard 'Initial Calibration Verification standard' to 'Quality control standard.' QCS is added in the Instrument QC and analytical run sequence.
- Section 8: Updated the section into Table format and have included the sample container, sample size requirements and method reference.
- Changed the verification standard concentration of ICV (3ppb) to 5ppb; CCV concentration remains 5ppb.
- Revised control limits to comply with Method 245.1
- Sec 9.1.1: Clarified QC limits for the Preparation Blank
- Sec 9.1.2: Revised the LCS limits for wastewater samples analyzed via Method 245.1 from ±20% to ±15%.
- Sec 9.1.4: Revised the MS limits for wastewater samples analyzed via Method 245.1 from ±20% to ±30%.
- Sec 9.1.5: Added Serial Dilution (L) in Sample QC
- Sec 9.2.1: Clarified the recovery limits of ICV for method 245.1; % Rec limits were revised from 10% to 5% to reflect actual laboratory practices.
- Sec 10.3.2: Added MCL in the analytical run sequence.
- Sec 10.3.3.2: Added 'Rinse Black' and changed Drain 'Black' to Drain 'Blue' in the list of Reagent lines.
- Sample size reduced from 100 ml to 30ml; preparation of the LCS (Sec 9.1.2) and MS (Sec 9.1.4) were revised to reflect this change in sample volume.
- Sec 10.1: Adjusted reagent volume to maintain sample to reagent ratios.
- Sec 11: Updated data processing in accordance with the new TALS.
- Sec 15: Added applicable references.
- Sec 16: Described the elimination of stannous chloride in the sample preparation and replacement of hot water bath as a method modification.

Attachment 1

SCP SCIENCE
Providing Innovative Solutions to Analytical Chemists

ACCREDITED

Certificate of Analysis

1.0 DESCRIPTION : ***Plasma*** **CAL – Custom Standard**

Catalogue Number : **141-110-11X**
Lot Number : **S130418021**
Matrix: 10.0% HNO3
Expiration Date : **July 2014**

2691783
ID: ME_Hg_1st_00013
Exp: 07/01/14 Post DLE
Mercury Calibration Stand

2.0 CERTIFIED VALUES AND ASSOCIATED UNCERTAINTY:

Method of Analysis: Inductively Coupled Plasma Atomic Emission Spectroscopy (ICP-AES)

Traceability: Applicable NIST Standard Reference Materials (see list below):

3101a	Al	3109a	Ca	3117a	Eu	3126a	Fe	3134	Mo	3142a	Pr	3151	Ag	3159	Th	3167a	Y
3102a	Sb	3110	Ce	3118a	Gd	3127a	La	3135a	Nd	3143	Re	3152a	Na	3160a	Tm	3168a	Zn
3103a	As	3111a	Cs	3119a	Ga	3128	Pb	3136	Ni	3144	Rh	3153a	Sr	3161a	Sn	3169	Zr
3104a	Ba	3112a	Cr	3120a	Ge	3129a	Li	3137	Nb	3145a	Rb	3154	S	3162a	Ti		
3105a	Be	3113	Co	3121	Au	3130a	Lu	3138	Pd	3147a	Sm	3155	Ta	3163	W		
3106	Bi	3114	Cu	3122	Hf	3131a	Mg	3139a	P	3148a	Sc	3156	Te	3164	U		
3107	B	3115a	Dy	3123a	Ho	3132	Mn	3140	Pt	3149	Se	3157a	Tb	3165	V		
3108	Cd	3116a	Er	3124a	In	3133	Hg	3141a	K	3150	Si	3158	Tl	3166a	Yb		

Certified Concentrations:

Hg 9.96 ± 0.06 µg/ml

Note: The uncertainty of the certified value has been calculated from applicable uncertainty contributors (u_i) such as the SRM inherited uncertainty, weighing and dilution errors and instrument variability. The combined uncertainty ($u_c = \sqrt{\Sigma u_i^2}$) has been multiplied by a coverage factor (k) of 2 to provide a 95% confidence interval.

3.0 REFERENCE VALUES:

Density: **1.051 g/ml** @ **20.9 °C**

4.0 APPROVAL AND DATE OF CERTIFICATION:

Certification Approval: Yaling Sui, Chemist Yaling Sui
Certification Date: **April 19, 2013**

INORGANIC VENTURES
300 Technology Drive
Christiansburg, VA 24073 • USA
inorganicventures.com

tel: 800.669.6799 • 540.585.3030
fax: 540.585.3012
info@inorganicventures.com

C.C. CERTIFICATE OF ANALYSIS

1.0 **INORGANIC VENTURES** is an ISO Guide 34 "General Requirements for the Competence of Reference Material Producers" and ISO 9001 registered manufacturer. Our manufacturing laboratory is accredited to ISO/IEC 17025 "General Requirements for the Competence of Testing and Calibration Laboratories."

ACCREDITED Testing Laboratory Certificate 883.01

ACCREDITED Reference Material Producer Certificate 883.02

2.0 **DESCRIPTION OF CRM** **10 µg/mL Mercury in 10% (v/v) HCL**

2035815
ID: ME_Hg_00019
Exp: 04/01/16 Prpd: CDC
Mercury Calibration Stand

Catalog Number:	MSHG-10PPM
Lot Number:	F2-HG02097
Starting Material:	Hg metal
Starting Material Purity (%):	100.0000
Starting Material Lot No:	R307HGA1
Matrix:	10% (v/v) HCL

3.0 **CERTIFIED VALUES AND UNCERTAINTIES**

Certified Concentration: 9.990 ± 0.074 µg/mL

Certified Density: 1.026 g/mL (measured at 20 ± 1°C)

The following equations are used in the calculation of the certified value and the uncertainty. Reported uncertainties represent expanded uncertainties expressed at approximately the 95% confidence level using a coverage factor of k = 2.

Certified Value $(\bar{x}) = \frac{\Sigma x_i}{n}$ ($\bar{x}$) = mean

x_i = individual results

n = number of measurements

Uncertainty (±) = $2 [\Sigma(s_i)^2]^{1/2}$ 2 = the coverage factor.

$[\Sigma(s_i)^2]^{1/2}$ = The square root of the sum of the squares of the most common errors (where 's' stands for the standard deviation) from instrumental measurement, density, NIST SRM uncertainty, weighing, dilution to volume, homogeneity, long term stability and short term stability.

4.0 **TRACEABILITY TO NIST AND VALUES OBTAINED BY INDEPENDENT METHODS**

- "Property of the result of a measurement or the value of a standard whereby it can be related to stated references, usually national or international standards, through an unbroken chain of comparisons all having stated uncertainties." (ISO VIM, 2nd ed.,

Title: SW-846 Method 6020B, Trace Metals Analysis of Water, Wastewater, Soil, Sediment and Leachate Samples by ICP-MS

Once printed, this is considered an uncontrolled document

Approvals (Signature/Date):

Laura Demone 06/16/22
Laura Demone Date
Department Manager

06/16/22
Dan Helfrich Date
Health & Safety Manager/Coordinator

06/16/22
Carl Armbruster Date
Quality Assurance Manager

06/16/22
Mark Acierno Date
Laboratory Director

Donald Evans 06/16/22

1.0 Scope and Application

1.1 Analytes, Matrix(s), and Reporting Limits

This SOP describes the procedures used to determine the concentration of dissolved elements in water samples, various elements in groundwater, aqueous samples, industrial wastes, soils, sludges, sediments, and other solid wastes for which total (acid-soluble) elements are required by Inductively Coupled Plasma - Mass Spectrometry (ICP-MS) using EPA SW-846 Method 6020B.

The routine target analytes and reporting limits are as follows:

Table 1

Analyte List and Method Reporting Limits (RL)

Element	Digested Water RL	Soil RL
	Prep Method: 3005A &3010A (ug/L)	Prep Method: 3050B (mg/Kg)
Aluminum (Al)	40	20
Antimony (Sb)	2	1
Arsenic (As)	2	1
Boron (B)	80	40
Barium (Ba)	4	2
Beryllium (Be)	0.8	0.4
Cadmium (Cd)	2	1
Calcium (Ca)	200	100
Cobalt (Co)	4	2
Chromium (Cr)	4	2
Copper (Cu)	4	2
Iron (Fe)	120	60
Lead (Pb)	1.2	0.6
Magnesium (Mg)	200	100
Manganese (Mn)	8	4
Molybdenum (Mo)	4	2
Nickel (Ni)	4	2
Potassium (K)	200	100
Selenium (Se)	2.5	1.25
Silver (Ag)	2	0.40
Sodium (Na)	200	100
Strontium (Sr)	4	2
Thallium (Tl)	0.8	0.4
Tin (Sn)	16	8
Titanium (Ti)	4	2

Vanadium (V)	4	2
Zinc (Zn)	16	8

On occasion clients may request modifications to this SOP. These modifications are handled following the procedures outlined in Sections 7 (Review of Work Request) and 19 (Test Methods and Method Validation) of TestAmerica Edison's Quality Assurance Manual (ED-QA-LQM).

2.0 Summary of Method

Prior to analysis by ICP-MS, the sample must be solubilized or digested using the sample preparation method appropriate to the matrix. Sample digestates are aspirated and nebulized into a spray chamber. A stream of argon gas carries the sample aerosol through the innermost of three concentric tubes and injects it into the middle of the donut-shaped plasma. The sample elements are dissociated, atomized, and excited to a higher energy level. The ions that are produced are entrained in the plasma and introduced, by means of an interface, into a mass spectrometer. The ions are sorted according to their mass to charge ratios and quantified with a channel mass spectrometer.

Aqueous samples are digested using SW846 Method 3005A or 3010A. Soil samples are digested using Method 3050B. Refer to TestAmerica Edison SOP Nos. ED-MTP-002, ED-MTP-003 and ED-MTP-005, for the digestion procedure (see Section 15.0 for complete references).

3.0 Definitions

For a complete list of definitions refer to Appendix 2 in the most current revision of TestAmerica Edison's Quality Assurance Manual (ED-QA-LQM).

4.0 Interferences

4.1. Isobaric elemental interferences are caused by isotopes of different elements which form singly or doubly charged ions of the same nominal mass-to-charge ratio and which cannot be resolved by the mass spectrometer in use. Isobaric polyatomic/molecular ion interferences are caused by ions consisting of more than one atom which have the same nominal mass-to-charge ratio as the isotope of interest and which cannot be resolved by the mass spectrometer in use. Most isobaric interferences that could affect the ICP-MS analysis for elements in this SOP have been identified. These can be managed by the selection of an alternate isotope, the use of the collision/reaction cell (used for non-potable samples only), or by the use of elemental interference equations when the collision/reaction cell is not in use.

4.2. Krypton affects the determination of both arsenic and selenium but can be greatly reduced with the use of high purity Krypton free argon. Krypton must be analyzed for every sample and standard.

4.3. Physical interferences are effects associated with the sample nebulization and transport processes as well as ion-transmission efficiencies. Changes in viscosity can cause significant inaccuracies, especially in samples containing high concentrations of dissolved solids or high acid concentrations. These changes in matrix can cause significant signal suppression or enhancement. Dissolved solids can deposit on nebulizer tips and interface cones (reducing the orifice size and the instrument's performance). Internal standards can be used to correct for physical interferences if they are carefully matched to the analyte so that both elements react similarly to the matrix changes.

4.4. Memory interferences can occur when analytes from a previous sample contribute to signals measured from subsequent samples. The memory effects can result from analyte deposition of sample on the sample tubing, joints, nebulizer, spray chamber, torch, and/or interface cones. Routine maintenance on the sample introduction system is necessary in order to minimize the memory interferences. The memory effects must be taken into account when setting up a suitable rinse times. The evaluation of a minimum of three replicate integrations will help to determine memory problems.

4.5. Contamination of the sample can occur when the preparation glassware and/or reagents contain the target elements. Reagent blanks (method blanks) must be analyzed as a check on contamination due to the sample digestion.

5.0 Safety

Employees must abide by the policies and procedures in the Corporate Environmental Health and Safety Manual (CW-E-M-001) and this document. This procedure may involve hazardous material, operations and equipment. This SOP does not purport to address all of the safety problems associated with its use. It is the responsibility of the user of the method to follow appropriate safety, waste disposal and health practices under the assumption that all samples and reagents are potentially hazardous. Safety glasses, gloves, lab coats and closed-toe, nonabsorbent shoes are a minimum.

5.1. Specific Safety Concerns or Requirements

The ICP plasma emits strong UV light and is harmful to vision. All analysts must avoid looking directly at the plasma.

5.2. Primary Materials Used

The following is a list of the materials used in this method, which have a serious or significant hazard rating. **Note: This list does not include all materials used in the method. The table contains a summary of the primary hazards listed in the MSDS for each of the materials listed in the table.** A complete list of materials used in the method can be found in the reagents and materials section.

Employees must review the information in the MSDS for each material before using it for the first time or when there are major changes to the MSDS.

Material	Hazards	Exposure Limit (1)	Signs and symptoms of exposure
Hydrochloric Acid	Corrosive Poison	5 ppm-Ceiling	Inhalation of vapors can cause coughing, choking, inflammation of the nose, throat, and upper respiratory tract, and in severe cases, pulmonary edema, circulatory failure, and death. Can cause redness, pain, and severe skin burns. Vapors are irritating and may cause damage to the eyes. Contact may cause severe burns and permanent eye damage.
Nitric Acid	Corrosive Oxidizer Poison	2 ppm-TWA 4 ppm-STEL	Nitric acid is extremely hazardous; it is corrosive, reactive, an oxidizer, and a poison. Inhalation of vapors can cause breathing difficulties and lead to pneumonia and pulmonary edema, which may be fatal. Other symptoms may include coughing, choking, and irritation of the nose, throat, and respiratory tract. Can cause redness, pain, and severe skin burns. Concentrated solutions cause deep ulcers and stain skin a yellow or yellow-brown color. Vapors are irritating and may cause damage to the eyes. Contact may cause severe burns and permanent eye damage.
1 – Exposure limit refers to the OSHA regulatory exposure limit.			

6.0 Equipment and Supplies

6.1. Instrumentation

- Agilent Agilent 7800, and Agilent 7900: inductively coupled plasma mass spectrometer with data system (microprocessor, monitor, printer) and autosampler. Mass Range 2-260 amu. Vacuum purged spectrophotometer with an axial plasma torch. Collision/Reaction Cell.
- Heat exchanger –PolyScience Model 6106T Recirculating Chiller or equivalent
- Autosampler –Agilent SPS 4 Autosampler

6.2. Supplies

- Reagent Water -18 megohm Reagent grade Type II water

- Volumetric Flasks (Class A): 50 mLs, 100 mLs, 500mls & 1000mls
- Eppendorf & Fisher Pipettes, varying volumes
- Polypropylene tubes

7.0. Reagents and Standards

7.1. Reagents

7.1.1. Concentrated Nitric Acid (HNO3) - Trace Grade or Equivalent; store at room temperature; for stability information, refer to manufacturer's instructions. The assay sheet of each lot of acid received into the lab must be reviewed to ensure the quality of the acid is sufficient for trace analysis of metals.

7.1.2. Concentrated Hydrochloric Acid (HCL) - Trace Grade or Equivalent; store at room temperature; for stability information, refer to manufacturer's instructions. The assay sheet of each lot of acid received into the lab must be reviewed to ensure the quality of the acid is sufficient for trace analysis of metals.

7.1.3. Argon supply - 99.9% (Liquid)

7.1.4. Helium and Hydrogen supply – 99.999% (Gas)

7.1.5. 18 megohm Reagent grade Type II water.

7.1.6. 5% HNO3 + 5% HCl: Add 1 liter of concentrated HNO3 and 1 liter of concentrated HCl to deionized water and bring to 20 liter volume with deionized water. Varying amounts of this reagent may be made (e.g., 10L) by proportionally adjusting the volume of acids used. Note: Always add acid to water. Record preparation in the TALS Reagent Module. Prepare every 12 months or refer to manufacturer's expiration date; store at room temperature.

7.2. Standards

Calibration and spike solutions are prepared from either certified stock solutions or from stock solutions purchased from vendors. Certificates of analysis or purity must be received with all neat compounds or stock solutions. Certificate of analysis are filed in Metals manager's office.

Storage requirements: all standards are stored at room temperature
Shelf-life: Stock standards – refer to manufacturer's instructions
Intermediate standards – 12 months

Working cal standards – 12 months
Working Initial calibration verification standard – prepared fresh daily
Interference Check Standards (A&B) - weekly
(Note: expiration date must not go beyond the expiration date of the source stock).

Concentration: Final concentrations for the calibration standards are given in Attachment 1. All standards must be prepared in 5% HNO3 / 5% HCl (Sec. 7.1.6).

Standards must be prepared every 12 months or sooner if needed or required. "If needed" means the standard has been exhausted; "if required" means that the standard does not meet the QC criteria.

All standards must be prepared in 5% HNO3 / 5% HCl (Sec. 7.1.6). Final concentrations for the calibration standards are given in Attachment 1.
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7.2.1. Calibration Standards: purchased from CPI International. Refer to manufacturer's instructions for stability and storage information.

6020B RL Standard contains: 20 mg/L Ca, K, Mg, Na
12 mg/L Fe
8 mg/L B
4 mg/L Al
1.6 mg/L Sn, Zn
0.8 mg/L Mn
0.4 mg/L Ba, Co, Cr, Cu, Mo, Ni, Sr, Ti, V
0.25 mg/L Se
0.2 mg/L Ag, As, Cd, Sb,
0.12 mg/L Pb
0.08 mg/L Be, Tl

TA-CAL-1 contains: 1,000 mg/L Si
100 mg/L As, Ba, Be, Cd, Co, Cr, Cu, Li, Mn, Mo, Ni, Pb, Sb, Se, Si, Sn, Sr, Ti, Tl, V

TA-CAL-2 contains: 1,000 mg/L Al, Ca, Fe, K, Mg, Na

Single Element Standard: Ag 1,000 mg/L
Single Element Standard: B 1,000 mg/L
Single Element Standard: Zn 1,000 mg/L

7.2.2. Working Calibration Standards:

CAL5: Add 100ml of 5% HNO3 / 5% HCl (Sec 7.1.6) to a clean 200ml flask. Add 0.4ml of TA-CAL-1 and 2ml of TA-CAL-2. Add 10

ml of MS-Cal-Int (see Sec 7.2.2.1). Bring to volume with 5% HNO3 / 5% HCl.

CAL4/CCV: Add 100ml of 5% HNO3 / 5% HCl (Sec 7.1.6) to a clean 200ml flask. Add 0.2ml of TA-CAL-1 and 1ml of TA-CAL-2. Add 5 ml of MS-Cal-Int (see Sec 7.2.2.1). Bring to volume with 5% HNO3 / 5% HCl.

CAL3: Add 100ml of 5% HNO3 / 5% HCl (Sec 7.1.6) to a clean 200ml flask. Add 0.1ml of TA-CAL-1 and 0.5ml of TA-CAL-2. Add 2.5 ml of MS-Cal-Int (see Sec 7.2.2.1). Bring to volume with 5% HNO3 / 5% HCl.

CAL2: Add 100ml of 5% HNO3 / 5% HCl to a clean flask. Add 10ml of 6020B RL Standard. Bring to volume with 5% HNO3 / 5% HCl (Sec 7.1.6).

CAL1: Add 100ml of 5% HNO3 / 5% HCl to a clean flask. Add 2ml of 6020B RL Standard. Bring to volume with 5% HNO3 / 5% HCl (Sec 7.1.6).

7.2.2.1. MS-Cal-Int: Add 100ml of 5% HNO3 / 5% HCl (Sec 7.1.6) to a clean 200ml flask. Add 2ml each of B and Zn. Add 0.4ml of Ag. Bring to volume with 5% HNO3 / 5% HCl (Sec 7.1.6).

Note: see Attachment 1 for final elemental concentrations.

7.2.3. Initial Calibration Verification Stock Standards: purchased from CPI International. Refer to manufacturer's instructions for stability and storage information. Standards must be from a different source than those used for the calibration standards.

TA-CAL1-SS contains: 1,000 mg/L Si
100 mg/L As, Ba, Be, Cd, Co, Cr, Cu, Li, Mn, Mo, Ni, Pb, Sb, Se, Si, Sn, Sr, Ti, Tl, V

TA-CAL2-SS contains: 2,000 mg/L Al, Ca, Fe, K, Mg, Na

2nd Source Single Element Standard: Ag 1,000 mg/L
2nd Source Single Element Standard: B 1,000 mg/L
2nd Source Single Element Standard: Zn 1,000 mg/L

7.2.4. ICV Working Standard: The ICV Working Standard must be at a concentration other than the concentrations used for the CCV working standard. Prepare by adding 100ml of 5% HNO3 / 5% HCl (Sec 7.1.6) to a clean 200ml flask. Add 0.16ml of TA-CAL-1-SS and 2.0 ml of TA-CAL-2-SS. Add 4 ml of MS-ICV-Int (see Sec 7.2.4.1). Bring to volume with 5% HNO3 / 5% HCl. The final concentrations of the various elements are listed in Attachment 1. This standard is prepared fresh monthly and stored at room temperature.

7.2.4.1. MS-ICV-Int: Add 100ml of 5% HNO3 / 5% HCl (Sec 7.1.6) to a clean 200ml flask. Add 2ml each of 2nd Source: B & Zn. Add 0.4ml of 2nd Source: Ag. Bring to volume with 5% HNO3 / 5% HCl (Sec 7.1.6).

7.2.5. Continuing Calibration Verification standard: see CAL4/CCV (Sec. 7.2.2).

7.2.6. Internal Standards: The following elements are used as the internal standards: Bi, In, Li6, Sc, Tb, and Ge.

- Testamerica-1 standard: supplier-Inorganic Ventures; a mixed standard containing 250 ppm each: Li6 and Sc and 50 ppm each: Bi, In and Tb
- MSGE-100ppm standard: supplier- Inorganic Ventures; this standard contains 100ppm Ge.
- Triton X-100 (Standard Stock Solution: Non-ionic Surfactant) purchased from Fisher Scientific, p/n BP151-100. Refer to manufacturer's instructions for stability and storage information. According to the ICPMS manufacturer (Agilent Technologies), Triton-X 100 will reduce the %RSD of the triplicate analysis.

Prepare a working internal standard solution in a 1000 ml volumetric flask: add 1 ml of Testamerica-1, 1 ml of MSGE-100ppm and 0.1 ml of Triton X-100. Bring to volume with 5% HNO3 / 5% HCl.

The following are the recommended analysis masses and internal standards:

Element	IS
Be9	Li6
B11	Li6
Na23	Sc45
Mg24	Sc45
Al27	Sc45
K39	Sc45
Ca40	Sc45
Ti47	Sc45
V51	Sc45

Cr52	Sc45
Mn55	Sc45
Fe56	Sc45
Co59	Sc45
Ni60	Sc45
Cu63	Sc45
Zn 66	Sc45
As75	Sc45
Se78	Sc45
Sr88	In115
Kr83**	(none)
Mo95	In115
Ag107	In115
Cd111	In115
Sn118	In115
Sb121	In115
Ba137	Tb159
Tl205	Bi209
Pb 208*	Bi209

*Pb 208 = Pb 208 + Pb 207 + Pb 206
**Kr83 is used for monitoring Krpyton levels only.

Different masses and internal standards may be utilized as matrix issues deem necessary. Samples are analyzed using collision/reaction cell technology (helium/hydrogen gas modes). Typical sample analysis: Calcium, Iron, and Selenium are analyzed in the Hydrogen reaction mode; Beryllium and Boron are analyzed in the No Gas mode; all other elements are analyzed in the helium collision mode.

Tune check criteria:

Instrument tunes must be performed daily, before calibration. A solution at ~10ppb of Be, Mg, Co, In, and Pb is analyzed, and the precision, mass calibration, and resolution are checked. Resolution is checked by analyzing Magnesium isotopes 24, 25, 26 and Lead isotopes 206, 207, 208.

The following limits are used to evaluate mass calibration, resolution, and instrument stability:

The following limits are used to evaluate the tune:

Mass calibration: +/-0.1 amu
Resolution check: <0.9 amu at 10% peak height
Stability (5reps): <5%

7.2.7. Spectral Interference Check (SIC) Solutions: A working stock solution may

be purchased for the ICSA solution. The stock solution which is purchased from CPI International contains the following elemental concentrations:

TA-ICPMS-ICSA contains: 10000 mg/L Chloride; 2000 mg/L Carbon; 1000 mg/L ea: Al, Ca, Fe, K, Mg, Na, P, S; 20 mg/L ea: Mo, Ti

7.2.8. Working SIC Standards (ICSA):

ICSA (6020B): Add 50ml of 5% HNO3 / 5% HCl (Sec 7.1.6) to a clean 100ml flask. Add 10ml of TA-ICPMS-ICSA stock solution. Bring to volume with 5% HNO3 / 5% HCl (Sec 7.1.6). This standard is made fresh weekly; store at room temperature. Refer to Attachment 2 for final elemental concentrations.

7.2.9. ICP-MS Matrix Spiking Solution, ICPMS LCS/SPK: purchased from High-Purity Standards. Refer to manufacturer's instructions for stability and storage information. Refer to attachment 1 for final elemental concentrations. Stock solution contains the following elemental concentrations:

Element	Conc. Of Stock (mg/L)
Aluminum (Al)	500
Antimony (Sb)	5
Arsenic (As)	10
Barium (Ba)	10
Beryllium (Be)	5
Boron (B)	100
Cadmium (Cd)	5
Calcium (Ca)	500
Chromium (Cr)	10
Cobalt (Co)	5
Copper (Cu)	10
Iron (Fe)	500
Lead (Pb)	5
Magnesium(Mg)	500
Manganese (Mn)	50
Molybdenum (Mo)	10
Nickel (Ni)	10
Potassium (K)	500
Selenium (Se)	10
Silver (Ag)	5
Sodium (Na)	500
Strontium (Sr)	10
Thallium (Tl)	4
Tin (Sn)	10

Element	Conc. Of Stock (mg/L)
Titanium (Ti)	10
Vanadium (V)	10
Zinc (Zn)	50

7.2.10. EPA Tune Check

7.2.10.1. EPA Tune Check Stock Standard: 2008TS purchased from Inorganic Ventures. Refer to manufacturer's instructions for stability and storage information. Contains: 10 ug/mL each: Be, Co, In, Mg, Pb.

7.2.10.2. Working EPA Tune Check Standard: add 100ml of 5% HNO3 / 5% HCl to a clean 1000ml flask. Add 10 mL of 2008TS. Bring to volume with 5% HNO3 / 5% HCl . This solution contains 100 ug/L of Be, Co, In, Mg and Pb. Analyze at a 10X dilution.

8.0. Sample Collection, Preservation, Shipment and Storage

Sample container, preservation techniques and holding times may vary and are dependent on sample matrix, method of choice, regulatory compliance, and/or specific contract or client requests. Listed below are the holding times and the references that include preservation requirements.

Matrix	Sample Container	Min. Sample Size	Preservation	Holding Time [1]	Reference
Metals Waters	Polyethylene or Glass	250 mLs	HNO_3 to pH < 2 prior to shipment; if not, acidify upon receipt in lab [2,3]	6 months	40 CFR Part 136.3
Soils	8 oz or 16 oz.Polyethylene or Glass	10 grams	Cool 4 ± 2°C	6 months	SW846 Method 6020B

[1] Inclusive of digestion and analysis.
[2] Acid preservation may be omitted for shipping; however, acid must be added upon receipt in the lab. Following acidification, mix the sample and hold for at least 24 hours. Just prior to digestion or direct analysis, verify pH<2. If pH≥2, repeat steps (i.e., add acid, hold for 24hrs, verify ph<2).
[3] Aqueous samples may be stored at room temperature.

Note: Samples for dissolved metals should be filtered in the field before acid is added to the sample. If the sample is to be filtered in the lab, no preservative is added to the sample until the sample is filtered.

Leachates are transferred to a plastic container after the applicable leachate extraction procedure. The sample is preserved with HNO3 to a pH<2. (If a matrix spike is necessary, then an aliquot is spiked prior to acid preservation). Leachate samples must be analyzed within 6 months of the extraction.

9.0. Quality Control

Note: If a batch of samples requires digestion, then the relating QC samples must be carried through the entire digestion process.

9.1. Sample QC - The following quality control samples are prepared with each batch of samples.

Quality Control	Frequency	Control Limit
Method blank	One per batch of twenty samples or less	<1/2 the RL; or project specific requirements; or <10% of the measured concentration in the sample
Lab Control Sample Water (LCSW)	One per batch of twenty samples or less	80-120%
Matrix Duplicate	One in 20 or fewer samples	If original sample and dup are both ≥ 5X CRQL, then ≤ 20% RPD. If original sample and duplicate are less than the CRQL, the RPD is not calculated; otherwise ±CRQL.
Matrix Spike[1]	One per batch of twenty samples or less	75-125%
Matrix Spike Duplicate[1]	Whenever the client requests	75-125%; If original sample and dup are both ≥ 5X CRQL, then ≤ 20% RPD. If original sample and duplicate are less than the CRQL, the RPD is not calculated; otherwise ±CRQL.
Post Digestion Spike	One per batch of twenty samples or less	See Section 9.1.7
Laboratory Control Sample Soil Reference Material (LCSSRM)	1 in 20 or fewer samples	Vendor's certified limits
Serial Dilution (1/5 dilution)	One per batch of twenty samples or less	See Section 9.1.6

[1] The sample for MS/MSD is randomly selected, unless specifically requested by a client.

9.1.1. Method Blank (MB): One laboratory method blank will be analyzed with each batch of samples prepared together (not to exceed 20 samples). The method blank is used to identify possible contamination during acid digestion. Results must be less than the highest of: (i) ½ the RL, (ii)project specific requirements, or (iii)10% of the measured concentration of the sample. If any analyte concentration in the blank is above the control limit, the batch must be prepared again for the element in question and the samples reanalyzed.

9.1.2. Laboratory Control Sample Water (LCS): A laboratory control sample must be analyzed with each group of samples digested. Refer to Water Matrix Spike (MS) concentration in Attachment 1 for final elemental concentrations. Results must be within the acceptable control limits, if not, all samples prepared in association with the LCS must be redigested and reanalyzed.

- Water samples - prepare the LCS by adding 0.25 ml of ICP-MS LCS/SPK (Sec. 7.2.10) in 50 ml deionized water. Results must be within ± 20% of the true value.

- Soil samples - LCS for solid matrices (LCSSRM) is obtained from a vendor supplied solid matrix and is carried through the same preparation procedure as the samples. The results of the solid LCS must fall within the 'QC Performance acceptance limits' of the reference material used for that sample. If not, all samples prepared in association with the LCS must be redigested and reanalyzed.

9.1.3. Matrix Duplicate: A duplicate is analyzed for each batch of samples. If original sample and duplicate are both ≥ CRDL, then ≤20% RPD. If original sample and duplicate are less than the CRDL, the RPD is not calculated; otherwise, ±CRDL.

9.1.4. Matrix Spike (MS): A matrix spike is prepared and analyzed for each batch of samples digested. A recovery of 75-125% is required (an exception to this occurs if the sample concentration exceeds the spike concentration by a factor of four or more). If the recovery is not within specified limits, a post digestion spike is required (see Sec 7.2.9). See Attachment 1 for final Matrix Spike concentrations for water and soil.

9.1.4.1. For aqueous MS sample, spike 0.25 ml of ICP-MS LCS/SPK (Sec 7.2.9) into 50 ml sample.

9.1.4.2. For soil MS sample, spike 1.0 ml of ICP-MS LCS/SPK into 1 gram of sample.

9.1.5. Matrix Spike Duplicate (MSD): A matrix spike duplicate is analyzed when

requested by the client. Spike the QC sample the same way as the Matrix Spike (MS) sample. If original sample and duplicate are both ≥ CRDL, then ≤20% RPD. If original sample and duplicate are less than the CRDL, the RPD is not calculated; otherwise, ±CRDL. A recovery of 75-125% is required (an exception to this occurs if the sample concentration exceeds the spike concentration by a factor of four or more). If the recovery is not within specified limits, a post digestion spike is required (see Sec 9.1.7). See Attachment 1 for final Matrix Spike concentrations for water and soil.

9.1.6. Serial Dilution QC Check: A 1/5 dilution is prepared and analyzed on one sample per batch to determine if matrix interferences are present.

9.1.6.1. Select a sample and dilute the digestate by a factor of 5 (DF=5).

9.1.6.2. Analyze the dilution using the same procedures used for the un-diluted aliquot.

9.1.6.3. Compare the results of the diluted and un-diluted aliquots of sample digestate.

9.1.6.4. If the analyte concentration is minimally a factor of 25 above the RL (Reporting Limit), then the results of the dilution should be within ±20% of the result of the undiluted sample (original sample). If not, then a chemical or physical interference effect should be suspected.

9.1.7. Post-Digestion Spike: To check for possible matrix interference, analyze a post digestion spike on a representative sample (minimum of 1 per batch).

9.1.7.1 Transfer 10mL of a digestate, preferably the same sample that was selected for the matrix spike, to a suitable vial.

9.1.7.2 Spike the sample with 0.01 ml of ICP-MS LCS/SPK (Sec 7.2.9). Analyze the spiked aliquot and an un-spiked aliquot (the un-spiked may have been analyzed previously and does not need to be reanalyzed). The final concentration of the post digestion spike is as follows:

Element	Post spike concentration (ug/L)
Aluminum (Al)	500
Antimony (Sb)	5

Element	Post spike concentration (ug/L)
Arsenic (As)	10
Barium (Ba)	10
Beryllium (Be)	5
Boron (B)	100
Cadmium (Cd)	5
Calcium (Ca)	500
Chromium (Cr)	10
Cobalt (Co)	5
Copper (Cu)	10
Iron (Fe)	500
Lead (Pb)	5
Magnesium(Mg)	500
Manganese (Mn)	50
Molybdenum (Mo)	10
Nickel (Ni)	10
Potassium (K)	500
Selenium (Se)	10
Silver (Ag)	5
Sodium (Na)	500
Strontium (Sr)	10
Thallium (Tl)	4
Tin (Sn)	10
Titanium (Ti)	10
Vanadium (V)	10
Zinc (Zn)	50

9.1.7.3 Calculate the percent recovery of the post digestion spike as follows:

$$\%REC = \frac{Cps - Cs}{C2} \otimes 100$$

Where: Cps = concentration of post digestion spike (ug/L)
Cs = concentration of un-spiked sample (ug/L)
C2 = concentration of spike (ug/L)

9.1.7.4 Recovery limits for post digestion spikes for Method 6020B are 75-125%; If both the MS and post digestion spike fail, then matrix effects are confirmed.

9.2. Instrument QC

Table 2 below describes the frequency, criteria, and corrective actions for the calibration and Quality control samples.

Table 2: Calibration, Quality control and Corrective Action Summary

QC Item	Frequency	Criteria	Corrective Action
Tune validation-Mass Calibration	Daily	Within 0.1amu from mass unit	Terminate analysis, fix problem and repeat
Tune validation-Resolution Check	Daily	<0.9 amu full width at 10% peak height	Terminate analysis, fix problem and repeat
Instrument Stability Check	Daily	<5% RSD for five replicates	Terminate analysis, fix problem and repeat
Initial Calibration: Multi-point-minimum 3 stds and 1 blank	Daily	Correlation ≥0.995	Recalibrate
Initial Calibration Verification Standard (ICV)	At the beginning of the analysis	+/- 10%	Recalibrate
Continuing Calibration Verification Standard (CCV)	At the beginning and end of the analysis, and every 10 samples	Within +/- 10% of the true value	Fix the problem and reanalyze the previous 10 samples.
Initial Calibration Blank (ICB)	After ICV	must be less than ½ the LLOQ (Lower limit of Quantitation)	Terminate the analysis, correct the problem and reanalyze the previous 10 samples
Continuing Calibration Blank (CCB)	After every CCV	Less than the LLOQ	
Interference check standards (ICSA)	At the beginning of an analysis and after every 12 hours of analysis	+/-20% of the true value or +/-2 times the LLOQ, whichever is greater	If an element is consistently out of the +/-20% or +/-2 times the LLOQ, the problem should be investigated.
Internal Standards	Analyzed with every standard and sample	30-140%	Standard-terminate the analysis and recalibrate Sample- a 5X dilution is required (The analyst should check the IS response for the bracketing CCV/CCBs to see if there is a similar response to the sample. Recalibration may be required.)
Linear Range Check (LRC)	After calibration	90-110%	If LRC is not within 90-110% recovery, then the linear range is the highest point in the calibration

9.2.1. Initial Calibration Verification (ICV): Analyze an initial calibration verification solution at the beginning of the run. ICV solution must have the same acid matrix as the calibration standard and it must come from a source other than the calibration standard source. The final concentrations of the various elements are listed in Attachment 1. The results for the target elements in the initial calibration verification (ICV) must be within +/-10% of the true value. If results are outside of the specified limits, terminate the analysis, correct the problem and recalibrate the instrument. See Sec 7.2.4 for the ICV standard preparation instructions.

9.2.2. Continuing Calibration Verification (CCV): The calibration of the ICP-MS must be verified every 10 samples and at the end of the analysis run by analyzing the QC Check Solutions (CCV). The same solution used for the calibration standard 3 is used for the CCV standard. The concentration of the CCV standard must be at or near mid-range levels of the calibration. The final concentrations of the various elements are listed in Attachment 1. The results for the target compounds must be within +/-10% of the true value. If results are outside of the specified limits, terminate the analysis, correct the problem, recalibrate the instrument, and reanalyze the samples following the last good CCV. See CAL3/CCV of Sec 7.2.2 for the preparation instructions.

9.2.3. Initial and Continuing Calibration Blank (ICB/CCB):

9.2.3..1. Initial Calibration Blank (ICB) must be analyzed after the calibration curve. The value of the initial calibration verification blank must not exceed ½ LLOQ/reporting limit (RL). If it does, terminate the analysis, correct the problem, and recalibrate. The initial calibration verification blank is the same blank solution as used for the calibration blank.

9.2.3..2. Continuing Calibration Blank (CCB) must be analyzed after the calibration curve, every 10 samples and at the end of the analytical run. The result of the calibration verification blank must not exceed the LLOQ/reporting limit (RL). If it does, terminate the analysis, correct the problem, recalibrate and reanalyze the samples following the last good CCB. The calibration verification blank is the same blank solution as used for the calibration blank.

9.2.4. Spectral Interference Check (SIC) Sample (ICSA): Verify the inter-element and background corrections by analyzing the interference check solution (ICSA) at the beginning of the analysis run and after every 12 hours of analysis. The ICSA is analyzed in order to demonstrate that proper corrections are being utilized for known interferences. Analyst must evaluate this solution in order to detect trends that require corrective

actions. Pay particular attention to false positives and false negatives for elements not present in the interference check solution. Results should fall within the control limit of +/-20% of the true value or +/-2 times the LLOQ of the true value, whichever is greater. See Attachment 2 for list of elements and the corresponding concentrations in ppb. See Sec 7.2.8 for preparation instructions.

9.2.5. Linear Range Standard (LRC): the LRC standards are prepared from single element stock standards of each metal obtained from a commercial source. The LRC's are prepared by taking the appropriate volume of each stock and adding it to a 500 mL volumetric flask partially filled with 5% HNO3 / 5% HCl and diluted to the mark after all elements have been added. The tables below lists the volume of each stock solution of each metal, volume used and final concentrations:

Linear Range Check (LRC) - A			
Elements	Stock Conc (mg/L)	Volume of stock (mL)	Final Conc (mg/L)
Tl	10000	0.05	1
Be, Co, Mo, Se	10000	0.1	2
As, Cd,Sr, Ti, V	10000	0.25	5
Mn	10000	0.5	10
Ba, Cr, Pb, Cu, Ni, Zn	10000	1	20

Linear Range Check (LRC) - B			
Elements	Stock Conc (mg/L)	Volume of stock (mL)	Final Conc (mg/L)
Al	10000	10	200
Ca, Fe, Mg, Na, K	10000	25	500

The LRC standards are analyed after each calibration. If the results fall within the control limit of +/-10% of the true value, then the linear range at that concentration has been verified; if not, the linear range is the highest point in the calibration.

9.3. Instrument Performance Criteria

Prior to the analysis of any samples, the following must be performed.

9.3.1. Instrument Detection Limits (IDLs): The IDL for each analyte must be determined for each wavelength used on each instrument.

9.3.1.1. The IDLs for each analyte must be determined for each prep method. The IDL is determined by analyzing 10 replicate

analysis of reagent blank solution (5% HNO3 / 5% HCl). Analyze the ten blanks and calculate the standard deviation and the mean (use zero for the mean if the mean is negative). The IDL = mean + (3 x Std Dev). The IDL must be determined when the instrument is initially set up, when the instrument is adjusted in any way that may affect the IDL (e.g., detector replacement), and annually.

10.0. Procedure

10.1. Sample Preparation

10.1.1. For samples to be analyzed by Method or 6020B, follow digestion procedure in TestAmerica SOP No. ED-MTP-002 (Method 3005A) or ED-MTP-003 (Method 3010A) for waters and ED-MTP-005 (Method 3050B) for soils.

10.2. Calibration

10.2.1. Turn the ICP-MS on and initiate the tune screen. Start the tune screen to allow the instrument to become thermally stable before beginning to analyze the calibration standards. While the instrument is warming up, aspirate the interference check solution (or similar solution) to precondition the cones.

10.2.2. Check the tune parameters for the proper sensitivities, precision, oxides, and double charged values following the instrument manufacturer's recommendations. Refer to sections *Preparations Before Analysis*, *Plasma ON/Executing a Startup*, and *Appendix (Recommended Values for Tuning Parameters)* of the MassHunter Workstation User Guide (*Agilent Technologies 7800/7900, p/n G7201-90403).*

10.2.3. Prior to calibration, run an EPA Tune check solution. Refer to Sec. 7.2.6 for the Tune check criteria.

10.2.4. Using method *BM New Method.icpms.template* (program in the Mass Hunter Workstation software containing calibration standards, check tables, etc.), calibrate the instrument using a blank and five standards. Refer to the section *Creating a Method* in the instrument manufacturer's instructions *Agilent ICP-MS (7800/7900) MassHunter Workstation User Guide (p/n G7201-90403)*.

10.2.5. Internal standards are added to the calibration standards, blank and samples in-stream by the use of T-fitting.

10.2.6. Analyze the calibration standards and calibrate the ICP-MS in accordance with the manufacturer's recommendations and the TestAmerica Corporate SOP No. CA-Q-S-005 Calibration Curves (General).

10.2.7. The instrument must be calibrated daily or once every 24 hours and each time the instrument is set up. The correlation coefficient (r) of the calibration curve must be ≥0.995 If not, the problem must be corrected, and the instrument must be recalibrated.

10.2.8. Calibration Curve Read-Back:

10.2.8.1. Low-Level readback for aqueous samples and for Silver for soil samples – evaluate Cal 1 which is at the LLOQ for aqueous samples. The %RE (relative error) must be +/- 20% (see Sec 11.5); if not, stop the analysis and recalibrate.

10.2.8.2. Low-Level readback for soil samples except Silver- evaluate Cal 2 which is at the LLOQ for soil samples. The %RE (relative error) must be +/- 20% (see Sec 11.5); if not, stop the analysis and recalibrate.

10.2.8.3. Mid-Level readback – evaluate Cal 4. The %RE (relative error) must be +/- 10% (see Sec 11.5); if not, stop the analysis and recalibrate.

10.3. Sample Analysis

10.3.1. All sample measurements must be made within the calibration range of the instrument. Preparation of all reference materials used for calibration must be documented.

10.3.2. Typical dilutions for samples and relating QC samples:

10.3.2.1. Water, SPLP, soil, and their relating batch QC (MB, LCS, Dup, MS, MSD) samples are typically analyzed undiluted with the exception of the LCSSRM. The LCSSRM is typically analyzed at a 5X dilution but the dilution used will be dependent upon the concentrations of the specific lots. Dilutions are prepared in 5% HNO3 / 5% HCl.

10.3.2.2. TCLP and the relating batch QC samples (LCS, Dup, MS, MSD, LB) are typically diluted at a 5X prior to analysis with the exception of the TCLP method blank (MB). The TCLP method blanks (MB) for method 3010A are typically analyzed undiluted. Dilutions are prepared in 5% HNO3 / 5% HCl.

10.3.3. The samples are analyzed only after the ICV, and ICB criteria are met.

10.3.4. The samples are analyzed in a sequence as follows:

Typical Analytical Sequence
INSTRUMENT WARM-UP (approx. 30 minutes)
TUNE CHECK
STANDARDIZATION/CALIBRATION (Blank, 4 5 Cal Stds)
ICV
ICB
CRI (200.8)
ICSA
ICSAB (200.8)
LRC-A
LRC-B
Rn Chk
Rn Chk
CCV
CCB
10 Samples
CCV
CCB
10 Samples
CCV
CCB
{continue until done; after 12 hrs run ICSA}

Note: The analytical sequence must end with the analysis of the CCV and CCB. The 10 samples include all QC samples/standards with the exception of CCVs and CCBs.

10.3.5. Determine the concentration of the samples and QC items using the procedures of Section 11.

10.3.5.1. The amount of sample digestate needed to prepare the desired dilution is determined from the following equation:

$$\text{V digest} = \frac{Vf\ vol}{DF}$$

Where:
Vf vol = final volume of diluted sample (mL)
V digest = volume of sample digestate used to make the dilution (mL)
DF = Dilution Factor

10.3.5.2. The dilution factor is calculated as follows:

$$DF = \frac{Vf\ vol}{V\ digest}$$

Where: Vf vol = final volume of diluted sample extract (mL)
V digest = volume of sample extract used to make the dilution (mL)

Note: The following examples are based on a final volume of 100mL. It may be more convenient to prepare dilutions at smaller final volumes.

EXAMPLE

A sample digestate is analyzed and one of the target analytes exceeds the linear range of the ICP-MS. 1.0mL of the digestate is added to a 100mL volumetric flask and the extract brought up to volume with 5% HNO3 / 5% HCl. What is the dilution factor?

$$DF = \frac{100ml}{1.0ml} = 100$$

Some samples may require multiple dilutions; that is, a dilution of a dilution will have to be made. In this case, the final dilution factor is the product of the individual dilutions.

11.0. Calculations / Data Reduction

11.1. Accuracy:

$$\text{ICV / CCV, LCS \% Recovery} = \frac{\text{observed concentration}}{\text{known concentration}} \times 100$$

$$\text{MS \% Recovery} = \frac{\text{(spiked sample) - (unspiked sample)}}{\text{spiked concentration}} \times 100$$

11.2. Precision (RPD):

$$\text{Matrix Duplicate (MD)} = \frac{|\text{orig. sample value - dup. sample value}|}{[(\text{orig. sample value + dup. sample value})/2]} \times 100$$

11.3. Aqueous and Leachate Samples:

Leachate samples are routinely reported in mg/L while the ICP-MS is routinely calibrated in ug/L. If the results are reported in ug/L, the conversion factor is omitted from the calculation.

11.3.1. The concentration of the target analyte in liquid samples is calculated as follows:

Concentration (mg/L) = $ug/L(from\ pr\,\mathrm{int}\,out)\otimes\frac{F}{V}\otimes DF\otimes\frac{1mg}{1000ug}$

Where: F = final volume of the sample digestate (L) -usually 50mL
V= volume of sample digested (L)
DF = dilution factor

11.3.2. The Reporting Limit (RL) of the target analyte in liquid samples is calculated as follows:

Concentration(mg/L) = $RL_{table1}\otimes\frac{F}{V}\otimes DF\otimes\frac{1mg}{1000ug}$

Where: $RL_{table\ 1}$ = reporting limit from this SOP (ug/L)
F = final volume of the sample digestate (L)
V= volume of sample digested (L)
DF = dilution factor

The Table 1 Reporting Limits assumes:
F = 50ml
V = 50ml
DF = 1

11.4. Soil/Solid Samples

Soils and solids are routinely reported in mg/kg while the ICP-MS is routinely calibrated in ug/L. If the results are reported in ug/kg, the conversion factor is omitted from the calculation.

11.4.1. The concentration of the target analyte in soil and solid samples is calculated as follows:

Concentration (mg/kg,dw) = $ug/L(from\ pr\,\mathrm{int}\,out)\otimes\frac{F}{W\otimes solids}\otimes DF\otimes\frac{1mg}{1000ug}$

Where: F = final volume of the sample digestate (L)
W = volume of sample digested (kg)
DF = dilution factor
solids = decimal equivalent of the percent solids (percent solids/100)

For example, if the percent solid is 85%, the decimal equivalent is 0.85; if the %solid is 100%, the decimal equivalent is 1.0.

11.4.2. The Reporting Limit (RL) of the target analyte in soil/solid samples is calculated as follows:

$$\text{Concentration(mg/kg,dw)} = RL_{table1} \otimes \frac{0.0010kg}{W \otimes solids} \otimes \frac{F}{0.100L} \otimes DF$$

Where:
$RL_{table\ 1}$= reporting limit from Table 1 of this SOP
W = weight of sample digested (kg)
F = final volume of the sample digestate (L)
V = volume of sample digested (L)
DF = dilution factor
solids = decimal equivalent of the percent solids (percent solids/100)
dw = dry weight

The Table 1 Reporting Limit assumes:
F = 0.100L
DF = 1
W = 0.0010kg (1.0g)
solids = 1.0

NOTE: All dry weight corrections are made in LIMS at the time the final report is prepared.

11.5. Relative Error (%RE):

% Relative Error = (100) (MC-TC)/TC

MC = Measured Concentration of the calibration standard
TC = True Concentration for the calibration standard

11.6. Data Reduction:

11.6.1. All data is recorded directly in TALS' Analyst Desktop II program.

11.6.2. Record standard preparations in TALS Reagent module.

11.6.3. Sample and standard preparations must be documented in the Analyst Desktop II program located in TestAmerica Laboratory System (TALS). The analyst must enter the following information: Source standard, Initial and final sample volume, spike name and amount used, all reagents and their corresponding lot numbers, creation dates and expiration dates.

11.6.4. Complete the Data Review Checker (DRC) in TALS: Prior to data submission (i.e., prior to 1st leveling data in the Analyst Desktop II module in TALS), the analyst must execute the DRC (Data Review Checker) program in the applicable analytical batch.

11.6.4.1. Open the analytical batch and click on the Edit tab above to enter the Edit Mode.

11.6.4.2. Press F8 command key on the computer keyboard or right click anywhere on the batch worksheet then click on 'Run checklist.'

11.6.4.3. Acknowledge by filling in responses to all unacknowledged findings.

11.6.4.3.1. Highlight the checklist with findings, then highlight the associated list of findings; right click and choose 'Acknowledge Item.'

11.6.4.3.2. Fill in appropriate comments in the response box, then hit 'OK.'

11.6.4.3.3. Acknowledge all Finding items in the 'Manual Batch Checklist' except for the *"2nd Level review complete?"* this is to be completed by the 2nd level reviewer.

12.0. Method Performance

12.1. Method/Instrument Detection Limit Study (MDL)

The method detection limit (MDL) is the lowest concentration that can be detected for a given analytical method and sample matrix with 99% confidence that the analyte is present. The MDL is determined according to the laboratory's MDL procedure in Section 19 (Test Methods and Method Validation) of TestAmerica Edison's Quality Assurance Manual (ED-QA-LQM). MDLs reflect a calculated (statistical) value determined under ideal laboratory conditions in a clean matrix, and may not be achievable in all environmental matrices. The laboratory maintains MDL studies for analyses performed; these are verified at least annually unless method requirements require a greater frequency.

12.2. Demonstration of Capabilities

For DOC procedure refer to Section 19 in the most current revision of TestAmerica Edison's Quality Assurance Manual (ED-QA-LQM).

12.3. Training Requirements

Refer to TestAmerica SOP No. ED-GEN-022, Training, for the laboratory's training program.

12.4. Lower-Limit of Quantitation (LLOQ):

12.4.1. LLOQ verification: prior to analyzing samples under method 6020B, after any change that may affect the LLOQ, and quarterly, the LLOQ must be verified. The LLOQ is initially verified by the analysis of 7 replicate samples spiked at the LLOQ. For aqueous, prep method 3005A and 3010A, use the same concentration as Cal 1 (see Sec 7.2.2 for preparation procedures). For LLOQ soil except Silver, preparation method 3050B, use the same concentration as Cal 2 (see Sec 7.2.2 for preparation procedures). For Silver, spike 2mL of the 6020B RL standard into a clean 50mL digestion cup and prepare using method 3050B. The seven replicates must be digested prior to analysis. See SOPs ED-MTP-002, ED-MTP-003 and ED-MTP-005 for digestion instructions. The LLOQ is verified when the mean recovery of the 7 replicates is +/-35% of the true value and the RSD $\leq$ 20%.

12.4.2. LLOQ on-going verification: the LLOQ is re-verified quarterly by the analysis of a method blank (MB) and 1 blank sample (deionized water) spiked at the LLOQ. Use the same concentrations as CAL 1 (3005A and 3010A) and Cal 2 (3050B) for soil, except Silver. For Silver, spike 2mL of the 6020B RL standard into a clean 50mL digestion cup and prepare using method 3050B. See Sec 7.2.2.2 for the preparation procedures. The MB and LLOQ samples must be digested prior to analysis. See SOPs ED-MTP-002, ED-MTP-003 and ED-MTP-005 for digestion instructions. The LLOQ is re-verified if the MB result is +/- 0.5 times the reporting limit for each analyte and when the recovery of the LLOQ sample is +/-35% of the true value.

13.0. Pollution Control

It is TestAmerica's policy to evaluate each method and look for opportunities to minimize waste generated (i.e., examine recycling options, ordering chemicals based on quantity needed, preparation of reagents based on anticipated usage and reagent stability). Employees must abide by the policies in Section 13 of the Corporate Environmental Health and Safety Manual (CW-E-M-001) for "Waste Management and Pollution Prevention."

14.0. Waste Management

14.1. Waste management practices are conducted consistent with all applicable rules and regulations. Excess reagents, samples and method process wastes are disposed of in an accepted manner. Waste description rules and land disposal restrictions are followed. Waste disposal procedures are incorporated by reference to TestAmerica SOP No. ED-SPM-008 (Laboratory Waste Disposal Procedures).

14.2. The following waste streams are produced when this method is carried out.

- Digested Samples: Corrosive Acid- Materials that are not above regulatory limits will be submitted for elementary neutralization. The neutralization system consists of a 55-gallon poly open top container. There is also a local snorkel exhaust for venting any noxious odors. Once the liquid is transferred into the container, the contents are sparged with air and a representative pH is measured and the amount of material is recorded in the discharge log. The pH value is recorded in the discharge log, and the liquid is neutralized to a pH of 6-9 SU using food grade sodium bicarbonate. Once the secondary pH has been taken, confirmed and recorded, the discharge valve is opened to the municipal sewer system and the neutralization process is complete. The date, type of waste stream, and volume discharged are recorded on a discharge log.

- Samples above regulatory limits and expired RCRA metals standards (Waste Corrosive Liquid, Acidic, Inorganic, n.o.s.) are collected in satellite accumulation and sent off site through a Waste disposal vendor.
  TSDF under Veolia Profile Number: 590598

- Soil Retain Samples - These containers are stored in the main sample-receiving laboratory cooler until they are designated for disposal. Once designated for disposal, the samples are transferred to the waste disposal area and placed into cubic-yard disposal containers, e.g., Jupiter-boxes. This waste is shipped directly to the TSDF under Veolia Profile Number 402535 and is subject to incineration.

15.0. References / Cross-References

15.1. Test Methods for Evaluating Solid Waste, Physical/Chemical Methods, 3rd ed.,U.S. Environmental Protection Agency, SW846 Manual, Method 6020A, Revision 1, February 2007.

15.2. TestAmerica Edison SOP ED-MTP-005, *Hot Block Digestion of Sediments and Sludges and Soils Using SW846 Method 3050B,* most current revision.

15.3. TestAmerica Edison SOP No. ED-MTP-003, *Digestion of Water and Wastewater Samples for Analysis by ICP and ICP-MS, SW846 Method No. 3010A,* most current revision.

15.4. TestAmerica Edison SOP ED-MTP-002, Digestion of Water and Wastewater Samples for Analysis by ICP and ICPMS, SW846 Method 3005A, most current revision

15.5. TestAmerica Corporate SOP No. CA-Q-S-005, *Calibration Curves (General)*, most current revision.

15.6. TestAmerica Edison Document No. ED-QA-LQM, *Laboratory Quality Manual*, most

current revision.

15.7. TestAmerica Edison SOP ED-GEN-022, *Training*, most current revision.

15.8. TestAmerica Corporate Environmental Health and Safety Manual, Document No. CW-E-M-001, most current revision.

15.9. Agilent ICP-MS (7800/7900) MassHunter Workstation User Guide, p/n G7201-90403, Rev. A, June 2017.

16.0. Method Modifications:

None

17.0. Attachments

Attachment 1: Standards and Matrix Spiking concentrations
Attachment 2: Interference Check standard elemental concentrations

18.0. Revision History

- Revision 10, dated 16 June 2022
  - Sec 1.1, Table 1 and throughout the SOP, revised to include digestion method SW846 3005A and SOP# ED-MTP-002 as prep method reference.
  - Sec 1, Table 1: Revised RL for Ag to 0.40 mg/kg.
  - Sec 14.2: Updated waste disposal procedures.
  - Sec 15.4: Added SOP# ED-MTP-002 to the list of references; adjusted subsequent sections accordingly.
  - Sec 10.2.8 and 12.4: Added information related to Ag QC spiking for LLOQ.

- Revision 9, dated 29 September 2021
  - Sec. 7.2.1 and 7.2.3: Revised the concentration of TA-CAL2 to 2,000 mg/L to reflect the concentration in the COA for the standard.
  - Sec. 7.2.4: Revised the volume of TA-CAL2-SS added in the preparation of the ICV working standard to 2.0 ml to reflect actual laboratory practices.

- Revision 8, dated 10 June 2020
  - Updated SOP header to Eurofins logo.
  - Throughout the SOP removed all method 6020A references.
  - Throughout the SOP removed all references to 2%HNO_3/0.5%HCl and replaced with 5%HNO_3/5%HCl.
  - Sec 1.1, Table 1: removed references to typical dilutions. Updated the RLs for Selenium
  - Sec 6.1: Added two ICPMS instruments, Agilent 7800 & 7900. Removed reference to resolution and grating. Added instrument mass range. Updated the models of the heat exchanger and autosampler.

- Sec 7.1.6: Added preparation instructions for 5%HNO_3/5%HCl.
- Sec 7.2.1: Replaced Inorganic Ventures with CPI International as the vendor for the stock cal stds. Updated all stock standards to reflect this change.
- Sec 7.2.2: Updated preparation instructions for the working cal standards.
- Sec 7.2.2.1: Added preparation instructions for MS-Cal-Int.
- Sec 7.2.3: Updated the Initial calibration verification stock standards.
- Sec 7.2.4: Updated preparation instructions for the ICV working standard.
- Sec 7.2.4.1: Added preparation instructions for MS-ICV-Int.
- Sec 7.2.6: Updated preparation instructions for the Internal standard. Changed recommended mass for Ca from 44 to 40. Updated typical referenced internal std for As & Se from Ge to Sc. Updated typical analysis mode used for Iron, Beryllium, and Boron.
- Deleted Sec 7.2.7, LLICV; subsequent sections adjusted accordingly.
- Sec 7.2.7: Replaced Inorganic Ventures with CPI International as the vendor for the stock ICSA standard. Added stock concentrations for ICSA.
- Sec 7.2.8: Updated preparation instructions for working ICSA.
- Sec 9.1.2: Updated preparation instructions for the LCS water samples.
- Sec 9.1.4.1: Updated preparation instructions for the aqueous matrix spikes (MS).
- Deleted Sec 9.2.4, LLICV/LLCCV; subsequent sections adjusted accordingly.
- Sec 9.2.5: Updated preparation instructions for Linear Range Standard (LRC), solutions (LRC)-A and (LRC)-B. Deleted (LRC)-C.
- Deleted Sec 9.3.2, Linear Dynamic Range.
- Sec 10.2.2: Added reference material, MassHunter Workstation User Guide, for the ICPMS instruments 7800 & 7900. Removed references to ICPMS1&2 (includes Chemstation software and relating Operation Manuals) from this SOP.
- Sec 10.2.4: Added instrument method program and reference material, MassHunter Workstation User Guide, for the ICPMS instruments 7800 & 7900.
- Sec 10.2.8.1 & 10.2.8.2: Added Low-Level readback evaluation instructions for aqueous and soil samples.
- Sec 10.2.8.3: Updated Mid-Level readback evaluation instructions.
- Sec 10.3.2: Updated typical dilution instructions.
- Sec 10.3.4: Updated Typical Analytical Sequence table to reflect current laboratory practices.
- Sec 11.5: Added %Relative Error calculation equation.
- Deleted Sec 12.4 LLQC; subsequent sections adjusted accordingly.
- Sec 12.4.1: Updated preparation instructions for LLOQ.
- Sec 12.4.2: Added instructions for the On-going LLOQ verification.
- Deleted references, ICPMS Operator's Manual, Sec 15.2, 15.9, 15.10, not applicable.
- Sec 15.8: Added reference for the Agilent ICPMS 7800/7900 User Guide.
- Sec 18.0, Attachment 1: Updated calibration, ICV, CCV concentrations; removed dilution instructions.
- Sec 18.0, Attachment 2: Updated ICSA analyte list and concentrations.

- Revision 7, dated 3 July 2018
  - Sec 1.1: Added Method 6020B to the list of methods applicable to this SOP
  - Throughout the SOP removed all references to Reagent Water and replaced with

2%HNO_3/0.5%HCl
- Sec 7.2 & Sec 7.2.4: Updated the ICV preparation frequency for method 6020B to daily.
- Sec 7.2.8 & 7.2.9: Renamed "ICP Interference Check Solutions" and 'Working Interference Standards" to Spectral Interference check solution (SIC).
- Sec 7.2.9: Added ICSA preparation instructions for method 6020B
- Sec 9.1 Table & Sec 9.1.1.2: Added MB control limit criteria for method 6020B
- Sec 9.1.6.5: Added serial dilution control limit criteria for method 6020B
- Sec 9.1.7.4: Added PDS control limit criteria for method 6020B
- Sec 9.2 Table 2: Added calibration curve correlation coefficient criteria, ICB/CCB criteria, Internal Standard recovery criteria, and Linear Range Check control criteria and frequency for Method 6020B.
- Sec 9.2.3: Added ICB and CCB control criteria for method 6020B
- Sec 9.2.6: Added LRC preparation and control limit criteria for method 6020B
- Sec 9.3.2.2: Added Linear Range check (LRC) to the Instrument Performance Criteria.
- Sec 10.2.7: Added calibration curve correlation coefficient criteria for 6020A and 6020B
- Sec 10.2.8: Added calibration curve read-back instructions for method 6020B
- Sec 10.3.4: added LRC-A, LRC-B, LRC-C to the typical Analytical Sequence Table.
- Sec 11.5.4: Removed the Metals Data Review Checklist. Added instructions to complete the Data Review Checker (DRC) in TALS.
- Sec 12.5: Added LLOQ standard preparation and criteria information.
- Remove Sec 15.11 – Removed reference to WI CA-Q-WI-044, WI not applicable.
- Attachment 1: Added LLQC and LLOQ on this Table.

- Revision 6, dated 27 April 2018
  - Sec 7.2.11.1: Added Magnesium as one of the working tune check standard constituents.
  - Sec 9.1.2: Revised to clarify the acceptance criteria used in the COA when evaluating recoveries for LCS in soil matrix.

- Revision 5, dated 29 November 2016
  - Sec 7.1.6: removed ICPMS Reagent Dilution logbook and replaced with TALS Reagent Module.
  - Sec 7.2.1: added calibration stock standards STLNJ-STD-1 & STLNJ-STD-2
  - Sec 7.2.2: added updated preparation instructions for Cal1 to reflect lab practices
  - Sec 7.2.6: added typical analysis mode for Selenium, i.e., Hydrogen. Updated the resolution check criteria to reflect lab practices
  - Sec 11.5.4: removed reference to checklist CA-Q-WI-004 and replaced with EDS-WI-146
  - Attachment 1: updated Cal1 standard concentrations; removed the linear range column

- Revision 4, dated 13 November 2015
  - Throughout the SOP removed all reference to SOP# ED-MTP-002 (SW method 3005).

- Sec 1.1: Revised RL for Ag to reflect actual laboratory's RL.
- Sec 7.2.1: Removed calibration standard solution STLNJ-STD-1 and STLNJ-STD-2 (Cal 1) and added standard TA-88.
- Sec 7.2.2: Revised preparation procedure for CAL1; replaced calibration standard solution STLNJ-STD-1 and STLNJ-STD-2 with standard TA-88. Deleted prep instructions for ME_Se_1ppm.
- Sec 7.2.6: Updated the preparation instruction of the working internal standard solution to include Triton X-100.
- Sec 9.1 and 9.1.5: Added Matrix Spike Duplicate (MSD) to the list of Sample QC. Subsequent sections adjusted accordingly.
- Sec 11.5.4: Updated the metals data review checklist form number.
- Sec 15: Removed reference to SOP ED-MTP-002 (method 3005A) in Sec 15.4; Deleted Data review checklist WI# EDS-WI-007 in Sec 15.10, checklist is referenced in the applicable digestion SOP; subsequent sections adjusted accordingly.
- Sec 15.11: Replaced Data review checklist (EDS-WI-123) with the standardized Data review checklist WI# CA-Q-WI-043.

- Revision 3, dated 19 August 2013
  - Sec 1 & 12: Revised LQM section references to reflect the most current LQM revision.
  - Sec 7.2.11: Added EPA Tune Check stock standard including preparation and analysis instructions.
  - Sec 10.2.2 & Sec 10.2.4: Added the Operating Manual and Tuning & Application Handbook calibration information for the Mass Hunter Software (ICPMS2)
  - Sec 11.5.4: Added requirement to complete the Initial Calibration Data Review checklist (EDS-WI-123) prior to data submission.
  - Sec 15.11 & 15.12: Added reference information for the Tuning & Application Handbook and MassHunter Workstation Operator's Manual.
  - Sec 15.13: Added reference information for TestAmerica Edison Metals Initial Calibration Data Review checklist.

- Revision 2, dated 23 August 2011
  - Sec 7.2.2 Revised preparation procedure for CAL 1; changed added amount of STLNJ-STD-1 and STLNJ-STD-2 from 2 ml to 4ml

- Revision 1, dated 10 March 2011
  - Sec 1.1 Table 1: Revised RL for soil and water
  - Sec 7.2.2: Revised preparation procedure for CAL1; added prep for ME_Se_1ppm.
  - Attachment 1: Revised the spiking concentration for the calibration, LLICV/LLCCV and MS concentration.

Attachment 1

STANDARDS AND MATRIX SPIKING CONCENTRATIONS

Name	Mass	Cal-1	Cal-2	Cal-3	Cal-4/CCV	Cal-5	ICV	*Water*	*Soil*
		(ug/L)	(ug/L)	(ug/L)	(ug/L)	(ug/L)	(ug/L)	*(ug/L)*	*(mg/Kg)*
Be	9	0.8	4	50	100	200	80	*25*	*5*
B	11	80	400	125	250	500	200	*500*	*100*
Na	23	200	1000	12500	25000	50000	20000	*2500*	*500*
Mg	24	200	1000	12500	25000	50000	20000	*2500*	*500*
Al	27	40	200	12500	25000	50000	20000	*2500*	*500*
K	39	200	1000	12500	25000	50000	20000	*2500*	*500*
Ca	40	200	1000	12500	25000	50000	20000	*2500*	*500*
Ti	47	4	20	50	100	200	80	*50*	*10*
V	51	4	20	50	100	200	80	*50*	*10*
Cr	52	4	20	50	100	200	80	*50*	*10*
Mn	55	8	40	50	100	200	80	*250*	*50*
Fe	56	120	600	12500	25000	50000	20000	*2500*	*500*
Co	59	4	20	50	100	200	80	*25*	*5*
Ni	60	4	20	50	100	200	80	*50*	*10*
Cu	63	4	20	50	100	200	80	*50*	*10*
Zn	66	16	80	125	250	500	200	*250*	*50*
As	75	2	10	50	100	200	80	*50*	*10*
Se	78	2.5	12.5	50	100	200	80	*50*	*10*
Sr	88	4	20	50	100	200	80	*50*	*10*
Mo	95	4	20	50	100	200	80	*50*	*10*
Ag	107	2	10	25	50	100	40	*25*	*5*
Cd	111	2	10	50	100	200	80	*25*	*5*
Sn	118	16	80	50	100	200	80	*50*	*10*
Sb	121	2	10	50	100	200	80	*25*	*5*
Ba	137	4	20	50	100	200	80	*50*	*10*
Tl	205	0.8	4	50	100	200	80	*20*	*4*
Pb	208	1.2	6	50	100	200	80	*25*	*5*

Attachment 2

ELEMENT	ICSA (ug/L)
Sodium	100000
Magnesium	100000
Aluminum	100000
Potassium	100000
Calcium	100000
Titanium	2000
Iron	100000
Molybdenum	2000
Chloride	1000000
Carbon	200000
Phosphorus	100000
Sulfur	100000

Title: Mercury Analysis for Solid and Semisolid Waste Samples using the Leeman Mercury Analyzer (Cold Vapor Technique) by SW846 Method 7471B

Once printed, this is considered an uncontrolled document

Approvals (Signature/Date):

Laura Demone	08/11/22	Donald Evans	08/11/22
Laura Demone Department Manager	Date	Donald Evans Operations Manager	Date
	08/11/22		08/11/22
Carl Armbruster Quality Assurance Manager	Date	Mark Acierno Laboratory Director	Date
Dan Helfrich Healthy & Safety Manager Coordinator	08/11/22 Date		

1.0 Scope and Application

1.1. Analytes, Matrix(s), and Reporting Limits

SW846 Method 7471B is applicable to the determination of total mercury (organic and inorganic) in soils, sediments, bottom deposits, and sludge-type materials. All samples must be digested prior to analysis. If this digestion procedure is not sufficient to dissolve a specific matrix type or sample, then this method is not applicable for that matrix.

The typical detection limit using a 0.6 gram sample size is 0.017 mg/Kg Hg.

On occasion clients may request modifications to this SOP. These modifications are handled following the procedures outlined in Sections 7 (Review of Work Request) and 19 (Test Methods and Method Validation) of TestAmerica Edison's Quality Assurance Manual (ED-QA-LQM).

2.0 Summary of Method

The sample is digested as described in this SOP and is analyzed using cold vapor atomic absorption. The mercury is reduced to the elemental state and aerated from solution in a closed system. The mercury vapor passes through a cell positioned in the light path of an atomic absorption spectrophotometer. Absorbance at 253.7-nm is measured as a function of mercury concentration.

3.0 Definitions

For a complete list of definitions refer to Appendix 2 in the most current revision of TestAmerica Edison's Quality Assurance Manual (ED-QA-LQM).

4.0 Interferences

4.1 Potassium permanganate is added to eliminate possible interferences from sulfide.

4.2 Copper may also be a potential interference although no effect has been observed for samples containing up to 10 mg/Kg Copper.

4.3 Samples high in chlorides require additional permanganate (as much as 25 mL) because, during the oxidation step, chlorides are converted to free chlorine, which also absorbs radiation of 254 nm. Care must therefore be taken to ensure that free chlorine is absent before the mercury is reduced and swept into the cell. This may be accomplished by using an excess of hydroxylamine hydrochloride reagent (25 mL).

4.4 Certain volatile organic materials that absorb at 253.7 nm may also cause interference. The analysis of the undigested sample should determine if this type of interference is present.

5.0 Safety

Employees must abide by the policies and procedures in the Corporate Environmental Health and Safety Manual (CW-E-M-001) and this document. This procedure may involve hazardous material, operations and equipment. This SOP does not purport to address all of the safety problems associated with its use. It is the responsibility of the user of the method to follow appropriate safety, waste disposal and health practices under the assumption that all samples and reagents are potentially hazardous. Safety glasses, gloves, lab coats and closed-toe, nonabsorbent shoes are a minimum.

5.1. Specific Safety Concerns or Requirements

Samples that contain high concentrations of carbonates or organic material or samples that are at elevated pH can react violently when acids are added.

5.2. Primary Materials Used

The following is a list of the materials used in this method, which have a serious or significant hazard rating. **Note: This list does not include all materials used in the method. The table contains a summary of the primary hazards listed in the MSDS for each of the materials listed in the table.** A complete list of materials used in the method can be found in the reagents and materials section. Employees must review the information in the MSDS for each material before using it for the first time or when there are major changes to the MSDS.

Material (1)	Hazards	Exposure Limit (2)	Signs and symptoms of exposure
Mercury (1,000 PPM in Reagent)	Oxidizer Corrosive Poison	0.1 mg/M^3 Ceiling (Mercury Compounds)	Extremely toxic. Causes irritation to the respiratory tract. Causes irritation. Symptoms include redness and pain. May cause burns. May cause sensitization. Can be absorbed through the skin with symptoms to parallel ingestion. May affect the central nervous system. Causes irritation and burns to eyes. Symptoms include redness, pain, and blurred vision; may cause serious and permanent eye damage.
Sulfuric Acid	Corrosive Oxidizer Dehydrator Poison	1 mg/M^3-TWA	Inhalation produces damaging effects on the mucous membranes and upper respiratory tract. Symptoms may include irritation of the nose and throat, and labored breathing. Symptoms of redness, pain, and severe burn can occur. Contact can cause blurred vision, redness, pain and severe tissue burns. Can cause blindness.
Nitric Acid	Corrosive Oxidizer Poison	2 ppm-TWA 4 ppm-STEL	Nitric acid is extremely hazardous; it is corrosive, reactive, an oxidizer, and a poison. Inhalation of vapors can cause breathing difficulties and lead to pneumonia and pulmonary edema, which may be fatal. Other symptoms may include coughing, choking, and irritation of the nose, throat, and respiratory tract. Can cause redness, pain, and severe skin burns. Concentrated solutions cause deep ulcers and stain skin a yellow or yellow-brown color. Vapors are irritating and may cause damage to the eyes. Contact may cause severe burns and permanent eye damage.
Hydrochloric Acid	Corrosive Poison	5 PPM-Ceiling	Inhalation of vapors can cause coughing, choking, inflammation of the nose, throat, and upper respiratory tract, and in severe cases, pulmonary edema, circulatory failure, and death. Can cause redness, pain, and severe skin burns. Vapors are irritating and may cause damage to the eyes. Contact may cause severe burns and permanent eye damage.

Material (1)	Hazards	Exposure Limit (2)	Signs and symptoms of exposure
Potassium Permanganate	Oxidizer	5 Mg/M^3 for Mn Compounds	Causes irritation to the respiratory tract. Symptoms may include coughing, shortness of breath. Dry crystals and concentrated solutions are caustic causing redness, pain, severe burns, brown stains in the contact area and possible hardening of outer skin layer. Diluted solutions are only mildly irritating to the skin. Eye contact with crystals (dusts) and concentrated solutions causes severe irritation, redness, and blurred vision and can cause severe damage, possibly permanent.
1 – Always add acid to water to prevent violent reactions.			
2 – Exposure limit refers to the OSHA regulatory exposure limit.			

6.0 Equipment and Supplies

6.1. Instrumentation

6.1.1. Leeman Laboratories Inc. Hydra IIAA Automated Hg Analyzer

6.1.2. Computer and Monitor with Leeman Envoy software.

6.1.3. Top loader balance, 300gm capacity, and minimum sensitivity of ±1.0 mg

6.1.4. Hotblock digestor: Adjustable and capable of maintaining a temperature between 95 ±3°C.

6.2. Supplies

6.2.1 Pipettes and tips in various sizes

6.2.2 100 ml volumetric flasks

6.2.3 15 ml sample cups

6.2.4 Pump tubing:

- Sample, viton, blue tab
- Reductant, red tab
- Drain, blue tab

- Rinse, Black tab

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6.2.5 Drying Tube – Purchased pre-packed with Magnesium Perchlorate from Leeman Labs. Located prior to the optical cell.

6.2.6 Nitrogen or Argon supply - capable of producing 80 PSI.

7.0 Reagents and Standards

Storage requirements: store at room temperature

Life of Reagent: Concentrated acids: refer to manufacturer's instructions
Laboratory prepared reagents and diluted acids: one year

Record reagent preparations in the TALS Reagent Module.

7.1 Reagents

7.1.1 Nitric acid - Concentrated (Trace Grade or Equivalent); store at room temperature; for stability information, refer to manufacturer's instructions

7.1.2 Hydrochloric acid-Concentrated (Trace Grade or Equivalent); store at room temperature; for stability information, refer to manufacturer's instructions.

7.1.3 Potassium Permanganate (ACS Grade); store at room temperature; for stability information, refer to manufacturer's instructions.

7.1.4 Sodium Chloride (analytical reagent grade); store at room temperature; for stability information, refer to manufacturer's instructions.

7.1.5 Hydroxylamine Hydrochloride (ACS Grade); store at room temperature; for stability information, refer to manufacturer's instructions.

7.1.6 Stannous Chloride (ACS Grade); store at room temperature; for stability information, refer to manufacturer's instructions.

7.1.7 Deionized water - 18 megohm minimum

7.1.8 10% Hydrochloric Acid- Cautiously add 200 ml of concentrated HCl to a container and bring to final volume of 2 liters with deionized water. Store at room temperature; stable for one year.

7.1.9 Stannous chloride solution - Add 50 g of $SnCl_2$ to 500 ml 10% HCl solution. Store at room temperature; stable for one year.

7.1.10 Sodium chloride/Hydroxylamine Hydrochloride solution - Dissolve 120 g of NaCl and 120 g of hydroxylamine hydrochloride in deionized water and dilute to 1 liter using deionized water. Store at room temperature; stable for one year.

7.1.11 Potassium permanganate ($KMnO_4$) 5% solution w/v - Dissolve 100 g of $KMnO_4$ in deionized water and dilute to 2 liters using deionized water. Store at room temperature; stable for one year.

7.1.12 0.15% Nitric Acid- Add approximately 5 liters of deionized water into an 8 liter carboy container. Cautiously add 12mL of concentrated HNO_3 and bring the final volume up to 8 liters with deionized water.

7.2 Standards

Storage requirements: all standards are stored at room temperature

Shelf-life:	Stock standards – refer to manufacturer's instructions Intermediate standards – made fresh daily Working standards – made fresh daily (Note: expiration date must not go beyond the expiration date of the source stock).
Concentration:	see Attachment 1 for example certificates of analysis (COA) of Hg standards listed below. The COA lists the manufacturer's Lot number, certified concentration and shelf life.

Document standard preparation in TALS, see Sec 11.5.3.

7.2.1 Stock Mercury Calibration (10 ppm Hg) - Purchase from SCP Science; store at room temperature; for stability information, refer to manufacturer's instructions.

7.2.2 Secondary Stock Mercury Calibration Standard (10 mg/L Hg) - Purchase from Inorganic Ventures; store at room temperature; for stability information, refer to manufacturer's instructions.

7.2.3 Intermediate Calibration Standard (DCAL-Int), 100 ug/L Hg: Dilute 1 ml of Hg calibration stock standard solution (Sec 7.2.1) to 100 ml with 0.15% HNO_3.

7.2.4 Intermediate Initial Calibration Verification Standard (DQCS-Int), 100 ug/L Hg: Dilute 1 ml of Hg stock Calibration Verification standard, 10ppm Hg (Sec 7.2.2) solution to 100 ml with 0.15% HNO_3.

7.2.5 Calibration Standards: Use six 50 ml hotblock cups to prepare the standards. Spike the calibrations standards cups as follows:

Calibration Standard	DCAL-Int Spike Volume (mL)	0.15% HNO_3 (mL)	Final Conc (ug/L)
Std1 (Cal Blk)	0	5	0
Std 2	0.1	4.9	0.2
Std 3	0.5	4.5	1
Std 4	1	4	2
Std 5	2.5	2.5	5
Std 6	5	0	10

Digest the standards following Sample preparation in Section 10.1.

7.2.6 Initial Verification Standard (ICV) 5.0 ppb: Using a hotblock cup, add 2.5 mL of 0.15 HNO_3 and 2.5 ml of DQCS-INT, (Sec 7.2.4). Digest the standard following Section 10.1. After digestion, the ICV will contain 5.0 ppb of Hg.

7.2.7 Continuing Calibration Verification Standard (CCV) 5.0 ppb: Follow the preparation instructions for Std 5 (see Sec 7.2.5)

8.0 Sample Collection, Preservation, Shipment and Storage

Sample container, preservation techniques and holding times may vary and are dependent on sample matrix, method of choice, regulatory compliance, and/or specific contract or client requests. Listed below are the holding times and the references that include preservation requirements.

Matrix	Sample Container	Min. Sample Size	Preservation	Holding Time [1]	Reference
Soils	Plastic Glass	5 grams	Cool 4 ± 2ºC	28 Days	SW846 Method 7471B

[1] Inclusive of digestion and analysis.

Samples are to be analyzed without drying. A separate procedure is used to determine the percent solids in the sample.

For sample homogenization procedures refer to TestAmerica Edison SOP ED-GEN-007 (Subsampling).

9.0 Quality Control

9.1. **Sample QC** - The following quality control samples are prepared with each batch of samples.

Quality Controls	Frequency	Control Limit
Method Blank (MB)	1 in 20 or fewer samples	< RL; 5% of the regulatory limit; 5% of the measured concentration in the sample
Laboratory Control Soil Sample Reference Material (LCSSRM) in soil samples	1 in 20 or fewer samples	Vendor's certified limit
Matrix Duplicate (DUP)[1]	1 in 20 or fewer samples	If original sample and dup are both ≥ 5X RL, then 20% RPD. If original sample and duplicate are less than the RL, the RPD is not calculated; otherwise ±RL.
Matrix Spike (MS)[1]	1 in 20 or fewer samples	80-120%
Matrix Spike Duplicate (MSD)[2]	1 in 20 or fewer samples	80-120%; If original sample and dup are both ≥ 5X RL, then 20% RPD. If original sample and duplicate are less than the RL, the RPD is not calculated; otherwise ±RL.
Serial Dilution (SD)	1 in 20 or fewer samples	±10%

[1] The sample for DUP and MS are randomly selected, unless specifically requested by a client; Use the same environmental sample for the matrix spike and matrix duplicate sample whenever possible. If insufficient sample amount is available, another environmental sample may be used for the duplicate sample.
[2] A MSD is prepared and analyzed when requested by the client.

9.1.1. Method Blank: One laboratory method blank will be analyzed with each batch of samples prepared together (not to exceed 20 samples). The method blank is used to identify possible contamination during acid digestion. Results must be less than the RL, 5% of the regulatory limit for that analyte, or 5% of the measured concentration in the sample. If the analyte concentration in the method blank is above this control limit, the batch must be prepared again and the samples reanalyzed.

9.1.2. Laboratory Control Sample Soil Reference Material (LCSSRM: A laboratory control sample must be analyzed with each group of samples digested. For solid matrices, a vendor supplied solid matrix with certified values is carried through the same preparation procedure as the samples. The results of the solid LCS must fall within the 'QC Performance acceptance limits' of the reference material used for that sample. If not, all samples prepared in association with the LCS must be redigested and reanalyzed.

9.1.3 Matrix Duplicate (DUP): A duplicate is analyzed for each batch of samples digested. If original sample and duplicate are both ≥ RL, then 20% RPD. If original sample and duplicate are less than the RL, the RPD is not calculated; otherwise, ±RL.

9.1.4 Matrix Spike (MS): A matrix spike is prepared and analyzed for each batch of samples. A portion of sample is spiked with 0.5 mL of DCAL-Int (Sec 7.2.3). This is equivalent to 1.0 ppb Hg (on instrument). A recovery of 80-120% is required.

9.1.5 Matrix Spike Duplicate (MSD): A matrix spike duplicate is prepared and analyzed when requested by the client. A portion of sample is spiked with 0.5 mL of DCAL-Int (Sec 7.2.3). This is equivalent to 1.0 ppb Hg (on instrument). A recovery of 80-120% is required. If matrix spike sample and matrix spike duplicate sample are both ≥ RL, then 20% RPD. If original sample and duplicate are less than the RL, the RPD is not calculated; otherwise, ±RL

9.1.6 Serial Dilution (SD): A five fold serial dilution must be performed on one sample per batch. The sample should contain a sufficiently high concentration; minimally a factor of 25 times the estimated detection limit. Dilute the sample by a minimum of five fold (1+4) and reanalyze. The results must agree within 10% of the original determination. If not, a chemical or physical effect should be suspected.

9.2. Instrument QC

9.2.1 Initial Calibration Verification (ICV): Initial calibration is verified after calibration using an independent check standard at a concentration near

the mid-point of the calibration (5.0ppb); see Sec 7.2.6 for preparation instructions. The results must be within 10% of the true value. If it's outside of the acceptable limits, terminate the analysis, correct the problem and recalibrate the instrument.

9.2.2 **Continuing Calibration Verification (CCV):** Calibration verification is performed after the calibration, after every 10 samples, and at the end of the run. Use a concentration of mercury at the midpoint of the calibration range (5.0 ppb). See Sec. 7.2.7 for preparation instructions. The value obtained must be within 10% of the true value. If not, stop the analysis and recalibrate. Re-analyze the previous ten samples following the last good calibration verification. .

9.2.3 **Initial and Continuing Calibration Blank (ICB/CCB):** ICB/ CCB must be analyzed after the calibration curve, every 10 samples, and at the end of the analytical run. The absolute value of the calibration verification blank must not exceed the reporting limit. If it does, terminate the analysis, correct the problem, recalibrate and reanalyze the samples following the last good CCB. The calibration verification blank is the same blank solution as used for the calibration blank.

10 Procedure

10.1. Sample Preparation (includes all samples, standards, and blanks)

10.1.1 Mix the sample well and weigh 0.5 - 0.6 grams of sample (including the LCSSRM) and place in the bottom of an appropriately identified 50 mL hotblock cup. For QA samples, weigh three portions of 0.5 - 0.6 grams of sample and place in the bottoms of three hotblock cups labeled as SAMPLE, DUP, and MS. Before adding any reagents, spike the MS sample with 0.5 mL of DCAL-Int standard.

10.1.2 Except the calibration standards, add 5 mL of deionized water to all hotblock cups (i.e., all field samples, MB, LCSSRM, Dup, and MS).

10.1.3 Add 1.5 ml concentrated HNO_3 and 4.5 ml concentrated HCl. Heat 2 min on the hotblock at 95 $\pm$3°C. Cool.

10.1.4 Add 15 mL deionized water and 15 mL potassium permanganate solution (Sec 7.1.11) to each hotblock cup. Mix well. The same amount of $KMnO_4$ must be added to the standards and samples.

10.1.5 Cap the hotblock cups loosely enough so that pressure does not build up but also tight enough so that the caps stay on and that volume loss due to heating is minimized. Heat 30 min on the hotblock at 95 $\pm$3°C. Cool.

10.1.6 To each hotblock cup, add 6 ml Sodium chloride - Hydroxylamine hydrochloride solution to reduce excess permanganate.

10.1.7 Using deionized water, bring to a 50 mL final volume. Cap and mix well.

10.2. Calibration

10.2.1 The instrument must be calibrated daily or once every 24 hours and each time the instrument is set up. The instrument is calibrated according to the manufacturer's specifications and must contain at least four standards and a blank. The laboratory currently uses five standards and a blank. The correlation coefficient of the calibration curve must be ≥0.995. If it does not, the problem must be corrected, and the instrument must be recalibrated. Standard preparations must be documented in the in TALS reagent module.

10.2.2 Prepare the calibration standards and Calibration Verification Standards as stated in Sections 7.2.5, 7.2.6., & 7.2.7.

10.2.3. Calibration Curve Read-Back:

10.2.3.1. Low-Level Readback (at the RL) – evaluate the 0.2 ug/L calibration standard. The %RE (relative error) must be +/- 20% of the true value (see Sec 11.4). If %RE is outside of the criteria limits, stop the analysis and recalibrate.

10.2.3.2. Mid- Level Readback – evaluate the 5.0 ug/L calibration standard (the mid-level calibration standard). The %RE (relative error) must be +/- 10% of the true value (see Sec 11.4). If %RE is outside of the criteria limits, stop the analysis and recalibrate

10.3. Sample Analysis

10.3.1 Following a sample digestion procedure, the samples are ready for instrumental analysis. It is advisable to investigate each matrix for any complexities, which might adversely affect the acquisition of valid data.

10.3.2 The following analytical run sequence is currently used for samples analyzed under Method 7471B:

Instrument Calibration (Blank and five standards)
ICV
ICB
CCV
CCB

10 Samples
CCV
CCB
10 Samples
CCV
CCB
Repeat until run is complete
CCV
CCB

10.3.3 Instrument Operation:

10.3.3.1 Turn on instrument, computer and monitor.

10.3.4 Plumbing the Reagent Lines:

10.3.4.1 One at a time, feed each of the pump tubes into a pump cassette, sliding the tube through the plastic clips at the bottom until the plastic tab is secure. Then, holding the tube taut, slide the loaded cassette onto the pump head and click the clamp, lever up. The tab end of the tube should be located at the front of the pump head.

10.3.4.2 Reductant (Red); Connect tab end of tube to the reductant bottle and the other end to the bottom of the mixing tee.

10.3.4.3 Sample (Blue); Connect tab end of tube to the autosampler probe and the other end to the top of the mixing tee.

10.3.4.4 Drain (Blue) Connect the tab end of tube to the sample discharge tube connected on the Liquid/Gas separator and the other end to the waste line.

10.3.4.5 Rinse (Black): Connect tab end of tube to rinse tubing that is connected to the rinse bottle. Connect the other end to the rinse tubing leading to the rinse cup.

10.3.5 Preparation of Reagents:

10.3.5.1 Pour the $SnCl_2$ solution into the reductant bottle and connect to the red reductant tube connector.

10.3.5.2 Pour the ten percent HCl solution into the Rinse reservoir bottle.

10.3.6 Starting Program:

10.3.6.1 Click the Envoy icon on the computer desktop

10.3.6.2 Click Method, select 7471_7471B, and select OK
10.3.6.3 On the main screen, click the StartUp icon. Wait 15 minutes before analyzing calibrating.
10.3.6.4 Click Sequence Tab on bottom of screen
10.3.6.5 Click Sequence on top of screen
10.3.6.6 Select Open, then select New
10.3.6.7 Using the Prep Batch Sheet and hand scanner, enter the sample barcodes into the Sample ID column. Include the Serial Dilution and all needed sample dilutions
10.3.6.8 Pour out the digested calibration standards and samples into the proper locations on the autosampler.
10.3.6.9 Click the Run Sequence icon to begin calibration and to run samples.

10.3.7 Creating the Raw Data PDF:

10.3.7.1 Click the Analysis Tab, select Detailed

10.3.7.2 Click Load, select Use This To Make PDF

10.3.7.3 Click Report, Click Printer

10.3.7.4 Include the calibration curve graph:

10.3.7.4.1 Go to Methods Tab and click Calibration, click Print

10.3.7.5 Combine both documents using the PDF Creator program. The new document is located in the Documents folder on the C:\ drive. Add this document to the "Doc's" location in the analytical batch.

10.3.8 Shutting Down the Instrument:

10.3.8.1 Click the Stop icon on the main screen

11.0. Calculations / Data Reduction

11.1. Accuracy:

$$\text{ICV / CCV, LCS \% Recovery} = \frac{\text{observed concentration}}{\text{known concentration}} \times 100$$

11.2. Precision (RPD):

Matrix Duplicate (MD) = $\frac{|\text{orig. sample value - dup. sample value}|}{[(\text{orig. sample value + dup. sample value})/2]} \times 100$

11.3. Concentration: (mg/Kg) = $\frac{C \times V1 \times D}{W}$

Where: C= Element concentration from instrument (ppb)
V1= Final volume of sample digested (in liters)
D= Dilution performed on sample
W= Initial weight of sample digested (in gram)

NOTE: All dry weight corrections are made in LIMS at the time the final report is prepared.

11.4. Relative Error (%RE):

% Relative Error = (100) (MC-TC)/TC

MC = Measured Concentration of the calibration standard
TC = True Concentration for the calibration standard

11.5. Data Processing:

11.5.1. All data is recorded directly in TALS' Analyst Desktop II program.

11.5.2. Import Data to TALS

11.5.2.1. Click the Analysis tab
11.5.2.2. Select the analytical run that needs to be imported
11.5.2.3. Select Statistics
11.5.2.4. Click Load and select TALS Import, click OK
11.5.2.5. Click Report, click CSV File
11.5.2.6. Name the import file (e.g., batch name, today's date)
11.5.2.7. The newly created import file is in the Import Folder on the desktop. Send to TALS Import Folder

11.5.3. Sample and standard preparations must be documented in the Analyst Desktop II program located in TestAmerica Laboratory System (TALS). The analyst must enter the following information: Source standard, Initial and final sample volume, spike name and amount used, all reagents and their corresponding lot numbers, creation and expiration dates.

11.5.4. All reagents must be recorded in TALS Reagent Module.

11.5.5. Complete the Data Review Checker (DRC) in TALS: Prior to data submission (i.e., prior to 1st leveling data in the Analyst Desktop II module in TALS), the analyst must execute the DRC (Data Review Checker) program in the applicable preparation batch.

11.5.5.1. Open the preparation batch and click on the Edit tab above to enter the Edit Mode.

11.5.5.2. Press F8 command key on the computer keyboard or right click anywhere on the batch worksheet then click on 'Run checklist.'

11.5.5.3. Acknowledge by filling in responses to all unacknowledged findings.

11.5.5.3.1. Highlight the checklist with findings, then highlight the associated list of findings; right click and choose 'Acknowledge Item.'

11.5.5.3.2. Fill in appropriate comments in the response box, then hit 'OK.'

11.5.5.3.3. Acknowledge all Finding items in the 'Manual Batch Checklist' except for the *"2nd Level review complete?"* this is to be completed by the 2nd level reviewer.

11.5.6. Record the following reagents and the volume used for sample preparation in the batch information page under "batch comments" in TALS Analyst Desktop II: concentrated sulfuric acid, concentrated nitric acid, potassium permanganate, potassium persulfate and sodium chloride-hydroxylamine hydrochloride. Record reagents in TALS by opening the prep batch, click on "edit" and then right click to choose "view batch information." Enter the information in the "batch comments" section.

12.0. Method Performance

12.1. Method Detection Limit Study (MDL)

The method detection limit (MDL) is the lowest concentration that can be detected for a given analytical method and sample matrix with 99% confidence that the analyte is present. The MDL is determined according to the laboratory's MDL procedure in Section 19 (Test Methods and Method Validation) of TestAmerica Edison's Quality Assurance Manual (ED-QA-LQM). MDLs reflect a calculated (statistical) value determined under ideal laboratory conditions in a clean matrix, and may not be achievable in all environmental matrices. The laboratory maintains MDL studies for analyses performed; these are verified at least annually unless method requirements require a greater frequency.

12.2 Instrument Detection Limit

The IDL for each analyte must be determined for each wavelength used on each instrument. The IDL must be determined annually or if the instrument is adjusted in any way that may affect the IDL. The IDL is determined by multiplying the average of the standard deviations obtained from the analysis of seven reagent blanks by 3.14.

12.3 Demonstration of Capabilities

For DOC procedure refer to Section 20 in the most current revision of TestAmerica Edison's Quality Assurance Manual (ED-QA-LQM).

12.4 Training Requirements

Refer to TestAmerica SOP No. ED-GEN-022, Training, for the laboratory's training program.

12.5. Lower Limit of Quantitation (LLOQ)

12.5.1. LLOQ verification: prior to analyzing samples under method 7470A, after any change that may affect the LLOQ, and quarterly, the LLOQ must be verified. The LLOQ is initially verified by the analysis of 7 replicate samples spiked at the LLOQ (0.2 ppb). The seven replicates must be digested prior to analysis. The LLOQ is verified when the mean recovery of the 7 replicates is +/-30% of the true value and the RSD < 20%.

12.5.2. LLOQ on-going verification: the LLOQ is re-verified quarterly by the analysis of a method blank (MB) and 1 blank sample (deionized water spiked at the LLOQ (0.2 ppb). The MB and LLOQ samples must be digested prior to analysis. The LLOQ is re-verified if the MB result is +/- 0.5 times the reporting limit for each analyte and when the recovery of the LLOQ sample is +/-30% of the .true value.

13.0. Pollution Control

It is TestAmerica's policy to evaluate each method and look for opportunities to minimize waste generated (i.e., examine recycling options, ordering chemicals based on quantity needed, preparation of reagents based on anticipated usage and reagent stability). Employees must abide by the policies in Section 13 of the Corporate Environmental Health and Safety Manual (CW-E-M-001) for "Waste Management and Pollution Prevention.

14.0. Waste Management

14.1. Waste management practices are conducted consistent with all applicable rules and regulations. Excess reagents, samples and method process wastes are disposed of in an accepted manner. Waste description rules and land disposal restrictions are followed. Waste disposal procedures are incorporated by reference to TestAmerica SOP No. ED-SPM-008 (Laboratory Waste Disposal Procedures).

14.2. The following waste streams are produced when this method is carried out:

- Digested Samples: Corrosive Acid- Materials that are not above regulatory limits will be submitted for elementary neutralization with 50% sodium hydroxide solution (Siedler Chemical SC-1824-03). Major concern is heat generated from the neutralization process. Initial volume of acid waste to be neutralized should be no more than 15 gallons. Finished neutralization with sodium bicarbonate (Siedler Chemical SC-0219-25) to a pH of 6 – 9 in the primary tank. Once pH has been established the primary tank is transferred through filter housing to a secondary tank. The pH is rechecked. If the pH is within specifications, the secondary tank is released to the municipal sewer system.

- Samples above regulatory limits and expired RCRA metals standards (Waste Corrosive Liquid, Acidic, Inorganic, n.o.s.) are collected in satellite accumulation and sent off site through a Waste disposal vendor.
  Onyx Profile WIP Number: 590598
  Teris Profile Number 50016653

15.0. References / Cross-References

15.1. Test Methods for Evaluating Solid Waste, Physical/Chemical Methods, 3rd ed.,U.S. Environmental Protection Agency, SW846 Manual, Method 7471B, Revision 2, January 1998.

15.2. Leeman Hydra IIAA Operating Manual.

15.3. TestAmerica Edison Document ED-QA-LQM, *Laboratory Quality Manual*, most current revision.

15.4. TestAmerica Edison SOP No. ED-SPM-008, *Laboratory Waste Disposal Procedures*, current revision.

15.5. Corporate Environmental Health and Safety Manual CW-E-M-001, most current revision.

15.6. TestAmerica Edison Subsampling SOP, *ED-GEN-007,* most current revision.

15.7. TestAmerica Edison SOP ED-GEN-022, *Training*, most current revision.

16.0. Method Modifications:

Item	Method #	Modification
1 (Sec 10.3.5.1)	7471B	Stannous Chloride is automatically added via the instrument versus the manual addition of Stannous Chloride as stated in the method. This is an instrument manufacturer's

		improvement that will reduce error due to loss of Mercury.
Sample preparation	7471B	The amount of DI water added to the sample at digestion is decreased to 15 ml from 50 ml (water bath procedure), while keeping the volume of reagents added during sample digestion the same. Sample final volume for hotblock procedure is modified to 50 ml (100 ml – water bath procedure) which allows for lesser sample volume hence less sample waste produced; hotblock digestion cup has maximum volume of 50 ml.

17.0. Attachments

Attachment 1: Certificate of Analysis of stock standards

18.0. Revision History

- Revision 5, dated 11 August 2022
  - Sec 12.5: Added the lower limit of quantitation (LLOQ), associated instructions and recovery limits.
- Revision 4, dated 03 August 2020
  - Updated SOP header with Eurofin logo.
  - Sec 6.1.1 & 15.2: Replaced Hydra AA with Hydra IIAA.
  - Sec 9.1 Table and 9.1.1: Revised MB control limits to <RL.
  - Sec 9.1, 9.1.4 and 9.1.5: Revised MS and MSD acceptance limits to 80-120%.
  - Sec 9.2.2: Revised CCV acceptance limits to +/-10%.
  - Sec 10.2.3: Added calibration curve read-back criteria.
  - Sec 11.4: Added Relative % error calculation.
  - Sec 11.5: Added Relative % error calculation.
  - Sec 11.5.6.: Added requirements to record reagent volumes in the batch information page of TALS ADII; subsequent sections adjusted accordingly.
- Revision 3, dated 12 October 2018
  - Sec 6.1.2: Removed WinHg instrument software reference and replaced with current instrument software, Envoy.
  - Sec 9.1.2: Revised to clarify the acceptance criteria used in the COA when evaluating recoveries for LCS in soil matrix
  - Sec 10.3.6: Updated the Starting Program instructions to reflect the new instrument software, Envoy.
  - Sec 10.3.7 & 10.3.8: Updated instructions for creating raw data pdf and shutting down instrument.
  - Sec 11.4.2: Updated instructions for importing data to TALS.
  - Sec 11.4.5: Replaced Metals Data Review Checklist with Data Review Checker (DRC) in TALS.

- Sec 15.8 & 15.9: Removed reference to EDS-WI-007 & EDS-WI-125; WI not applicable.

- Revision 2, dated 29 Nov 2016
  - Sec 6.1.4, Sec 10.1.3, & 10.1.5: updated the hotblock temperature range per method 7471B requirements
  - Sec 6.2.7: added Argon gas
  - Sec 7.2.4: the name of the intermediate ICV standard has been changed from DICV-Int to DQCS-Int
  - Sec 7.2.6: removed the CCV preparation instructions using the second source standard. The name of the intermediate ICV standard has been changed from DICV-Int to DQCS-Int
  - Sec 7.2.7: added preparation instructions for the CCV using the primary source standard
  - Sec 9.1: added the quality control sample MSD to the table
  - Sec 9.1.5: added quality control information for the MSD
  - Sec 11.4.5: Metals Data Review Checklist control # has been updated from ED-WI-007 to CA-Q-WI-042

- Revision 1, dated 12 May 2014
  - Sec 1.1: Revised the typical detection limit from 0.033 mg/kg to 0.017 mg/kg to reflect the 50mL final volume – hotblock procedure.
  - Sec 1 & 12: Updated LQM section references to reflect the most current LQM revision.
  - Sec 6.1.4: Added hotblock digestor to the list of equipment; removed autoclave from the list.
  - Sec 6.2: Added 50 mL hotblock digestion cups to replace the BOD bottles. Removed supplies which are no longer applicable (BOD bottles, graduated cylinders).
  - Sec 7.0 & 11.4.4: Removed Hg Reagent Logbook and replaced with TALS Reagent Module
  - Sec 7.1. Deleted Sulfuric Acid (previously referenced in Sec 7.1.1). Added 0.15% HNO_3 to list of reagents.
  - Sec 7.2.5: Updated Calibration standards' spiking instructions to reflect the 50 ml final volume.
  - Sec 7.2.6: Updated ICV/CCV spiking instructions to reflect the 50 ml final volume.
  - Sec 8.1: Added plastic container to the list of acceptable sample containers.
  - Sec 9.1 & 9.1.3: Clarified the acceptance limits for Matrix duplicate to reflect actual laboratory practices.
  - Sec 9.1.4: Revised the spiking amount added to the MS sample.
  - Replaced LCSS reference with LCSSRM in various sections of the SOP to reflect TALS QC type reference.
  - Sec 10.1: Revised sample preparation procedure to include hotblock digestor procedure; deleted all reference to autoclave digestion procedure.

- Sec 11.4.5 & 15.9: Added Work instructions EDS-WI-125 (TestAmerica Edison Metals Initial Calibration Data Review for Mercury).
- Sec 16: Added Sample prep method modification (i.e. final volume).

- Revision 0, dated 04 February 2011
  New

Attachment 1

Certificate of Analysis

Catalogue Number :	141-110-111/141-110-112/141-110-115
Description :	*Plasma*CAL - **ICP-MS Verification Standard 1 Solution B**
Lot Number :	**SC8298812**
Expiration Date :	**January 2010**

Analysis of Solution Standard by Inductively Coupled Plasma Spectroscopy (ICP-AES) traceable to NIST Standard Reference Materials : 3133

Actual Concentrations

Hg : 9.98 µg/ml

Matrix : **10% HNO_3**
Density : **1.040 g/ml @ 22.0°C**

MS 0882
Rec'd
1/14/09

Certified by : _____
Thomas Znoj, Chemistry Manager

Certification Date : **October 28, 2008**

This ICP-AES & ICP-MS Standard is guaranteed to be stable and accurate to within plus or minus 1.0% of the actual concentration up to the expiry date, provided the solution is kept tightly capped and stored under normal laboratory conditions. For these solutions, 18 megohm/cm double deionized water, high-purity acids, Class A glassware and acid-cleaned bottles are used. The Material Safety Data Sheet and this Certificate of Analysis are available on our web site. (Également disponible en Français)

Manufactured according to an ISO 9001:2000 Quality System and ISO 17025 (in-process)

SCP SCIENCE
21800 Clark Graham, Baie D'Urfé, QC, Canada H9X 4B6
Phone : (514) 457-0701 Fax : (514) 457-4499
Web Site: www.scpscience.com

INORGANIC VENTURES
195 Lehigh Avenue, Suite 4
Lakewood, New Jersey 08701 - USA
inorganicventures.com
tel: 800.669.6799 · 732.901.1900
fax: 732.901.1903
info@inorganicventures.com

CERTIFICATE OF ANALYSIS

MS0879
Rec'd 1-12-09

1.0 **INORGANIC VENTURES** is an ISO Guide 34 "General Requirements for the Competence of Reference Material Producers" and ISO 9001:2000 registered manufacturer. Our manufacturing laboratory is accredited to ISO/IEC 17025 "General Requirements for the Competence of Testing and Calibration Laboratories."

2.0 **DESCRIPTION OF CRM** **10 µg/mL Mercury in 10% (v/v) HCL**

Catalog Number:	MSHG-10PPM
Lot Number:	**B2-HG02061**
Starting Material:	Hg metal
Starting Material Purity (%):	99.999549
Starting Material Lot No:	05214TX
Matrix:	10% (v/v) HCL

3.0 **CERTIFIED VALUES AND UNCERTAINTIES**

Certified Concentration: 10.027 ± 0.020 µg/mL

Certified Density: 1.019 g/mL (measured at 22° C)

The following equations are used in the calculation of the certified value and the uncertainty. Reported uncertainties represent expanded uncertainties expressed at approximately the 95% confidence level using a coverage factor of k = 2.

$$\text{Certified Value } (\bar{x}) = \frac{\Sigma x_i}{n}$$

$$\text{Uncertainty } (\pm) = \frac{2[(\Sigma s_i)^2]^{1/2}}{(n)^{1/2}}$$

$(\bar{x})$ = mean
x_i = individual results
n = number of measurements
Σs_i = The summation of all significant estimated errors (Most common are the errors from instrumental measurement, weighing, dilution to volume, and the fixed error reported on the NIST SRM certificate of analysis.)

4.0 **TRACEABILITY TO NIST AND VALUES OBTAINED BY INDEPENDENT METHODS**

- "Property of the result of a measurement or the value of a standard whereby it can be related to stated references, usually national or international standards, through an unbroken chain of comparisons all having stated uncertainties." (ISO VIM, 2nd ed., 1993, definition 6.10)
- This product is Traceable to NIST via an unbroken chain of comparisons to the following NIST SRMs:

Title: Digestion of Water and Wastewater Samples for Analysis by ICP and ICP-MS, SW846 Method 3005A

Once printed, this is considered an uncontrolled document

Approvals (Signature/Date):

Laura Demone — 02/10/22
Laura Demone — Date
Department Manager

Dan Helfrich — 02/03/22
Dan Helfrich — Date
Health & Safety Manager/Coordinator

Carl Armbruster — 02/03/22
Carl Armbruster — Date
Quality Assurance Manager

Mark Acierno — 02/03/22
Mark Acierno — Date
Laboratory Director

Donald Evans — 02/02/22
Donald Evans — Date
Operations Manager

Facility Distribution No. Electronic **Distributed To:** Facility Intranet

1.0 Scope and Application

1.1. Analytes, Matrix(s), and Reporting Limits

Method SW846 3005A covers the preparation procedures for the determination of elemental constituents in water samples, wastewater samples by inductively coupled plasma (SW846 Method 6010D) and inductively coupled plasma – mass spectrometry (SW846 Method 6020B). Use Method 3010A for the acid digestion of leachate samples.

On occasion clients may request modifications to this SOP. These modifications are handled following the procedures outlined in Sections 7 (Review of Work Request) and 19 (Test Methods and Method Validation) of TestAmerica Edison's Quality Assurance Manual (ED-QA-LQM).

The elements analyzed for by this method and the laboratory's reporting limits (RLs) are summarized below:

Element	Method 6010D Water (ug/L)	Method 6020B Water (ug/L)
Aluminum	200	40
Barium	200	4
Beryllium	2	0.8
Boron	50	80
Cadmium	4	2
Calcium	5000	500
Chromium	10	4
Cobalt	50	4
Copper	25	4
Iron	150	120
Manganese	15	8
Magnesium	5000	200
Molybdenum	20	4
Nickel	40	4
Potassium	5000	200
Silver	10	2
Sodium	5000	500
Strontium	20	4
Tin	50	16
Titanium	20	4
Vanadium	50	4
Zinc	30	16
Antimony	20	2
Arsenic	15	2
Lead	10	1.2
Selenium	20	2.5
Thallium	20	0.8
Silicon	500	n/a
Sulfur	200	n/a

2.0 Summary of Method

A volume of sample is heated with nitric and hydrochloric acids. After the sample is reduced in volume, it is filtered if necessary, diluted to volume, and is then ready for analysis by inductively coupled plasma (ICP) by SW846 Method 6010D or inductively coupled plasma-mass spectrometry (ICP-MS) analysis by SW846 Method 6020B.

3.0 Definitions

For a complete list of definitions refer to Appendix 5 in the most current revision of TestAmerica Edison's Quality Assurance Manual (ED-QA-LQM).

4.0 Interferences

4.1 Whenever reflux is required or sample is to be reduced, NEVER BOIL THE SAMPLE. Boiling could result in a significant loss of sample constituents.

4.2 See the current analytical SOPs for further information related to interferences: TestAmerica SOP Nos. ED-MT-004 *Trace Metals Analysis by Inductively Coupled Plasma Emission Spectroscopy by SW846 Method 6010D* and ED-MT-034, *SW-846 Method 6020B, Trace Metals Analysis of Water, Wastewater, Soil, Sediment and Leachate Samples by ICP-MS*.

5.0 Safety

Employees must abide by the policies and procedures in the Corporate Environmental Health and Safety Manual (CW-E-M-001) and this document. This procedure may involve hazardous material, operations and equipment. This SOP does not purport to address all of the safety problems associated with its use. It is the responsibility of the user of the method to follow appropriate safety, waste disposal and health practices under the assumption that all samples and reagents are potentially hazardous. Safety glasses, gloves, lab coats and closed-toe, nonabsorbent shoes are a minimum.

5.1. Specific Safety Concerns or Requirements

Samples that contain high concentrations of carbonates or organic material or samples that are at elevated pH can react violently when acids are added.

5.2. Primary Materials Used

The following is a list of the materials used in this method, which have a serious or significant hazard rating. **Note: This list does not include all materials used in the method. The table contains a summary of the primary hazards listed in the MSDS for each of the materials listed in the table.** A complete list of materials used in the method can be found in the reagents and materials section. Employees must review the information in the MSDS for each material before using it for the first time or when there are major changes to the MSDS.

Material [1]	Hazards	Exposure Limit [2]	Signs and symptoms of exposure
Hydrochloric Acid	Corrosive Poison	5 ppm-Ceiling	Inhalation of vapors can cause coughing, choking, inflammation of the nose, throat, and upper respiratory tract, and in severe cases, pulmonary edema, circulatory failure, and death. Can cause redness, pain, and severe skin burns. Vapors are irritating and may cause damage to the eyes. Contact may cause severe burns and permanent eye damage.
Nitric Acid	Corrosive Oxidizer Poison	2 ppm-TWA 4 ppm-STEL	Nitric acid is extremely hazardous; it is corrosive, reactive, an oxidizer, and a poison. Inhalation of vapors can cause breathing difficulties and lead to pneumonia and pulmonary edema, which may be fatal. Other symptoms may include coughing, choking, and irritation of the nose, throat, and respiratory tract. Can cause redness, pain, and severe skin burns. Concentrated solutions cause deep ulcers and stain skin a yellow or yellow-brown color. Vapors are irritating and may cause damage to the eyes. Contact may cause severe burns and permanent eye damage.
1 – Always add acid to water to prevent violent reactions.			
2 – Exposure limit refers to the OSHA regulatory exposure limit.			

6.0 Equipment and Supplies

6.1. Instrumentation

- Block Digestor: Adjustable and capable of maintaining a temperature of 90ºC-95ºC (Environmental Express or equivalent).
- AutoBlock: Environmental Express

6.2. Supplies

- watch glasses
- 75 mm funnels
- 50 ml and 100 ml Hot Block Digestion Cups
- Whatman # 41 filter paper or equivalent
- 0.45 micron filter units and vacuum pump
- Pipettes varying volumes: Eppendorfs & Fisher
- Reflux Caps, p/n SC506 Environmental Express or equivalent

7.0 Reagents and Standards

7.1. Reagents

7.1.1. Nitric Acid, Concentrated - Trace Grade or equivalent; store at room temperature; for stability information, refer to manufacturer's instructions.

7.1.2. Hydrochloric Acid, Concentrated - Trace Grade or equivalent; store at room temperature; for stability information, refer to manufacturer's instructions.

7.1.3. Reagent Grade Water 18 Megohm Minimum

7.2. Standards

7.2.1 Stock ICP Spike Standards:

7.2.1.1 Solutions A, B, C, and D (ICP-Spk), Part No. 4400-100419DD01: commercially purchased from CPI International. Store at room temperature; for stability information, refer to manufacturer's instructions. See Attachment 1 for the standards certified concentrations and catalog numbers. 1000ppm each of Silicon and Sulfur purchased from CPI International or equivalent.

7.2.2 Stock ICP-MS Spike Standards:

7.2.2.1 ICPMS LCS/SPK, Part No. SM-606-111: commercially purchased from High-Purity Standards. Store at room temperature; for stability information, refer to manufacturer's instructions. This stock standard doesn't require any pre-dilutions and is ready to be spiked directly into the QC samples. See Attachment 1 for the standards certified concentrations and catalog numbers.

7.3 Working Spike Standards:

7.3.1. ME_LCS-int (for ICP prep) - Add the following to a 1000 ml volumetric flask and bring to volume using 5% HNO_3: 50 ml each of Part No. 4400-100419DD01 Solutions A, B, C, and D (Sec 7.2.1.1). Record standard preparations in TALS Reagent module. **Note**: Standard must not exceed the earliest expiration date of any one of its components.

8.0 Sample Collection, Preservation, Shipment and Storage

Sample container, preservation techniques and holding times may vary and are dependent on sample matrix, method of choice, regulatory compliance, and/or specific contract or client requests. Listed below are the holding times and the references that include preservation requirements.

Matrix	Sample[4] Container	Min. Sample Size	Preservation	Holding Time [1]	Reference
Water	Polyethylene, glass	50 ml	HNO_3 to pH < 2 prior to shipment; if not, acidify upon receipt in lab [2,3]	180 Days	40 CFR Part 136.3

[1] Inclusive of digestion and analysis.
[2] Acid preservation may be omitted for shipping; however, acid must be added upon receipt in the lab. Following acidification, mix the sample and hold for at least 24 hours. Just prior to digestion or direct analysis, verify pH<2. If pH≥ 2, repeat steps (i.e., add acid, hold for 24hrs, verify ph<2).
[3] Aqueous samples may be stored at room temperature.
[4] All containers must be pre-washed with detergents, acids and water.

9.0 Quality Control

9.1. Sample QC - The following quality control samples are prepared with every 20 samples or every batch of samples digested whichever comes first.

Quality Controls	Frequency	Control Limit
Method Blank (MB)	1 in 20 or fewer samples	See the Quality Control Section of the referring analytical SOP (i.e., SOP No. ED-MT-004 and ED-MT-034)
Laboratory Control Sample (LCS)	1 in 20 or fewer samples	
Matrix Spike (MS)[1]	1 in 20 or fewer samples	
Sample Duplicate (DUP)[1]	1 in 20 or fewer samples	
Matrix Spike Duplicate (MSD)	When requested by the client	

[1] The sample selection for MS and Sample Duplicate is random unless specifically requested by a client. Quality control samples prepared with each set of samples digested include Matrix Spike, Sample Duplicate, Method Blank, Laboratory Control Sample and Matrix Spike Duplicate (MSD: when requested by the client). Use the same environmental sample for the matrix spike and duplicate sample whenever possible. If insufficient sample volume is available, another environmental sample may be used for the duplicate sample.

9.2 Instrument QC

None

10.0 Procedure

10.1. Sample Preparation

10.1.1. Filtration Procedure for Dissolved Metals not filtered in the Field

10.1.1.1. The unpreserved sample must be filtered through a 0.45um filter unit as soon as practical after collection.

10.1.1.2. Collect the required volume of filtrate by using a 0.45um filter unit and a vacuum pump.

10.1.1.3. Acidify the filtrate with 1:1 HNO_3 to a pH of <2.

10.1.1.4. The method blank (MB) must be filtered and digested under the same conditions as the lab filtered samples.

10.1.2. During digestion, at least 2-3 ml of sample solution must be maintained in the digestion cup. Sample must never be allowed to go to dryness. If sample should go to dryness, discard sample and redigest.

10.1.3. Pour out 50 ml of well-mixed sample into a 50 ml digestion cups *[note:100 mL hot block digestion cups can be used, except use the same sample volume (50 mL) for the entire preparation batch].* Label the cup with the sample number using a permanent marker. Prepare the QC samples as follows.

10.1.3.1. Method Blank (MB): Pour out 50 ml of deionized reagent water into the 50 ml digestion cup. Label the cup MB mm/dd/yy (this being the date the sample was prepared) and batch number. The Method Blank is carried through the complete digestion process.

10.1.3.2. Matrix Duplicate (DU): Label the digestion cup assigned for the duplicate sample with sample number and suffix 'DU'. Pour 50 ml of the well-mixed sample.

10.1.3.3. Matrix Spike Sample (MS): Measure 50 ml of well-mixed sample into the appropriately labeled 50 ml digestion cups (cup is labeled with the sample number and suffix 'MS'). The resulting elemental concentrations are listed in Attachments 2 & 3.

- For ICP preps: spike the ICP Matrix Spike sample with 1 ml of ME_LCS-int (Sec 7.3.1). If Silicon and Sulfur are needed, spike 0.2 mL each of 1000ppm Sulfur and Silicon.

- For ICP-MS preps: spike the sample with 0.25 ml ICPMS LCS/SPK (Sec 7.2.2.1).

10.1.3.4. Laboratory Control Sample (LCS): Pour out 50 ml of deionized reagent water sample into the appropriately labeled 50 ml digestion cups. Label the cup 'LCS' and the batch number. Spike the ICP-LCS and ICPMS- LCS (as appropriate) in the same way as the Matrix Spike Sample, see Sec. 10.1.3.3. Refer to Attachments 2 & 3 for final elemental concentrations.

10.1.4. Using Hot Block (if using AutoBlock, skip to section 10.1.5)

10.1.4.1. Add 1 ml concentrated HNO_3 and 2.5 ml of hydrochloric acid, cover with ribbed watch glass, place digestion cup on hot block at 90-95 ^oC, and reduce volume to approximately 15-20 ml. Do not allow sample to boil or dry.

10.1.4.2. Allow sample to cool down.

10.1.4.3. Wash down watchglass and digestion cup walls with deionized water. If necessary, accurately label a new 50 ml sample cup and filter samples through a #41 Whatman filter paper to remove any silicates and other insoluble material that could clog the nebulizer on the instrument. Bring the sample volume up to 50 ml.

10.1.4.4. Enter all batch/sample information into TALS (see Sec 11.4. for instructions). Print the sample labels from the batch and attach to the corresponding sample cups. These labels contain information such as: Job & sample number, Batch number and container number.

10.1.5. Using AutoBlock

10.1.5.1. Place a Reflux Cap (sec 6.2) on every 50 ml hotblock cup.

10.1.5.2. Turn on AutoBlock then click the *AutoBlock Plus* icon on the computer desktop.

10.1.5.3. Check the waste drum. If needed, properly dump the waste.

10.1.5.4. Check the reagents (i.e., HCL, HNO3, and Deionized Water). Refill if necessary.

10.1.5.5. Click *Operator Test* and click *Select Method.*

10.1.5.6. Select *3005A-Waters.mtd*

10.1.5.7. Under *Cell Position Selection*, click the hotblock locations where you will have the samples.

10.1.5.8. After closing the see-through plastic door, click on *Start Method.*

10.1.5.9. Don't open the door until the method has been completed.

10.1.5.10. When finished, *Method Completed* will be displayed in the software.

10.1.5.11. Wash down the watchglass and digestion cup walls with deionized water. If necessary, accurately label a new 50 ml sample cup and filter samples through a #41 Whatman filter

paper to remove any silicates and other insoluble material that could clog the nebulizer on the instrument. Bring the sample volume up to 50 ml.

10.1.5.12. Enter all batch/sample information into TALS (see Sec 11.4. for instructions). Print the sample labels from the batch and attach to the corresponding sample cups. These labels contain information such as: Job & sample number, Batch number and container number.

10.2. Calibration

10.2.1. The volume of the 50ml and 100 ml hotblock digestion cups are verified for each lot received. The 50 ml and 100 ml volume verification with the appropriate Lot number is documented in the 'Metals Labware Volume Verification Logbook.'

10.2.2. Per manufacturer's instructions, every 30 days, recalibrate the AutoBlock:

10.2.2.1. Place an empty hotblock cup on an analytical balance and 'zero' the balance.
10.2.2.2. Place that empty hotblock cup in the first position, I1, in the AutoBlock.
10.2.2.3. Click the *AutoBlock Plus* icon on the computer desktop
10.2.2.4. Click the *Manuel Screen* tab.
10.2.2.5. Under *Reagent Selection*, click *H2O(6).* This is Port 6 which contains Deionized Water.
10.2.2.6. Under *Cell Position Selection,* click *I1.*
10.2.2.7. Under *System Control*, click *Move to Position.*
10.2.2.8. In the *Injection Amount (ml)* box, type "30" and press *Inject.* The Autoblock will dispense 30 mL of DI Water into the cup.
10.2.2.9. Click *Move to Drain* and press *Exit.*
10.2.2.10. Weigh the cup containing the DI Water on the zeroed balance to determine the weight of the water.
10.2.2.11. Press the *Service Screen* tab.
10.2.2.12. Press the *Pump Cal* tab
10.2.2.13. Under *Pump 2 Actual Injection Amount (ml)*, enter the weight of the water and press *Pump 2 Save Cal.*

10.3. Sample Analysis

10.3.1. Refer to the Analytical SOP, TestAmerica Edison SOP No. ED-MT-004, and SOP No. ED-MT-034.

11.0. Calculations / Data Reduction

11.1. Accuracy:

$$\text{ICV / CCV, LCS \% Recovery} = \frac{\text{observed concentration}}{\text{known concentration}} \times 100$$

$$\text{MS \% Recovery} = \frac{\text{(spiked sample) - (unspiked sample)}}{\text{spiked concentration}} \times 100$$

11.2. Precision (RPD):

$$\text{Matrix Duplicate (MD)} = \frac{|\text{orig. sample value - dup. sample value}|}{[(\text{orig. sample value + dup. sample value})/2]} \times 100$$

11.3. Concentration:

Refer to TestAmerica SOP No. ED-MT-004 and ED-MT-034.

11.4. Documenting and Reporting

11.4.1. Sample preparations must be documented in the Analyst Desktop II program located in TestAmerica Laboratory System (TALS).

11.4.1.1. Double click the TALS icon on the computer desktop. Enter your username and password and select Login.

11.4.1.2. Under TALS Menu, click the 'plus' sign next to Analyst. Double click Analyst Desktop II. Click the 'plus' sign next to Methods.

11.4.1.3. Right click the prep method that you are using to prep the samples. Select Create New Batch-From Scratch.

11.4.1.4. The analyst must enter the following information: Sample names (use scanners), initial and final sample volume, spike name and amount used, and all reagents and their corresponding lot numbers.

11.4.1.5. After saving the batch information in TALS, select Print-Batch Sheets (which must be submitted to metals analytical dept. along with the samples) and select Print-Batch Labels (which must be attached to the corresponding final digestate sample cups in the batch).

11.4.2. Complete the Data Review Checker (DRC) in TALS: Prior to data submission (i.e., prior to 1st leveling data in the Analyst Desktop II module in TALS), the analyst must execute the DRC (Data Review Checker) program in the applicable preparation batch.

11.4.2.1. Open the preparation batch and click on the Edit tab above to enter the Edit Mode.

11.4.2.2. Press F8 command key on the computer keyboard or right click anywhere on the batch worksheet then click on 'Run checklist.'

11.4.2.3. Acknowledge by filling in responses to all unacknowledged findings.

11.4.2.3.1. Highlight the checklist with findings, then highlight the associated list of findings; right click and choose 'Acknowledge Item.'

11.4.2.3.2. Fill in the appropriate comments in the response box, then hit 'OK.'

11.4.2.3.3. Acknowledge all finding items in the 'Manual Batch Checklist' except for the *"2nd Level review complete?"* this is to be completed by the 2nd level reviewer.

12.0. Method Performance

12.1. Method Detection Limit Study (MDL)

The method detection limit (MDL) is the lowest concentration that can be detected for a given analytical method and sample matrix with 99% confidence that the analyte is present. The MDL is determined according to the laboratory's MDL procedure in Section 19 (Test Methods and Method Validation) of TestAmerica Edison's Quality Assurance Manual (ED-QA-LQM). MDLs reflect a calculated (statistical) value determined under ideal laboratory conditions in a clean matrix, and may not be achievable in all environmental matrices. The laboratory maintains MDL studies for analyses performed; these are verified at least annually unless method requirements require a greater frequency.

12.2. Demonstration of Capabilities

For DOC procedure refer to Section 19 in the most current revision of TestAmerica Edison's Quality Assurance Manual (ED-QA-LQM).

12.3. Training Requirements

Refer to TestAmerica SOP No. ED-GEN-022, Training, for the laboratory's training program.

13.0. Pollution Control

It is TestAmerica's policy to evaluate each method and look for opportunities to minimize waste generated (i.e., examine recycling options, ordering chemicals based on quantity needed, preparation of reagents based on anticipated usage and reagent stability). Employees must abide by the policies in Section 13 of the Corporate Environmental Health and Safety Manual (CW-E-M-001) for "Waste Management and Pollution Prevention."

14.0. Waste Management

14.1. Waste management practices are conducted consistent with all applicable rules and regulations. Excess reagents, samples and method process wastes are disposed of in an accepted manner. Waste description rules and land disposal restrictions are followed. Waste disposal procedures are incorporated by reference to TestAmerica SOP No. ED-SPM-008 (Laboratory Waste Disposal Procedures).

14.2. The following waste streams are produced when this method is carried out:

- Digested Samples: Corrosive Acid- Materials that are not above regulatory limits will be submitted for elementary neutralization. The neutralization system consists of a 55-gallon poly open top container. There is also a local snorkel exhaust for venting any noxious odors. Once the liquid is transferred into the container, the contents are sparged with air and a representative pH is measured and the amount of material is recorded in the discharge log. The pH value is recorded in the discharge log, and the liquid is neutralized to a pH of 6-9 SU using food grade sodium bicarbonate. Once the secondary pH has been taken, confirmed and recorded, the discharge valve is opened to the municipal sewer system and the neutralization process is complete. The date, type of waste stream, and volume discharged are recorded in the discharge log.

- Samples above regulatory limits and expired RCRA metals standards (Waste Corrosive Liquid, Acidic, Inorganic, n.o.s.) are collected in satellite accumulation and sent off site through a Waste disposal vendor.
  TSDF under Veolia Profile Number: 590598

- Soil Retain Samples - These containers are stored in the main sample receiving laboratory cooler until they are designated for disposal. Once designated for disposal, the samples are transferred to the waste disposal area and placed into cubic-yard disposal containers, e.g., Jupiter-boxes. This waste is shipped directly to the TSDF under Veolia Profile Number 402535 and is subject to incineration.

15.0. References / Cross-References

15.1. Test Methods for Evaluating Solid Waste, Physical/Chemical Methods, 3rd ed.,U.S. Environmental Protection Agency, SW846 Manual, Method 3005A, Revision 1, July 1992.

15.2. TestAmerica Edison Document No. ED-QA-LQM, *Laboratory Quality Manual*, most current revision.

15.3. TestAmerica Edison SOP ED-GEN-022, *Training*, most current revision

15.4. TestAmerica Edison SOP No. ED-MT-004, *Trace Metals Analysis by Inductively Coupled Plasma Emission Spectroscopy by SW846 Method 6010D*, most current revision.

15.5. TestAmerica Edison SOP No. ED-MT-034, *SW-846 Method 6020B, Trace Metals Analysis of Water, Wastewater, Soil, Sediment and Leachate Samples by ICP-MS,*

most current revision.

15.6. TestAmerica Edison SOP No. ED-SPM-008, *Laboratory Waste Disposal Procedures*, current revision.

15.7. TestAmerica Corporate Environmental Health and Safety Manual, Document No. CW-E-M-001, most current revision.

16.0. Method Modifications:

Sample initial and final volumes are modified to 50 ml which allows for lesser sample volume needed for digestion and less sample waste produced.

17.0. Attachments

Attachment 1: Certificate of Analysis of ICP and ICPMS stock standard.
Attachment 2: ICP: Laboratory Control Sample and Matrix Spike Final Concentration in Soln (ug/L)
Attachment 3: ICP-MS Matrix Spiking Solution, ICPMS LCS/SPK

18.0. Revision History

- Revision 8, dated 10 February 2022
  - SOP reinstated, updated all applicable sections to reflect the most current procedures for QC, Reagents, Standards, Calibration, Reporting and SOP references.

- Revision 7, dated 10 November 2011
  - Sec 7.2.1.1: Updated the stock ICP spike standards to reflect actual lab practices.
  - Sec 7.2.2.1: Updated the ICPMS LCS/SPK which is now purchased from High-Purity Standards.
  - Sec 7.3.1: Updated preparation procedure for ME-LCS-int to reflect actual lab practices.
  - Sec 8: Added footnote #4: 24 hr waiting period after lab acid preservations.
  - Sec 10.1.1.4: Added procedures for filtering Method blanks for dissolved metals.
  - Sec 10.1.8: Added instructions for adding TALS' generated labels to digestion cups
  - Sec 11.4: Expanded procedures for documenting and reporting information via TALS.
  - Attachment 2: Updated the ICPMS final concentration for Boron and Zinc in *LCS & Matrix Spike, Final Concentrations in Solutions* Table.
  - Attachment 3: Updated COA's of stock standards.
  - Sec 15: Added applicable references.

- Revision 6, dated 20 October 2009

  - Revised SOP format in accordance with TestAmerica Corporate Quality SOP No. CW-QS-002 (Rev 0) Writing a Standard Operating Procedure (SOP).
  - Sec1.1: Deleted analysis by FLAA spectroscopy and added analysis by ICP-MS.
  - Sec 1.2: Added reporting limit for all elements analyzed by this method.
  - Sec 6: Replaced hot plate with block digestor. Deleted glasswares, (graduated

cylinders, beakers, volumetric flasks), supplies not applicable.

- Sec. 7.2 & 7.3: Revised the source of the stock spike standards and the preparation instructions for the ICP working spike standards.
- Sec 10: Replaced beakers with hot block digestion cups
- Sec 15: Applicable references added.
- Sec 17: Attachments added.

Attachment 1

USA
5580 Skylane Boulevard P: 707.525.5788
Santa Rosa, CA 95403 P: 800.878.7654
F: 707.545.7901

CPI INTERNATIONAL
Advanced Analytical, Semiconductor and Life Science Solutions
www.cpiinternational.com
www.calibag.com

Europe
P.O. Box 2704 P: +31 20 638 05 97
1000 CS Amsterdam F: +31 20 420 28 36
The Netherlands

SOLN. A
DE
4-4-11

Expiry: 21-Sep-12

Certificate of Analysis

Date Rec'd
03/22/11 MP

Part Number: **4400-100419DD01**
Lot Number: **11C157**
Shelf Life: **18 Months**

TestAmerica/Edison
Custom Standard
5% HNO3

Concentrations in ug/mL ± 0.5%

Fe	1000	Zn	500
Se	2000	Mn	500
TL	2000	NI	500
Be	50		
Cd	50		
Cr	200		
Co	500		
Cu	250		
Pb	500		
Ag	50		
Sr	500		
V	500		

This standard solution was prepared using high-purity starting materials, high-purity acid (if required) and 18-megaohm de-ionized water. The starting materials were weighed to five significant figures and diluted in volumetric glassware calibrated to five significant figures.

Starting materials were analyzed at 1000µg/mL by ICP-MS for trace impurities. The standard solution concentrations were certified instrumentally against the National Institute of Standards and Technology's SRM 3100 series, NIST approved second source and/or gravimetrically.

Accuracy and stability are guaranteed to within plus or minus 0.5% of the certified value for the stated shelf life from the date of shipment. The solution should be kept tightly capped and stored under normal laboratory conditions. See attached MSDS for proper handling information.

For questions or comments please call 1-800-878-7654 in the USA, +31 20 638 05 97 in Europe or visit our web-site at www.cpiinternational.com.

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www.ceilag.com

Europe
P.O. Box 2704 P: +31 20 638 05 97
1000 CS Amsterdam F: +31 20 420 28 36
The Netherlands

Expiry: 21-Sep-12

Certificate of Analysis

Part Number: **4400-100419DD01** **Solution B**
Lot Number: **11C157**
Shelf Life: **18 Months**

Date Rec'd
03/22/11 MP

TestAmerica/Edison
Custom Standard
10% HNO3

Concentrations in ug/mL ± 0.5%

Al	2000
Mg	20000
K	20000
Ca	20000
Na	20000

This standard solution was prepared using high-purity starting materials, high-purity acid (if required) and 18-megaohm de-ionized water. The starting materials were weighed to five significant figures and diluted in volumetric glassware calibrated to five significant figures.

Starting materials were analyzed at 1000µg/mL by ICP-MS for trace impurities. The standard solution concentrations were certified instrumentally against the National Institute of Standards and Technology's SRM 3100 series, NIST approved second source and/or gravimetrically.

Accuracy and stability are guaranteed to within plus or minus 0.5% of the certified value for the stated shelf life from the date of shipment. The solution should be kept tightly capped and stored under normal laboratory conditions. See attached MSDS for proper handling information.

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USA
5580 Skylane Boulevard P: 707.525.5788
Santa Rosa, CA 95403 P: 800.878.7654
F: 707.545.7901

CPI INTERNATIONAL
Advanced Analytical, Semiconductor and Life Science Solutions

Europe
P.O. Box 2704 P: +31 20 638 05 97
1000 CS Amsterdam F: +31 20 420 28 36
The Netherlands

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Expiry: 21-Sep-12

Certificate of Analysis

Part Number: **4400-100419DD01** **Solution D**
Lot Number: **11C157**
Shelf Life: **18 Months**

Date Rec'd
05/22/11 MH

TestAmerica/Edison
Custom Standard
2% HNO3

Concentrations in ug/mL ± 0.5%

As	2000
Ba	2000
B	500

This standard solution was prepared using high-purity starting materials, high-purity acid (if required) and 18-megaohm de-ionized water. The starting materials were weighed to five significant figures and diluted in volumetric glassware calibrated to five significant figures.

Starting materials were analyzed at 1000µg/mL by ICP-MS for trace impurities. The standard solution concentrations were certified instrumentally against the National Institute of Standards and Technology's SRM 3100 series, NIST approved second source and/or gravimetrically.

Accuracy and stability are guaranteed to within plus or minus 0.5% of the certified value for the stated shelf life from the date of shipment. The solution should be kept tightly capped and stored under normal laboratory conditions. See attached MSDS for proper handling information.

For questions or comments please call 1-800-878-7654 in the USA, +31 20 638 05 97 in Europe or visit our web-site at www.cpiinternational.com

USA
5580 Skylane Boulevard
Santa Rosa, CA 95403
P: 707.525.5788
P: 800.878.7654
F: 707.545.7901

CPI INTERNATIONAL
Advanced Analytical, Semiconductor and Life Science Solutions

Europe
P.O. Box 2704
1000 CS Amsterdam
The Netherlands
P: +31 20 638 05 97
F: +31 20 420 28 36

www.cpiinternational.com
www.calitag.com

Expiry: 21-Sep-12

Certificate of Analysis

Part Number: **4400-100419DD01** **Solution C**
Lot Number: **11C157**
Shelf Life: **18 Months**

Date Rec'd
03/22/11 MP

TestAmerica/Edison
Custom Standard
5% HNO3 + 2% HF

Concentrations in ug/mL ± 0.5%

Sb	500
Mo	500
Sn	500
Ti	500

This standard solution was prepared using high-purity starting materials, high-purity acid (if required) and 18-megachm de-ionized water. The starting materials were weighed to five significant figures and diluted in volumetric glassware calibrated to five significant figures.

Starting materials were analyzed at 1000µg/mL by ICP-MS for trace impurities. The standard solution concentrations were certified instrumentally against the National Institute of Standards and Technology's SRM 3100 series, NIST approved second source and/or gravimetrically.

Accuracy and stability are guaranteed to within plus or minus 0.5% of the certified value for the stated shelf life from the date of shipment. The solution should be kept tightly capped and stored under normal laboratory conditions. See attached MSDS for proper handling information.

For questions or comments please call 1-800-878-7654 in the USA, +31 20 638 05 97 in Europe or visit our web-site at www.cpiinternational.com.

HIGH-PURITY STANDARDS
www.highpuritystandards.com
P.O. Box 41727
Charleston, SC 29423
Phone: (843) 767-7900
Fax: (843) 767-7906

Certificate of Analysis

SM-606-111
(ICPMS LCS/SPK)
Lot # 1031503

Source	Source Purity	Matrix	Standard Concentration
High Purity Metals, Salts and Oxides	99.98+%	HNO_3, 4% + Tr HF	µg/mL ± 0.5% See element list on reverse

This spectrometric standard solution has been prepared from high-purity reference materials. Sub-boiling distilled high-purity acid has been used to place the materials in solution and to stabilize the standard. The matrix is as noted above in 18 megaohm deionized water. The reference materials have been assayed by inductively coupled plasma optical emission spectrometry (ICP-OES).

The standard has been prepared gravimetrically by weighing the reference material to 5 significant figures. Volumetric glassware has been calibrated gravimetrically to 5 significant figures. The standard concentration has been verified by ICP-OES against an independent source which is directly traceable to National Institute of Standards and Technology, Standard Reference Material No. 3100 series.

This standard is valid for one year from the shipping date provided the solution is kept tightly capped and stored under normal laboratory conditions.

Theodore C. Rains

Theodore C. Rains, Ph.D.
President

Exp Date: NOV 15 2011
MSDS ATTACHED

SM-606-111
(ICPMS LCS/SPK)
Element List
(µg/mL)

Aluminum	500
Antimony	5
Arsenic	10
Barium	10
Beryllium	5
Boron	100
Cadmium	5
Calcium	500
Chromium	10
Cobalt	5
Copper	10
Iron	500
Lead	5
Magnesium	500
Manganese	50
Molybdenum	10
Nickel	10
Potassium	500
Selenium	10
Silver	5
Sodium	500
Strontium	10
Thallium	4
Tin	10
Titanium	10
Vanadium	10
Zinc	50

Attachment 2

ICP: LCS & Matrix Spike Final concentration in solution	
ELEMENT	ug/L
Aluminum	2000
Antimony	500
Arsenic	2000
Barium	2000
Beryllium	50
Cadmium	50
Calcium	20000
Chromium	200
Cobalt	500
Copper	250
Iron	1000
Lead	500
Manganese	500
Magnesium	20000
Nickel	500
Potassium	20000
Selenium	2000
Silver	50
Sodium	20000
Thallium	2000
Vanadium	500
Zinc	500
Boron	500
Molybdenum	500
Tin	500
Titanium	500
Strontium	500
Silicon	4000
Sulfur	4000

Attachment 3

ICPMS Matrix Spiking Solution, ICPMS LCS/SPK	
Element	**Matrix Spike Conc. (ug/L)**
Aluminum (Al)	2500
Antimony (Sb)	25
Arsenic (As)	50
Barium (Ba)	50
Beryllium (Be)	25
Boron (B)	500
Cadmium (Cd)	25
Calcium (Ca)	2500
Chromium (Cr)	50
Cobalt (Co)	25
Copper (Cu)	50
Iron (Fe)	2500
Lead (Pb)	25
Magnesium(Mg)	2500
Manganese (Mn)	250
Molybdenum (Mo)	50
Nickel (Ni)	50
Potassium (K)	2500
Selenium (Se)	50
Silver (Ag)	25
Sodium (Na)	2500
Strontium (Sr)	50
Thallium (Tl)	20
Tin (Sn)	50
Titanium (Ti)	50
Vanadium (V)	50
Zinc (Zn)	250

Title: Hot Block Digestion of Sediments, Sludges, and Soils by USEPA Method No(s). SW846 3050B

Once printed, this is considered an uncontrolled document

Approvals (Signature/Date):

Laura Demone — 09/29/21
Laura Demone — Date
Department Manager

Dan Helfrich — 09/29/21
Dan Helfrich — Date
Health & Safety Manager / Coordinator

09/29/21
Carl Armbruster — Date
Quality Assurance Manager

0929/21
Mark Acierno — Date
Laboratory Director

Donald Evans — 09/29/21

1.0 Scope and Application

1.1. Analytes, Matrix(s), and Reporting Limits

This SOP covers the acid digestion procedure (SW846 3050B) for the determination of total recoverable elemental constituents in sediments, soils and sludges by inductively coupled plasma emission spectroscopy (EPA Method 6010D) and Inductively Coupled Plasma – Mass Spectrometry (EPA Method 6020B).

1.2. For a complete list of analytes and Reporting limits, refer to the appropriate analytical TestAmerica SOPs: SOP ED-MT-004, *Trace Metals Analysis by Inductively Coupled Plasma Emission Spectroscopy, Method No. SW846 Method 6010D;* SOP ED-MT-029; *Trace Metals Analysis for Water, Wastewater, Soil,Sediment and Leachate Samples by ICP-MS Method SW-846 6020B and* SOP ED-MT-034, *SW846 Method 6020B, Trace Metals Analysis of Water, Wastewater, Soil, Sediment, and Leachate Samples by ICPMS*

1.3. On occasion clients may request modifications to this SOP. These modifications are handled following the procedures outlined in Sections 7 (Review of Work Request) and 19 (Test Methods and Method Validation) of TestAmerica Edison's Quality Assurance Manual (ED-QA-LQM).

2.0 Summary of Method

A portion of sample is digested with repeated additions of nitric acid and hydrogen peroxide. Hydrochloric acid is added to the digestate and the sample is refluxed and then brought to a final volume of 100 ml. The resulting digestate is analyzed by ICP-AES or ICP-MS.

3.0 Definitions

For a complete list of definitions refer to Appendix 2 in the most current revision of TestAmerica Edison's Quality Assurance Manual (ED-QA-LQM).

4.0 Interferences

4.1. Interferences are discussed in the applicable analytical SOPs. Spiked samples are prepared to determine if any interference are present.

5.0 Safety

Employees must abide by the policies and procedures in the Corporate Environmental Health and Safety Manual (CW-E-M-001) and this document. This procedure may involve hazardous material, operations and equipment. This SOP does not purport to address all of the safety problems associated with its use. It is the responsibility of the user of the method to follow appropriate safety, waste disposal and health practices under the

assumption that all samples and reagents are potentially hazardous. Safety glasses, gloves, lab coats and closed-toe, nonabsorbent shoes are a minimum.

5.1. Specific Safety Concerns or Requirements

Samples that contain high concentrations of carbonates or organic material or samples that are at elevated pH can react violently when acids are added.

Hydrogen peroxide (H2O2) is a strong oxidizer and is corrosive. The digestion must be cooled sufficiently before the addition of H2O2 to avoid a reaction and possible violent effervescence, or boiling over of the digestion. A splash/splatter hazard is possible and a face shield should be worn.

5.2. Primary Materials Used

The following is a list of the materials used in this method, which have a serious or significant hazard rating. **Note: This list does not include all materials used in the method. The table contains a summary of the primary hazards listed in the MSDS for each of the materials listed in the table.** A complete list of materials used in the method can be found in the reagents and materials section. Employees must review the information in the MSDS for each material before using it for the first time or when there are major changes to the MSDS.

Material (1)	Hazards	Exposure Limit (2)	Signs and symptoms of exposure
Hydrochloric Acid	Corrosive Poison	5 ppm-Ceiling	Inhalation of vapors can cause coughing, choking, inflammation of the nose, throat, and upper respiratory tract, and in severe cases, pulmonary edema, circulatory failure, and death. Can cause redness, pain, and severe skin burns. Vapors are irritating and may cause damage to the eyes. Contact may cause severe burns and permanent eye damage.
Nitric Acid	Corrosive Oxidizer Poison	2 ppm-TWA 4 ppm-STEL	Nitric acid is extremely hazardous; it is corrosive, reactive, an oxidizer, and a poison. Inhalation of vapors can cause breathing difficulties and lead to pneumonia and pulmonary edema, which may be fatal. Other symptoms may include coughing, choking, and irritation of the nose, throat, and respiratory tract. Can cause redness, pain, and severe skin burns. Concentrated solutions cause deep ulcers and stain skin a yellow or yellow-brown color. Vapors are irritating and may cause damage to the eyes. Contact may cause severe burns and permanent eye damage.
Hydrogen Peroxide	Oxidizer Corrosive	1 ppm-TWA	Vapors are corrosive and irritating to the respiratory tract. Vapors are very corrosive and irritating to the eyes and skin.
1 – Always add acid to water to prevent violent reactions.			
2 – Exposure limit refers to the OSHA regulatory exposure limit.			

6.0 Equipment and Supplies

6.1. Instrumentation

- Block digestor: Environmental Express or equivalent
- AutoBlock: Environmental Express
- Weighing balance capable of accurate weighing to 0.01g.

6.2. Supplies

- watch glasses
- 75 mm funnels
- 50 ml Hot Block Digestion Cups
- Whatman # 41 filter paper or equivalent
- Pipettes varying volumes: Eppendorfs & Fisher
- 4oz Snap Cap Cups from Fisher or equivalent
- Reflux Caps, p/n SC506 Environmental Express or equivalent

7.0 Reagents and Standards

7.1. Reagents

7.1.1. Concentrated Nitric Acid- Trace Grade or equivalent; store at room temperature; for stability information, refer to manufacturer's instructions.
7.1.2. Concentrated Hydrochloric Acid- Trace Grade or equivalent; store at room temperature; for stability information, refer to manufacturer's instructions.
7.1.3. Reagent Grade Water 18 Megohm Minimum
7.1.4. Hydrogen peroxide (30%), H_2O_2. Store at room temperature; for stability information, refer to manufacturer's instructions.

7.2. Standards -

Storage requirements: all standards are stored at room temperature
Shelf-life: Stock standards – refer to manufacturer's instructions
Intermediate standards – 12 months
Working standards – 12 month
(Note: expiration date must not go beyond the expiration date of the source stock).
Concentration: see Attachment 3 for example certificates of analysis (COA) for all of the standards mixes listed below. The COA lists the manufacturer's part number, certified concentration and shelf life.

Standards must be prepared every 12 months or sooner if needed or required. "If needed" means the standard has been exhausted; "if required" means that the standard does not meet the QC criteria.

7.2.1. Stock ICP Spike Standards:

- Solutions A, B, C, and D (ICP-Spk), Part No. 4400-100419DD01: commercially purchased from CPI International. Store at room temperature; for stability information, refer to manufacturer's instructions. See Attachment 1 for the standards certified concentrations and catalog numbers.
- Sulfur 1000 mg/L, single element standard commercially purchased from CPI International. Store at room temperature; for stability information, refer to manufacturer's instructions

7.2.2. Working Spike Standard, ICP Working Spike Solution ME_ LCS-int (for ICP prep) - Add the following to a 1000 ml volumetric flask and bring to volume using 5% HNO_3: 50 ml each of Part No. 4400-100419DD01 Solutions A, B, C, and D (Sec 7.2.1.). Record standard preparations in TALS Reagent module. **Note**: Standard must not exceed the earliest expiration date of any one of its components.

7.2.3. Stock ICPMS Spike Standards (Method 6020B)

- ICPMS LCS/SPK: commercially purchased from High-Purity Standards. Store at room temperature; for stability information, refer to manufacturer's instructions. This stock standard doesn't require any pre-dilutions and is ready to be spiked directly into the QC samples. See Attachment 3 for the standards certified concentrations and catalog numbers.

7.2.4. Laboratory Control Sample Reference Material (LCSSRM), Metals in Soil: obtained from Environmental Resource Associates. Store at room temperature; for stability information, refer to manufacturer's instructions.

8.0 Sample Collection, Preservation, Shipment and Storage

Sample container, preservation techniques and holding times may vary and are dependent on sample matrix, method of choice, regulatory compliance, and/or specific contract or client requests. Listed below are the holding times and the references that include preservation requirements.

Matrix	Sample Container[1]	Min. Sample Size	Preservation	Holding Time[2]	Reference
Soils	Glass, plastic	5 grams	Cool 4 ±2°C	6 months	SW846 Method 3050B

[1] All sample containers must be demonstrated to be free of contaminants below the reporting limit.
[2] Holding time is 6 months, however sample analysis should begin as soon as possible.

9.0 Quality Control

9.1. Sample QC - The following quality control samples are prepared with each batch of samples digested or with every Sample Delivery Group (SDG), whichever comes first.

Quality Control	Frequency	Control Limit: Method 6010D & 6020B
Method Blank (MB)	1 in 20 or fewer samples	See the quality control section of the associated analytical SOP
Laboratory Control Sample(LCSSRM)	1 in 20 or fewer samples	Vendor's certified limits
Matrix Duplicate (DU)	1 in 20 or fewer samples	See the quality control section of the associated analytical SOP
Matrix Spike (MS)[1]	1 in 20 or fewer samples	75-125%
Matrix Spike Duplicate (MSD)[1]	When requested by client	See the quality control section of the associated analytical SOP

[1] The sample for DU/MS/MSD is randomly selected, unless specifically requested by a client; predetermined by the prep lab. Use the same environmental sample for the matrix spike and matrix duplicate sample whenever possible. If insufficient sample amount is available, another environmental sample may be used for the duplicate sample.
[2] Statistical control limits are updated annually and are updated into LIMS.

9.2. Instrument QC

None

10.0 Procedure

10.1. Sample Preparation

10.1.1. All digestion cups and filtration apparatus related to a sample must be labeled with that sample number.

10.1.2. Whenever reflux is required, sample should never be allowed to boil or "bump" as this could result in significant loss of analyte. Sample must never be allowed to go to dryness or be baked.

10.1.3. Mix the sample thoroughly to achieve homogeneity. Weigh 1.00-1.50

grams of sample to the nearest .01 g into a 50 ml hot block digestion cup. QC samples are prepared as follows:

10.1.3.1. Method Blank (MB): Use a blank 50 ml digestion cup for the Method Blank. Attach the TALS (TestAmerica Laboratory Systems) generated label which contains the sample ID number and batch number. The Method Blank is carried through the complete digestion process.

10.1.3.2. Matrix Duplicate (DU): Weigh 1.00-1.50 gm of a duplicate sample to the nearest 0.01 gram into a 50 ml hot block digestion cup. Attach the TALS generated label for the matrix duplicate sample.

10.1.3.3. Matrix Spike Sample (MS): Weigh 1.00 -1.50 gram of duplicate sample to the nearest 0.01 gram into a 50 ml hot block digestion cup. Attach the TALS generated label for the matrix spike sample.

- For ICP preps: Spike 2 ml of ME_LCS-int solution (ICP predigestion spike, Sec 7.2.2) to the digestion cup. If Sulfur is needed, also spike 0.4 mL of Sulfur 1000 mg/L (sec 7.2.1). The resulting elemental amounts are listed in Table 1 (attachment 1). NOTE: the spike must be added prior to the addition of any acids.
- For ICP-MS preps: spike the ICP-MS QC sample with 1.0 ml ICPMS LCS/SPK (Sec 7.2.3). The resulting elemental concentrations are listed in Attachment 2

10.1.3.4. Laboratory Control Sample Reference Material (LCSSRM): Weigh out 1.00-1.50 gm of LCS (sec 7.2.6) to the nearest 0.01 grams into a 50 ml hot block digestion cup. Attach the TALS generated label for the laboratory control sample.

10.1.3.5. Laboratory Control Sample (LCS) for Sulfur:
Using a 50 mL hot block cup, spike 0.4 mL of 1000 mg/L Sulfur (sec 7.2.1).

10.1.3.6. Matrix Spike Duplicate (MSD): when a MSD is requested by the client, prepare the sample in the same manner as the Matrix spike sample (MS), see Matrix Spike Sample prep instructions (Sec 10.1.3.3)..

10.1.4. Using Hot Block (if using AutoBlock, skip to section 10.1.5)

10.1.4.1. Add 10 ml of 1:1 HNO_3, mix the slurry, and cover with a watchglass. Transfer digestion cup to a hot block with the dial setting so that the contents of digestion cup are at

approximately 95°C ±3
°C and reflux for 10 to 15 minutes without boiling or drying the sample.

10.1.4.2. Allow sample to cool, add 5 ml of concentrated HNO_3, replace the watchglass, and reflux for 30 minutes.

10.1.4.3. Repeat step above until no brown fumes are generated.

10.1.4.4. Using a ribbed watchglass, cover the vessel and allow the solution to either evaporate to approximately 5ml or evaporate for 2 hours without boiling. Do not allow sample to go to dryness.

10.1.4.5. After step 10.1.7 has been completed, cool the sample; add 2 ml of deionized water and 3 ml of 30% H_2O_2. Warm cup, and make certain no losses occur due to vigorous effervescence. Heat until effervescence subsides and cool cup.

10.1.4.6. Continue to add 30% H_2O_2 in 1.0 ml aliquots with warming until the effervescence is minimal until the general sample appearance is unchanged. *Note*: Do not add a total of more than 10 ml of 30% H_2O_2.

10.1.4.7. Using a ribbed watchglass, cover the vessel and allow the solution to either evaporate to approximately 5ml or evaporate for 2 hours without boiling. Do not allow sample to go to dryness.

10.1.4.8. Add 10 ml of concentrated HCL to the sample and reflux for 15 min at 95°C ±3.

10.1.4.9. Filter the sample through Whatman 41 filter paper or equivalent into a 4oz Fisher snap cap cup or equivalent. Rinse the 50ml cup using DI water and filter into the 4oz cup. Using deionized water, bring to a final volume of 100 ml. This digestate is now ready for ICP or ICP-MS analyses.

10.1.5. Using AutoBlock

10.1.5.1. Place a Reflux Cap (sec 6.2) on every 50 ml hotblock cup.

10.1.5.2. Turn on AutoBlock then click the *AutoBlock Plus* icon on the computer desktop.

10.1.5.3. Check the waste drum. If needed, appropriately dump the waste.

10.1.5.4. Check the reagents (i.e., HCL, HNO3, 1:1HNO3, 30% H2O2, Deionized Water). Refill if necessary.

10.1.5.5. In the software, click the *Manuel Screen* tab. Enter 95 in the *Temp Control* box. Click *Enable Heat.*

10.1.5.6. Place a 50ml hotblock cup filled with DI water on the autoblock. Cover with a reflux cap. After the hotblock has reached a stable temperature of 95 deg C (~15min), record the temperature using a calibrated thermometer.

10.1.5.7. In the software, click *Exit.*

10.1.5.8. Click *Operator Test* and click *Select Method.*

10.1.5.9. Select *SOILmethod3050B.*

10.1.5.10. Under *Cell Position Selection*, click the hotblock locations where you will have the samples.

10.1.5.11. After closing the see-through plastic door, click on *Start Method.*

10.1.5.12. Don't open the door until the method has been completed.

10.1.5.13. Watch for brown fumes after Nitric Acid has been added by the autoblock. If brown fumes are generated, make note to discard this sample. This sample must be prepared using a Hot Block (sec 10.1.4).

10.1.5.14. When finished, *Method Completed* will be displayed in the software. Record the temp (sec 10.1.5.5).

10.1.5.15. Filter the sample through Whatman 41 filter paper or equivalent into a 4oz Fisher snap cap cup or equivalent. Rinse the 50ml cup using DI water and filter into the 4oz cup. Using deionized water, bring to a final volume of 100 ml. This digestate is now ready for ICP or ICP-MS analyses.

10.2. Calibration

10.2.1. The volume of the 100ml cups are verified for each lot received. The 100 ml volume verification with the appropriate Lot number is documented in the 'Metals labware volume verification logbook.'

10.2.2. Per manufacturer's instructions, recalibrate the AutoBlock every 30 days.

10.2.2.1. Place an empty hotblock cup on an analytical balance and 'zero' the balance.
10.2.2.2. Place that empty hotblock cup in the first position, I1, in the AutoBlock.
10.2.2.3. Click the *AutoBlock Plus* icon on the computer desktop
10.2.2.4. Click the *Manuel Screen* tab
10.2.2.5. Under *Reagent Selection*, click *H2O(6).* This is Port 6 which contains Deionized Water
10.2.2.6. Under *Cell Position Selection,* click *I1*
10.2.2.7. Under *System Control*, click *Move to Position*
10.2.2.8. In the *Injection Amount (ml)* box, type "30" and press *Inject.* The Autoblock will dispense 30 mL of DI Water into the cup.
10.2.2.9. Click *Move to Drain* and press *Exit*
10.2.2.10. Weigh the cup containing the DI Water on the zero'd balance to determine the weight of the water
10.2.2.11. Press the *Service Screen* tab
10.2.2.12. Press the *Pump Cal* tab
10.2.2.13. Under *Pump 2 Actual Injection Amount (ml)*, enter the weight of the water and press *Pump 2 Save Cal.*
10.2.2.14. Document the calibration on the Autoblock maintenance logbook.

10.3. Sample Analysis

Refer to the appropriate Analytical SOPs listed in Sec 1.2.

11.0. Calculations / Data Reduction

11.1. Accuracy:

$$\text{ICV / CCV, LCS \% Recovery} = \frac{\text{observed concentration}}{\text{known concentration}} \times 100$$

$$\text{MS \% Recovery} = \frac{\text{(spiked sample) - (unspiked sample)}}{\text{spiked concentration}} \times 100$$

11.2. Precision (RPD):

$$\text{Matrix Duplicate (MD)} = \frac{|\text{orig. sample value - dup. sample value}|}{[(\text{orig. sample value + dup. sample value})/2]} \times 100$$

11.3. Concentration :

Refer to appropriate Analytical SOPs listed in Sec 1.2.

11.4. Documenting and Reporting

11.4.1. Sample preparations must be documented in the Analyst Desktop II program located in TestAmerica Laboratory System (TALS).

11.4.1.1. Double click the TALS icon on the computer desktop. Enter your username and password and select Login.

11.4.1.2. Under TALS Menu, click the plus sign ('+') next to Analyst. Double click Analyst Desktop II. Click the plus sign ('+') next to Methods.

11.4.1.3. Right click the prep method that you are using to prep the samples. Select 'Create New Batch' then select 'From Scratch.'

11.4.1.4. The analyst must enter the following information: Sample names (use scanners), initial and final sample volume, spike name and amount used, and all reagents and their corresponding lot numbers.

11.4.1.5. After saving the batch information in TALS, select Print-Batch Sheets (which must be submitted to metals analytical dept. along with the samples) and select Print-Batch Labels (which must be attached to the corresponding final digestate sample cups in the batch).

11.4.2. Complete the Data Review Checker (DRC) in TALS: Prior to data submission (i.e., prior to 1st leveling data in the Analyst Desktop II module in TALS), the analyst must execute the DRC (Data Review Checker) program in the applicable preparation batch.

11.4.2.1. Open the preparation batch and click on the Edit tab above to enter the Edit Mode.

11.4.2.2. Press F8 command key on the computer keyboard or right click anywhere on the batch worksheet then click on 'Run checklist.'

11.4.2.3. Acknowledge by filling in responses to all unacknowledged findings.

11.4.2.3.1. Highlight the checklist with findings, then highlight the associated list of findings; right click and choose 'Acknowledge Item.'

11.4.2.3.2. Fill in the appropriate comments in the response box, then hit 'OK.'

11.4.2.3.3. Acknowledge all Finding items in the 'Manual Batch Checklist' except for the *"2nd Level review complete?"* this is to be completed by the 2nd level reviewer.

12.0. Method Performance

12.1. Method Detection Limit Study (MDL)

The method detection limit (MDL) is the lowest concentration that can be detected for a given analytical method and sample matrix with 99% confidence that the analyte is present. The MDL is determined according to the laboratory's MDL procedure in Section 19 (Test Methods and Method Validation) of TestAmerica Edison's Quality Assurance Manual (ED-QA-LQM). MDLs reflect a calculated (statistical) value determined under ideal laboratory conditions in a clean matrix, and may not be achievable in all environmental matrices. The laboratory maintains MDL studies for analyses performed; these are verified at least annually unless method requirements require a greater frequency.

12.2. Demonstration of Capabilities

For DOC procedure refer to Section 19 in the most current revision of TestAmerica Edison's Quality Assurance Manual (ED-QA-LQM).

12.3. Training Requirements

Refer to TestAmerica SOP No. ED-GEN-022, Training, for the laboratory's training program.

13.0. Pollution Control

It is TestAmerica's policy to evaluate each method and look for opportunities to minimize waste generated (i.e., examine recycling options, ordering chemicals based on quantity needed, preparation of reagents based on anticipated usage and reagent stability). Employees must abide by the policies in Section 13 of the Corporate Environmental Health and Safety Manual (CW-E-M-001) for "Waste Management and Pollution Prevention."

14.0. Waste Management

14.1. Waste management practices are conducted consistent with all applicable rules and regulations. Excess reagents, samples and method process wastes are disposed of in an accepted manner. Waste description rules and land disposal restrictions are followed. Waste disposal procedures are incorporated by reference to TestAmerica SOP No. ED-SPM-008 (Laboratory Waste Disposal Procedures).

14.2. The following waste streams are produced when this method is carried out:

- Digested Samples: Corrosive Acid- Materials that are not above regulatory limits will be submitted for elementary neutralization with 50% sodium hydroxide solution (Siedler Chemical SC-1824-03). Major concern is heat generated from the neutralization process. Initial volume of acid waste to be neutralized should be no more than 15 gallons. Finished neutralization with sodium bicarbonate (Siedler Chemical SC-0219-25) to a pH of 6 – 9 in the

primary tank. Once pH has been established the primary tank is transferred through filter housing to a secondary tank. The pH is rechecked. If the pH is within specifications, the secondary tank is released to the municipal sewer system.

- Samples above regulatory limits and expired RCRA metals standards (Waste Corrosive Liquid, Acidic, Inorganic, n.o.s.) are collected in satellite accumulation and sent off site through a Waste disposal vendor.
  Onyx Profile WIP Number: 590598
  Teris Profile Number 50016653

- Soil Retain Samples - These samples if not flagged in the system for any hazardous constituents are transferred to poly-lined cubic yard boxes. These boxes when full are sent to stabilization or incineration. These materials are sent out as hazardous for lead and chromium

  Teris Profile Number (incineration): 50016710
  Onyx Profile Number: (stabilization) 402535

15.0. References / Cross-References

15.1. Test Methods for Evaluating Solid Waste Physical/Chemical Methods (SW846), Third Edition, September 1986, Final Update I, July 1992, Final Update IIA, August 1993, Final Update II, September 1994; Final Update IIB, January 1995; Final Update III, December 1996; SW-846, Method 3050B.

15.2. TestAmerica Edison Document No. ED-QA-LQM, *Laboratory Quality Manual*, most current revision.

15.3. TestAmerica Corporate Environmental Health and Safety Manual, Document No. CW-E-M-001, most current revision.

15.4. TestAmerica Edison SOP ED-GEN-022, *Training*, most current revision.

15.5. TestAmerica Edison SOP No. ED-MT-004, Trace Metals Analysis by Inductively Coupled Plasma Emission Spectroscopy via SW846 Method 6010D, most current revision.

15.6. TestAmerica Edison SOP No ED-MT-029, Trace Metals Analysis for Water, Wastewater, Soil,Sediment and Leachate Samples by ICP-MS Method SW-846 6020B, most current revision.

15.7. TestAmerica Edison SOP ED-MT-034, *SW846 Method 6020B, Trace Metals Analysis of Water, Wastewater, Soil, Sediment, and Leachate Samples by ICPMS*, most current revision.

16.0. Method Modifications:

None

17.0. Attachments

Attachment 1: Table 1, Matrix Spike concentration for Soil samples by 3050B-ICP
Attachment 2: Table 2, Matrix Spike concentration for Soil samples by 3050B-ICPMS
Attachment 3: Certificate of analysis of each stock standard.

18.0. Revision History

- Revision 15, dated 29 September 2021
  - Sec. 10.1.3.3: Revised the amount of ICPMS LCS/SPK added for ICP-MS preps to 1.0 ml to reflect actual laboratory practices.

- Revision 14, dated 30 December 2019
  - Sec 6.1: Added AutoBlock to the instrument list.
  - Sec 6.2: Added Reflux Caps to the supplies list
  - Sec 7.2.1: Added Sulfur to the stock ICP spike standards list.
  - Sec 10.1.3.3: Added matrix spiking instructions for Sulfur.
  - Sec 10.1.3.5: Added LCS preparation instructions for Sulfur.
  - Sec 10.1.5: Added instructions for using the AutoBlock.
  - Sec 10.2.1: Replaced the 50ml volume verification with a 100ml volume verification.
  - Sec 10.2.2: Added procedure for the monthly recalibration (volume dispenser) of the Autoblock.
  - Table 1: Added spike amount for Sulfur.

- Revision 13, dated 05 August 2019
  - Updated SOP header with Eurofins emblem.
  - Throughout the SOP deleted all references to Methods 6010B, 6010C, 6020 and 6020A.
  - Sec 2.0: Revised the final volume of digestates from 50ml to 100ml to reflect actual lab practices.
  - Sec 6.2: Added 4oz snap seal cups to the list of supplies.
  - Sec 10.1.3.3: Revised the spiking volume to add for ICP-MS LCS from 1ml to 2ml.
  - Sec 10.1.12: Clarified instructions for filtering digestates; added final volume of digestates.

- Revision 12, dated 06 September 2018
  - Throughout the SOP - revised to reference the analytical methods 6010D and 6020B.
  - Sec 11.4.: Updated instructions for documenting and reporting preparation batch information in TALS and instruction for using TALS Data Review Checker (DRC) program.

- Revision 11, dated 13 July 2016

  - Sec 1.1: Added method 6010C and 6020A
  - Sec 1.2: Deleted reference to SOP ED-MT-033; SOP has been combined with SOP ED-MT-004.
  - Sec 9.1: Added MSD to the list of Sample QC including the frequency and control limit requirements.
  - Sec 10.1.3.5: Added preparation instructions for MSD.
  - Sec 15.7: Removed reference to SOP No ED-MT-033, subsequent section adjusted accordingly.

- Revision 10, dated 05 June 2014

  - Sec 1.2: Removed SOP reference ED-MT-031 and added SOP reference ED-MT-033 and ED-MT-034.
  - Sec 2.0: Corrected typo error for the final volume from 100 ml to 50 ml as described in this SOP.
  - Sec 6.2: Removed 125 ml specimen cups.
  - Sec 10.1.1: Deleted the use of glassware – not applicable for this SOP.
  - Sec 15.7 & 15.8: Added TestAmerica Edison SOP No. ED-MT-033 and ED-MT-034 to the list of reference.
  - Attachment 1: Added Boron to the ICP matrix spike list.
  - Attachment 2: Added Zinc to the ICPMS matrix spike list and deleted Mercury from this list; corrected Boron's matrix spike concentration.

- Revision 9, dated 31 May 2012

  - Sec 1.3 and 12: Revised LQM references to reflect the most current LQM revision.
  - Throughout the document: Removed all references to CLP SOW ISM 01.2
  - Sec 6.1: added 'or equivalent'.
  - Sec 7.2.1: Updated the ICP stock standard to reflect current lab practices.
  - Sec 7.2.2: Updated the preparation of the working spike standard.
  - Sec 7.2.3: Replaced the vendor for the ICPMS stock standards to High-Purity Standards (former vendor- CPI International).
  - Deleted Sec 7.2.4 & 7.2.5 (CLP ISM01.2 Standards, not applicable); subsequent sections adjusted accordingly.
  - Sec 9.1 Table: MB and DU control limits have been updated and now reference the associated analytical SOP for the most current QC limits.
  - Sec 10.1.3.1 – 10.1.3.4: Updated the digestion cup labeling procedure using the TALS generated labels
  - Sec 10.1.12: Revised procedure to reflect actual lab practices - *samples are brought to a 50ml final volume and then filtered.* Final digestate volume revised from 100 ml to 50 ml.
  - Sec 10.2.1: Replaced the 125 ml hot-block cups with 50 ml size cups
  - Sec 13 & 14: Updated to comply with TestAmerica Corporate Quality SOP No. CW-QS-002, Writing a Standard Operating Procedure (SOP), most current revision.
  - Attachment 3: Replaced COAs to reflect the updated stock standards.

- Revision 8, dated 30 March 2010

  - Sec 1.1: Deleted Table which listed the 6010B RLs for soils; added Sec 1.2 to include list of analytical SOPs where the appropriate RLs can be found.
  - Sec 7: Added matrix spike and LCS standards for Method 6020 and ISM01.2
  - Sec 8: Added the holding time for soil samples analyzed by Method CLP SOW ISM01.2.
  - Sec 9.1: Added control limits for CLP SOW ISM01.2.; Deleted Blank spike and LCS duplicate.
  - Sec 10: Revised amount of sample to be weighed to 1.00-1.50 grams as per CLP Method ISM01.2
  - Sec 10: Revised hot block temperature to 95± 3 deg, to follow the more stringent setting.
  - Deleted section previously identified 10.1.3.2, Blank spike; subsequent sections adjusted.
  - Sec 10.1.3.3 & Sec 10.1.3.4: Revised to include spike procedures for ICP and ICPMS, Method 6020 and ISM01.2.
  - Sec 10.1.3.5: Deleted LCS duplicate
  - Sec 10.1.9: Section added to reflect actual lab practices; subsequent section numbers adjusted.
  - Sec 10.1.10: Text added: '*Note: For samples which are being digested for CLP SOW ISM01.2 ICP-MS, do not perform this step.'*
  - Sec 15: Added applicable references.
  - Sec 17: Added attachments 2 & 3
  - Attachment 3: Added applicable COAs
  - Added this revision history.

- Revision 7, dated June 13, 2008
  - Revised SOP format in accordance with TestAmerica Corporate Quality SOP No. CW-QS-002 (Rev 0) Writing a Standard Operating Procedure (SOP).
  - Section 1.1: Added laboratory's reporting limits in Table format.
  - Section 7 Reagents & Standards: Included storage and stability information of reagents and standards. Updated the source of the stock spike standards and the preparation instructions for the working spike standards.
  - Removed all reference related to GFAA analysis including standard solutions, digestion procedure and Table 2: Spike amount for soil samples by GFAA; clarified the digestion procedure for ICP-AES analysis.
  - Section 10.1 Sample Preparation: Deleted acid rinsing of glassware since 50 ml digestion cups are being used for digesting samples.
  - Section 10.1 Sample Preparation: Replaced hot plate setting of 2.5 to hot block setting of 95°C.
  - Section 15 References: Added applicable references

Attachment 1

TABLE 1

Matrix Spike Concentration for Soil Samples by ICP	
ELEMENT	SPIKE AMOUNT (mg/Kg)
Aluminum	200
Antimony	50
Arsenic	200
Barium	200
Beryllium	5.0
Cadmium	5.0
Boron	50
Calcium	2000
Chromium	20
Cobalt	50
Copper	25
Iron	100
Lead	50
Manganese	50
Magnesium	2000
Molybdenum	50
Nickel	50
Potassium	2000
Selenium	200
Silver	5.0
Sodium	2000
Strontium	50
Thallium	200
Tin	50
Titanium	50
Vanadium	50
Zinc	50
Sulfur	400

Matrix spike sample concentration: Dry weight correction is made at final calculation

Attachment 2

ICPMS Matrix Spike Concentration Method 6020	
Element	**Matrix Spike Conc. (mg/Kg)**
Aluminum (Al)	500
Antimony (Sb)	5
Arsenic (As)	10
Barium (Ba)	10
Beryllium (Be)	5
Boron (B)	100
Cadmium (Cd)	5
Calcium (Ca)	500
Chromium (Cr)	10
Cobalt (Co)	5
Copper (Cu)	10
Iron (Fe)	500
Lead (Pb)	5
Magnesium(Mg)	500
Manganese (Mn)	50
Molybdenum (Mo)	10
Nickel (Ni)	10
Potassium (K)	500
Selenium (Se)	10
Silver (Ag)	5
Sodium (Na)	500
Strontium (Sr)	10
Thallium (Tl)	4
Tin (Sn)	10
Titanium (Ti)	10
Vanadium (V)	10
Zinc (Zn)	50

Note: For Matrix spike sample concentration, dry weight correction is made at final calculation.

Attachment 3

ERA
A Waters Company

Certificate of Analysis

Lot No. D075-540

Metals in Soil

Catalog No. 540
Issue Date: October 11, 2011
Revision Date: Original

Certification

Parameter	Total Concentration [1] (mg/kg)	Certified Value [2] (mg/kg)	Uncertainty [3]	QC PALs™ [4] (mg/kg)	PT PALs™ [5] (mg/kg)
aluminum	68100*	10100	0.8%	5110 - 15100	4560 - 15600
antimony	228	113	0.8%	DL - 252	22.7 - 286
arsenic	266	237	2.9%	197 - 278	169 - 306
barium	678	252	2.4%	212 - 292	186 - 318
beryllium	105	93.3	4.1%	77.7 - 109	69.4 - 117
boron	135	118	1.3%	87.3 - 149	71.9 - 164
cadmium	215	191	4.5%	159 - 223	140 - 242
calcium	25400*	6840	5.3%	5640 - 8040	5080 - 8600
chromium	364	128	3.8%	104 - 151	89.3 - 166
cobalt	208	178	3.5%	149 - 207	132 - 223
copper	145	123	9.0%	103 - 143	92.3 - 154
iron	42900*	13100	4.8%	6650 - 19600	4290 - 22000
lead	105	103	6.7%	85.6 - 120	73.0 - 132
magnesium	9060	2990	4.4%	2280 - 3710	1980 - 4010
manganese	918	333	5.5%	274 - 391	250 - 415
mercury	12.8	12.4	10.1%	8.88 - 15.9	6.36 - 18.4
molybdenum	113	91.1	4.8%	74.2 - 108	63.3 - 122
nickel	152	118	6.0%	96.8 - 139	85.8 - 150
potassium	19700*	2870	4.0%	1950 - 3780	1780 - 3950
selenium	123	110	1.6%	86.6 - 134	72.6 - 148
silver	51.6	47.3	3.8%	31.3 - 63.2	31.4 - 63.2
sodium	17400*	550	3.8%	405 - 694	289 - 811
strontium	252	128	4.0%	104 - 153	90.6 - 166
thallium	177	158	3.7%	128 - 188	108 - 208
tin	121	107	0.8%	84.5 - 130	61.7 - 153
titanium	3280*	282	4.8%	65.4 - 498	0.00 - 606
vanadium	205	119	4.0%	94.9 - 143	81.2 - 157
zinc	197	183	6.9%	150 - 216	127 - 239

Please see footnotes on back

DL = Detection Limit

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The Netherlands

SOLN. A
DE
4-4-11

Expiry: 21-Sep-12

Certificate of Analysis

Part Number: **4400-100419DD01**
Lot Number: **11C157**
Shelf Life: **18 Months**

Date Recd
03/22/11 MP

TestAmerica/Edison
Custom Standard
5% HNO3

Concentrations in ug/mL ± 0.5%

Fe	1000	Zn	500
Se	2000	Mn	500
TL	2000	Ni	500
Be	50		
Cd	50		
Cr	200		
Co	500		
Cu	250		
Pb	500		
Ag	50		
Sr	500		
V	500		

This standard solution was prepared using high-purity starting materials, high-purity acid (if required) and 18-megaohm de-ionized water. The starting materials were weighed to five significant figures and diluted in volumetric glassware calibrated to five significant figures.

Starting materials were analyzed at 1000µg/mL by ICP-MS for trace impurities. The standard solution concentrations were certified instrumentally against the National Institute of Standards and Technology's SRM 3100 series, NIST approved second source and/or gravimetrically.

Accuracy and stability are guaranteed to within plus or minus 0.5% of the certified value for the stated shelf life from the date of shipment. The solution should be kept tightly capped and stored under normal laboratory conditions. See attached MSDS for proper handling information.

For questions or comments please call 1-800-878-7654 in the USA, +31 20 638 05 97 in Europe or visit our web-site at www.cpiinternational.com.

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Expiry: 21-Sep-12

Certificate of Analysis

Part Number: **4400-100419DD01** **Solution B**
Lot Number: **11C157**
Shelf Life: **18 Months**

TestAmerica/Edison
Custom Standard
10% HNO3

Date Recd
03/22/11 MP

Concentrations in ug/mL ± 0.5%

Al	2000
Mg	20000
K	20000
Ca	20000
Na	20000

This standard solution was prepared using high-purity starting materials, high-purity acid (if required) and 18-megaohm de-ionized water. The starting materials were weighed to five significant figures and diluted in volumetric glassware calibrated to five significant figures.

Starting materials were analyzed at 1000µg/mL by ICP-MS for trace impurities. The standard solution concentrations were certified instrumentally against the National Institute of Standards and Technology's SRM 3100 series, NIST approved second source and/or gravimetrically.

Accuracy and stability are guaranteed to within plus or minus 0.5% of the certified value for the stated shelf life from the date of shipment. The solution should be kept tightly capped and stored under normal laboratory conditions. See attached MSDS for proper handling information.

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Expiry: 21-Sep-12

Certificate of Analysis

Part Number: **4400-100419DD01** **Solution C**
Lot Number: **11C157**
Shelf Life: **18 Months**

Date Rec'd
03/22/11 MP

TestAmerica/Edison
Custom Standard
5% HNO3 + 2% HF

Concentrations in ug/mL ± 0.5%

Sb	500
Mo	500
Sn	500
Ti	500

This standard solution was prepared using high-purity starting materials, high-purity acid (if required) and 18-megaohm de-ionized water. The starting materials were weighed to five significant figures and diluted in volumetric glassware calibrated to five significant figures.

Starting materials were analyzed at 1000µg/mL by ICP-MS for trace impurities. The standard solution concentrations were certified instrumentally against the National Institute of Standards and Technology's SRM 3100 series, NIST approved second source and/or gravimetrically.

Accuracy and stability are guaranteed to within plus or minus 0.5% of the certified value for the stated shelf life from the date of shipment. The solution should be kept tightly capped and stored under normal laboratory conditions. See attached MSDS for proper handling information.

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P.O. Box 2704 P: +31 20 638 05 97
1000 CS Amsterdam F: +31 20 420 28 36
The Netherlands

Expiry: 21-Sep-12

Certificate of Analysis

Part Number: **4400-100419DD01** **Solution D**
Lot Number: **11C157**
Shelf Life: **18 Months**

Date Rec'd
05/22/11 MP

TestAmerica/Edison
Custom Standard
2% HNO3

Concentrations in ug/mL ± 0.5%

As	2000
Ba	2000
B	500

This standard solution was prepared using high-purity starting materials, high-purity acid (if required) and 18-megaohm de-ionized water. The starting materials were weighed to five significant figures and diluted in volumetric glassware calibrated to five significant figures.

Starting materials were analyzed at 1000µg/mL by ICP-MS for trace impurities. The standard solution concentrations were certified instrumentally against the National Institute of Standards and Technology's SRM 3100 series, NIST approved second source and/or gravimetrically.

Accuracy and stability are guaranteed to within plus or minus 0.5% of the certified value for the stated shelf life from the date of shipment. The solution should be kept tightly capped and stored under normal laboratory conditions. See attached MSDS for proper handling information.

SM-606-111
(ICPMS LCS/SPK)
Element List
(µg/mL)

Aluminum	500
Antimony	5
Arsenic	10
Barium	10
Beryllium	5
Boron	100
Cadmium	5
Calcium	500
Chromium	10
Cobalt	5
Copper	10
Iron	500
Lead	5
Magnesium	500
Manganese	50
Molybdenum	10
Nickel	10
Potassium	500
Selenium	10
Silver	5
Sodium	500
Strontium	10
Thallium	4
Tin	10
Titanium	10
Vanadium	10
Zinc	50

Title: Extraction of Semi-Volatile Organic Compounds in Aqueous Samples and Leachates - Separatory Funnel, SW846 Method 3510C

Once printed, this is considered an uncontrolled document

Approvals (Signature/Date):

Sylvanus Klusey	03/26/2018 Date	Dan Helfrich	03/26/2018 Date
Organics Operations Manager		Health & Safety Manager / Coordinator	
Carl Armbruster	03/26/2018 Date	Mark Acierno	03/26/2018 Date
Quality Assurance Manager		Laboratory Director	

1.0 Scope and Application

1.1. **Analytes, Matrix(s), and Reporting Limits** SW846 Method 3510C describes a procedure for isolating semivolatile organic compounds from aqueous samples and leachates, including concentration techniques suitable for preparing the extract for GC/MS analysis. This SOP is applicable to the isolation and concentration of water-soluble and slightly water-soluble semivolatile organics in preparation for analysis by SW846 Methods 8270C or 8270D.

1.2. For a complete discussion of analytes and reporting limits (RLs) please refer to TestAmerica Edison SOP Nos. ED-MSS-002, *Semivolatile Organic Compounds by Gas Chromatography/Mass Spectrometry (GC/MS) SW846 Method 8270C*, current revision and ED-MSS-009, *Semivolatile Organic Compounds by Gas Chromatography/Mass Spectrometry (GC/MS) SW846 Method 8270D*, current revision

1.3. On occasion clients may request modifications to this SOP. These modifications are handled following the procedures outlined in Section 7 (*Review of Work Request*) and 19 (*Test Methods and Method Validation*) of TestAmerica Edison's Quality Assurance Manual (ED-QA-LQM).

2.0 Summary of Method

2.1. A measured volume of sample (~250 mL) is serially extracted with methylene chloride at a pH less than 2 and again at a pH greater than 11 using separatory funnel extraction. The methylene chloride extract is dried and concentrated to a volume of approximately 2 mL. Nitrogen blowdown is employed as the final concentration step. The extract is subsequently analyzed by SW846 Method 8270C or 8270D (GC/MS) by a large volume injection (LVI) technique. This procedure is referred to throughout as Reduced Volume Extraction (RVE) and Large Volume Injection (LVI).

2.2. An option for preparing aqueous samples using a larger initial volume (~1000 ml) is also described. This procedure is referred to as 'large volume' throughout the document.

3.0 Definitions

For a complete list of definitions refer to Appendix 2 in the most current revision of TestAmerica Edison's Quality Assurance Manual (ED-QA-LQM).

4.0 Interferences

4.1. Solvents, reagents, glassware, and other sample hardware may yield artifacts and/or interferences to sample analysis. All these materials must be demonstrated to be free from interferences under the conditions of the analysis by analyzing method blanks.

4.2. Phthalate esters contaminate many products commonly found in the laboratory. Plastics, in particular, must be avoided, because phthalates are often used as plasticizers and are easily extracted from plastic material.

Phthalate contamination may result at any time if consistent quality control is not practiced.

4.3. The decomposition of some analytes has been demonstrated under basic extraction conditions. Phthalate esters may exchange and phenols may react to form tannates. These reactions increase with increasing pH, and are decreased by shorter reaction times. Performing the initial extraction at an acid pH will optimize the recovery of phenols

5.0 Safety

Employees must abide by the policies and procedures in the Corporate Environmental Health and Safety Manual (CW-E-M-001) and this document. This procedure may involve hazardous material, operations and equipment. This SOP does not purport to address all of the safety problems associated with its use. It is the responsibility of the user of the method to follow appropriate safety, waste disposal and health practices under the assumption that all samples and reagents are potentially hazardous. Safety glasses, gloves, lab coats and closed-toe, nonabsorbent shoes are a minimum.

5.1. Specific Safety Concerns or Requirements

During Kuderna-Danish (KD) concentration, do not allow the extract to boil to dryness. The solvent vapors remaining in the KD apparatus may superheat and create an explosion or fire hazard.

The following analytes have been tentatively classified as known or suspected, human or mammalian carcinogens: benzo(a)anthracene, benzidine, 3,3'dichlorobenzindine, benzo(a)pyrene, alpha-BHC, beta-BHC, gamma-BHC, delta-BHC, dibenz(a,h)anthracene, N-nitrosodimethylamine, 4,4'-DDT, and polychlorinated biphenyl compounds. Primary standards of these toxic compounds should be prepared in hood.

The use of Kevlar gloves is required for the assembly/disassembly of ground glass joints in addition to those tasks that present the potential risk for injury.

The use of separatory funnels to extract aqueous samples with Methylene Chloride creates excessive pressure very rapidly. Initial venting should be done immediately after the sample container has been sealed and inverted, periodic venting may be necessary during the extraction. Vent the funnel into the hood away from people and other samples. This is considered a high-risk activity, the use of a face shield over safety glasses or goggles is recommended. Keep the sash on the fume hood as low as reasonably possible.

5.2. Primary Materials Used

The following is a list of the materials used in this method, which have a serious or significant hazard rating. **Note: This list does not include all materials used in the method. The table contains a summary of the primary hazards listed in the MSDS for each of the materials listed in the table.** A complete list of materials used in the method can be found in

the reagents and materials section. Employees must review the information in the MSDS for each material before using it for the first time or when there are major changes to the MSDS.

Material (1)	Hazards	Exposure Limit (2)	Signs and symptoms of exposure
Methylene Chloride	Carcinogen Irritant	25 ppm-TWA 125 ppm-STEL	Causes irritation to respiratory tract. Has a strong narcotic effect with symptoms of mental confusion, light-headedness, fatigue, nausea, vomiting and headache. Causes irritation, redness and pain to the skin and eyes. Prolonged contact can cause burns. Liquid degreases the skin. May be absorbed through skin.
Sulfuric Acid	Corrosive Oxidizer Dehydra-dator	1 mg/m^3	This material will cause burns if comes into contact with the skin or eyes. Inhalation of vapors will cause irritation of the nasal and respiratory system.
Sodium Hydroxide	Corrosive Poison	2 ppm, 5 mg/m^3	This material will cause burns if comes into contact with the skin or eyes. Inhalation of Sodium Hydroxide dust will cause irritation of the nasal and respiratory system.
Methanol (MeOH)	Flammable Poison Irritant	200 ppm-TWA	A slight irritant to the mucous membranes. Toxic effects exerted upon nervous system, particularly the optic nerve. Symptoms of overexposure may include headache, drowsiness and dizziness. Methyl alcohol is a defatting agent and may cause skin to become dry and cracked. Skin absorption can occur; symptoms may parallel inhalation exposure. Irritant to the eyes.
1 – Always add acid to water to prevent violent reactions.			
2 – Exposure limit refers to the OSHA regulatory exposure limit.			

6.0 Equipment and Supplies

6.1. Instrumentation

- Separatory funnel rotator, APR Machine or equivalent
- Analytical Evaporator (N-Evap) Organomation
- Centrifuge, Varifuge F; Hereaus Sepatech
- Six Position Steam Bath, Fisher 15-496 or equivalent

6.2. Supplies

- 250 ml Erlenmeyer Flask, AMK Glass ERL-0252 or equivalent
- 2000 ml or 500 ml Separatory Funnel, AMK Glass SFC or equivalent
- 100 mm o. d. glass funnels, Fisher or equivalent

- 10 ml jacketed, graduated Concentrator Tubes, AMK Glass KD-0018 or equivalent
- 19/22 Ground Glass Stoppers
- 3 Ball Snyder Columns, TEC Glass TG6-03 or equivalent
- 1 ml Gastight Syringe, Hamilton 81317 or equivalent
- 150 ml Centrifuge Tube
- 100 ml Graduated Cylinder
- Pasteur 5 ¾" Disposable Pipets, Fisher 13-678-20B or equivalent
- Kuderna Danish Flask (500 ml), TEC Glass TG7-01 or equivalent
- Vials, 2ml amber screw cap with Teflon liner
- Glass Wool
- Dessicator
- Standard Taper Clamps (Size 19, blue)
- Boiling Stones, Troemner P/N 133-B or equivalent, rinsed with Methylene Chloride
- pH Paper
- Watch Glass
- Wax Pencil
- 1 Liter Graduated Cylinder
- Marking Tags

7.0 Reagents and Standards

7.1 Reagents

Note: Each lot of Methylene Chloride, Acetone, Methanol and Sulfuric Acid is screened for contaminants before being used for analysis as detailed in TestAmerica Corporate Quality SOP No. CA-Q-S-001 (*Solvent & Acid Lot Testing & Approval*) and TestAmerica Edison SOP No. ED-GEN-023 (*Bulk Solvent Testing and Approval*).

7.1.1 Methylene Chloride – JT Baker Ultra-Resi 9254-03 or equivalent

7.1.2 Acetone, J.T. Baker Ultra-Resi 9264-03 or equivalent

7.1.3 Methanol, J.T. Baker, Pesticide Grade, 9077-02 or equivalent

7.1.4 Concentrated Sulfuric Acid - Baxter 2876-9 or equivalent

7.1.5 Sodium Hydroxide Pellets – Baxter 7708-500NY or equivalent

7.1.6 Sodium Sulfate Crystals – Mallinckrodt MA8024-06 or equivalent (Must be baked in the muffle furnace for four hours at 400°C and serially rinsed with Methylene Chloride prior to use.)

7.1.7 Sodium Hydroxide (10 N) - Fill a precleaned 1000 ml volumetric flask with 500 mls deionized water. Weigh out 400 g NaOH pellets and dispense slowly into the flask. Stir slowly until the pellets dissolve, then add more deionized water until the 1000 ml level is

reached. Be careful as this procedure generates heat. Never add water to the reagent that is to be dissolved.

7.2 Standards

7.2.1 Most stock target analyte standard solutions are purchased as prepared solutions; other standards are prepared in the laboratory using neat compounds (see table below). Most stock solutions are diluted (in volumetric glassware) to working concentration using methylene chloride as the diluent as described below. Stock standards of similar quality from other suppliers may be substituted as required.

NOTE: The standards listed here are used as calibration standards and spiking standards. Separate source calibration verification standards are addressed in the analytical SOPs.

Standard Name	Concentration	Vendor	Catalog #
8270 List 1/ Std#1 MegaMix	500/1000/2000pmm	RESTEK	570666
8270 List 1/ Std#10Benzoic Acid	2000ppm	RESTEK	569731
8270 List 1/ Std#9	2000ppm	RESTEK	569730
Custom SVO Mix	2000ppm	SPEX	SVO-TANJ-16-5
Bisphenol A	1000ppm	SPEX	S-509-MC
8270 List 1/ Std#11	2000ppm	RESTEK	569732
8270 Surrogate Standard	5000 ppm	RESTEK	567685
Aromatic Amines Custom Mix	2000 ppm	Supelco	21467482

7.2.1.1. **Spiking Standard:** For use in spiking aqueous samples including TCLP/SPLP leach being prepared for BNA analysis by SW846 Method 8270. Prepare the second source spiking solution for MS/MSD/Blank Spike (LCS) as detailed in the following table using the applicable standards listed in 7.2.1. Use volumetric glassware. Dilute to the volume marker with methanol.

Standard Name	Concentration	Volume of Standard added to final volume of 200ml (solvent)	Final Concentration
8270 List 1/ Std#1 MegaMix	500/1000/2000pmm	20 ml (methanol)	50/100/200 ppm
8270 List 1/ Std#10	2000ppm	10 ml (methanol)	100 ppm
8270 List 1/	2000ppm	10 ml (methanol)	100 ppm

Standard Name	Concentration	Volume of Standard added to final volume of 200ml (solvent)	Final Concentration
Std#9			
Custom SVO Mix-SPEX	2000ppm	10 ml (methanol)	100 ppm
Bisphenol A-SPEX	1000ppm	10ml (methanol)	50ppm
8270 List 1/ Std#11	2000ppm	20ul (methanol) 100ul(methanol)	Used neat for LVI Used neat for non-LVI
Aromatic Amines Custom Mix**	2000ppm	10 ml (methylene chloride)	100 ppm

*** As needed based on clients requirement.*

Note: Neat spiking/surrogate standards are stored per vendor requirements (either room temp or 4 deg C, as appropriate). All prepared standard solutions are refrigerated at 4 deg C.

7.2.2 **8270 Surrogate Standard Spiking Solution**: For use in spiking all blanks, samples and associated QC prior to extraction. Prepare as detailed in the following table using the applicable standards listed in 7.2.1. Use volumetric glassware. Dilute to the volume marker with methanol.

Standard Name	Concentration	Volume of Standard added to final volume of 1000 ml methanol	Final Concentration
8270 Surrogate Standard	5000 ppm	20 ml	100 ppm

7.2.3 Internal standard is prepared added by the analytical department. For details see analytical SOPs No. ED-MSS-002 (SW846 8270C) or ED-MSS-009 (SW846 8270D).

7.2.4 The preparation of all standards must be documented in TestAmerica LIMS (TALS) or a standard preparation logbook. Information such as standard supplier, lot number, original concentration, and a description of how standard was prepared are required along with a laboratory lot number, analyst's initials, date prepared and verification signature. Standards must be made every 6 months or sooner if signs of degradation appear.

8.0 Sample Collection, Preservation, Shipment and Storage

Sample container, preservation techniques and holding times may vary and are dependent on sample matrix, method of choice, regulatory compliance, and/or specific contract or client requests. Listed below are the holding times and the references that include preservation requirements.

8.1 All samples must be stored at 4ºC (± 2ºC) upon receipt.

8.2 Sample Extract Storage. Samples extracts must be protected from light and refrigerated at 4ºC (± 2ºC) from time of extraction until analysis.

8.3 Sample Extract Holding Time. All sample extracts must be analyzed within 40 days of extraction.

Matrix	Sample Container	Min. Sample Size	Preservation	Holding Time	Reference
Water or Leachate	250ml Amber (RVE)/ Amber glass, 1L	250ml-RVE/ 1000 ml	Cool 4 ± 2°C	7 days to extraction; Analyze within 40 days of extraction	EPA Method SW846 8270C

9.0 Quality Control

9.1. **Sample QC** - The following quality control samples are prepared with each batch of samples. Refer to TestAmerica Edison analytical SOPs No. ED-MSS-002 (SW846 8270C*)* or ED-MSS-009 (SW846 8270D) for details on analysis and evaluation of these QC elements:

Quality Controls	Frequency	Control Limit
Method Blank (MB)	1 in 20 or fewer samples	< Rpt. Limit
Laboratory Control Sample (LCS)[1]	1 in 20 or fewer samples	Statistical Limits [4]
Matrix Spike (MS)[2]	1 in 20 or fewer samples	Statistical Limits [4]
MS Duplicate (MSD)[2]	1 in 20 or fewer samples	Statistical Limits [4]
Surrogates	every sample[3]	Statistical Limits [4]

[1] LCS Duplicate (LCD) is performed only when insufficient sample is available for the MS/MSD or when requested by the client/project/contract.
[2] The sample selection for MS/MSD are randomly selected, unless specifically requested by a client….predetermined by the extraction lab.
[3] Analytical and QC samples (MB, LCS, MS/MSD, Method Blank)
[4] Statistical control limits are updated annually and are updated into lab reporting software.

9.1.1. **Method blanks** are extracted with every sample batch on each day that samples are extracted.

9.1.2. **Matrix Spike (MS)/Matrix Spike Duplicate (MSD):** A Matrix Spike/Matrix Spike Duplicate (MS/MSD) pair is extracted and analyzed with every batch of 20 environmental samples. (Note: an

LCS/LCSD may be substituted for the MS/MSD if insufficient client environmental sample volume is available).

9.1.3. Laboratory Control Sample (LCS)/Laboratory Control Sample Duplicate (LCSD): A Laboratory Control Sample (LCS) (aka blank spike) must be extracted and analyzed with each batch of 20 environmental samples.

9.1.3.1 A Laboratory Control Sample Duplicate (LCSD) is extracted and analyzed only when insufficient client sample is available for preparation of an MS/MSD pair. The LCS/LSCD is evaluated in the same manner as the MS/MSD (see Section 9.1.2)

9.1.4. Surrogate Standards: All samples, blanks and QC samples are spiked with a six (6) component surrogate standard mix (see Section 7.2.2).

10.0 Procedure

NOTE: The sample preparation procedure for aqueous samples (Section 10.1) contains two options: reduced volume extraction (RVE) (250ml) for large volume injection (LVI) analysis and large volume extraction (1000ml),

10.1. Sample Preparation for Aqueous/ Leachates Samples

10.1.1. Rinse the required number of 500-ml separatory funnels (when RVE is required) or 2000-ml separatory funnels (when large volume extraction is required) and 250-ml Erlenmeyer flasks twice with a 1:1 mixture of Methylene Chloride:Acetone and once with Methylene Chloride.

10.1.2. Place a small amount of glass wool into a 100-mm funnel and fill with pre-baked sodium sulfate crystals. Rinse three (3) times with Methylene Chloride. Also rinse the outside of the funnel stem three (3) times with Methylene Chloride (since the stem is likely to come into contact with the extract). Allow time for all of the rinsate to drain out of the funnel into a waste container.

10.1.3. Record the lab sample numbers on the separatory funnels with red wax pencil.

10.1.4. Make up tags with the following information and place on a 250ml Erlenmeyer flask:

BNAs		**BNs**	**AEs**
Acid Fraction	BN Fraction		
Sample Number	Sample Number	Sample Number	Sample Number
Fraction-Matrix	Fraction-Matrix	Fraction-Matrix	Fraction-Matrix
Date of Extraction	Date of Extraction	Date of Extraction	Date of Extraction

10.1.5. Place the 100ml funnel containing rinsed sodium sulfate crystals onto the flask.

10.1.6. Mark the fluid level on the sample bottles with a black Sharpie. Pour each sample into its corresponding separatory funnel. Fill each sample bottle to the black line with tap water. Pour this into the graduated cylinder used for measuring sample volumes. Note the volume for each sample on the Organic Extraction Data Sheet.

10.1.7. Rinse out a graduated cylinder with lab reagent water two to three times. Using the graduated cylinder obtain 250 ml when RVE is used (or 1000-ml when large volume is required) of lab reagent water from the Millipore filtering apparatus located in the Wet Chemistry laboratory for each of the method blank and the laboratory control sample (LCS) (aka blank spike).

10.1.8. Pour each the reagent water for the method blank and LCS into the corresponding separatory funnels.

10.1.9. Rinse syringes eight (8) to ten (10) times with Methylene Chloride.

10.1.10. If you are performing BNAs add 0.2 ml when RVE is used (or 1.0 ml when large volume is required) of Surrogate Standard Spiking Solution (see Section 7.2.2) to each sample/QC sample and proceed to Section 10.1.11. If you are performing *BN only* extraction, add 0.2 ml when RVE is used (or 1.0 ml when large volume is required) of Surrogate Standard Spiking Solution (see Section 7.2.2) to each sample/QC sample and proceed to Section 10.1.20. If you are performing *AE only* extraction add 0.2 ml when RVE is used (or 1.0 ml when large volume is required) of Surrogate Standard Spiking Solution (see Section 7.2.2) to each sample/QC sample and proceed to Section 10.1.11.

10.1.10.1. If extracting QC samples (MS, MSD, LCS or LCSD), add 0.2 ml when RVE is used (or 1.0 ml when large volume is required of the Spiking (see Section 7.2.1.1) to the appropriate separatory funnel. Note: When spiking the samples, make sure to get all bubbles out of the syringe. In addition, hold the syringe just above the level of the liquid when adding the spike. Do not touch the tip of the syringe to the liquid or the side of the separatory funnel.

10.1.11. Add concentrated sulfuric acid to each sample to adjust the pH to <2. (Usually you only need to add 1ml using small disposable Pasteur pipette). Note: pH adjustments must be documented in the extraction log.

10.1.12. Shake each sample for a short time and check pH using pH paper. The pH must be 2 or less. If the pH has not been lowered sufficiently, add more acid.

10.1.13. Add 15 ml of Methylene Chloride when RVE is used (or 60 ml when large volume is required) to each sample bottle.

10.1.14. Swirl the bottle and add 15 ml of Methylene Chloride when RVE is used (or 60 ml when large volume is required) to its corresponding separatory funnel.

10.1.15. After making sure the funnels are properly secured, start the rotators. Stop the rotator and vent the funnels after about 10 seconds. Resume rotating for 2 minutes.

10.1.16. Stop the rotation and allow the sample to settle.

10.1.17. Drain the bottom (organic) layer from the separatory funnel into the funnel/Erlenmeyer apparatus.

10.1.18. Repeat steps 10.1.13 through 10.1.17 twice, adding the Methylene Chloride directly to separatory, rather than rinsing the sample container as in 10.1.15.

10.1.18.1. If an emulsion forms during extraction, rinse a centrifuge tube well with methylene chloride and then drain the lower layer from the separatory funnel into the tube. Centrifuge for 3 to 5 minutes at 2000 rpm. Make sure the levels (and weights) of the samples directly opposite one another in the centrifuge are approximately equal. After the centrifuging process is finished, there will be two layers: water on top and the methylene chloride on the bottom. A 1 ml disposable pipette should be used to transfer the methylene chloride (bottom) layer from the centrifuge tube to the appropriate Erlenmeyer flask. With this method, care must be taken not to transfer the water (top) layer. The top layer that remains is poured back into the separatory funnel with the rest of the original sample.

10.1.19. If you are preparing the sample for acid extractables analysis only, you are finished and you can now discard the remaining liquid in each separatory funnel and proceed to Section 10.1.27. If you are required to extract base/neutrals, proceed with Section 10.1.20.

10.1.20. Adjust the pH of the sample to >11 by adding 10N sodium hydroxide (NaOH) to the sample in each separatory funnel. NOTE: pH adjustment must be documented in the extraction logbook.

10.1.21. Shake each separatory funnel for a short time and check pH. It should be basic, >pH 11. If the pH is not as high as it should be, add more 10N sodium hydroxide (NaOH).

10.1.22. Add 15 ml of Methylene Chloride when RVE is used (or 60 ml when large volume is required)

10.1.23. After making sure the funnels are properly secured, start the rotators. Stop the rotator and vent the funnels after about 10 seconds. Resume rotating for 2 minutes.

10.1.24. Stop the rotation and allow the sample to settle.

10.1.25. Drain the bottom (organic) layer from the separatory funnel into the funnel/Erlenmeyer apparatus.

10.1.26. Repeat steps 10.1.22 through 10.1.25 twice.

10.1.26.1. If an emulsion forms during extraction, rinse a centrifuge tube well with methylene chloride and then drain the lower layer from the separatory funnel into the tube. Centrifuge for 3 to 5 minutes at 2000 rpm. Make sure the levels (and weights) of the samples directly opposite one another in the centrifuge are approximately equal. After the centrifuging process is finished, there will be two layers: water on top and the methylene chloride on the bottom. A 1 ml disposable pipette should be used to transfer the methylene chloride (bottom) layer from the centrifuge tube to the appropriate Erlenmeyer flask. With this method, care must be taken not to transfer the water (top) layer. The top layer that remains is poured back into the separatory funnel with the rest of the original sample.

10.1.27. Pour the entire methylene chloride extract from the Erlenmeyer flask into a KD concentration tube apparatus (pre-rinsed three times with acetone). Rinse the Erlenmeyer flask from which the sample came twice with methylene chloride and pour both rinsates into the KD apparatus.

10.1.28. Attach Snyder column (pre-rinsed three times with acetone) to the top of the KD apparatus.

10.1.29. For RVE extractions blow the extract directly down to a final volume of 2-ml on the N-Evap. Remove the Snyder column from the top of the KD flask. Remove the blue taper clamp from the ground glass joint and dry the exterior with a Kimwipe. Transfer the concentrator tube with the 5ml extract to the N-Evap and “blow down” the extract until the volume is 2.0 ml.

10.1.30. For large volume (1000 ml) extractions concentrate the extract to approximately 5-ml in Steam Bath Remove the Snyder column from the top of the KD flask. Remove the blue taper clamp from the ground glass joint and dry the exterior with a Kimwipe. Transfer the concentrator tube with the 5ml extract to the N-Evap and “blow down” the extract until the volume is 1.0 ml. Bring the volume of each extract up to 2ml with methylene chloride

10.1.31. Split each extract into two (2) -1ml aliquots and transfer each aliquot to a separate 2ml amber screw cap vial with Teflon liner. Label one vial as 'SIM" and the second vial as 'Total'. Transfer custody of the vials to the Semivolatile GC/MS laboratory for analysis (see TestAmerica Edison analytical SOPs No. ED-MSS-002 (SW846 8270C*)* or ED-MSS-009 (SW846 8270D).

10.2. Required Documentation:

The organic prep technician is responsible for completing the following items.

10.2.1. The Standards Prep Logbook or TALS Reagent Database must be completed in full with the required information whenever standards are logged and/or prepared.

10.2.2. Each time an extraction is performed, the applicable TALS data record must be completed and reviewed the Organic Prep Supervisor or designee. Lot numbers of all reagents and solvents used or added to samples during preparation must be documented in the database.

10.2.3. Each sample extracted must be included in a batch and be recorded in the TALS database.

10.2.4. Following the extraction procedure, the technician must complete all TALS data fields pertaining to the samples extracted.

11.0. Calculations/Data Reduction

n/a

12.0. Method Performance

12.1. Method Detection Limit Study (MDL)

The method detection limit (MDL) is the lowest concentration that can be detected for a given analytical method and sample matrix with 99% confidence that the analyte is present. The MDL is determined according to the laboratory's MDL procedure in Section 19 (Test Methods and Method Validation) of TestAmerica Edison's Quality Assurance Manual (ED-QA-LQM). MDLs reflect a calculated (statistical) value determined under ideal laboratory conditions in a clean matrix, and may not be achievable in all environmental matrices. The laboratory maintains MDL studies for analyses performed; these are verified at least annually unless method requirements require a greater frequency.

12.2. Demonstration of Capabilities

For demonstration of capability procedure refer to Section 19 in the most current revision of TestAmerica Edison's Quality Assurance Manual (ED-QA-LQM).

12.3. Training Requirements

Refer to TestAmerica SOP No. ED-GEN-022, *Training*, for the laboratory's training program.

13.0. Pollution Control

13.1 It is TestAmerica's policy to evaluate each method and look for opportunities to minimize waste generated (i.e., examine recycling options, ordering chemicals based on quantity needed, preparation of reagents based on anticipated usage and reagent stability). Employees must abide by the policies in Section 13 of the Corporate Environmental Health and Safety Manual (CW-E-M-001) for "Waste Management and Pollution Prevention."

14.0. Waste Management

14.1 Waste management practices are conducted consistent with all applicable rules and regulations. Excess reagents, samples and method process wastes are disposed of in an accepted manner. Waste description rules and land disposal restrictions are followed. Waste disposal procedures are incorporated by reference to TestAmerica Edison SOPs Nos. ED-SPM-007 (*Disposal of Samples and Associated Laboratory Waste, current revision)* and ED-SPM-008 (*Laboratory Waste Disposal Procedures, current revision).* The following waste streams are produced when this method is carried out:

- Extractions Waste water. This material is created when 50% Acetone and 50% Methylene Chloride are added to 1 liter of sample water. The water is shaken with the solvent. The solvent is collected with the compounds of interest and the water is discarded into the Extractions Waste Water drum. This drum is removed to the walk-in hood in the waste room. A ½ inch PVC pipe is inserted into the bung hole of the drum and air is passed through the solution over night. The solution is then transferred into the first drum of the neutralization system and neutralized to a pH of 6 – 9. This solution is discharged into the municipal sewer system.

- Mixed Solvent Waste. This material is collected from rinsing and other processes into 5 gallon solvent cans as satellite accumulation. These cans are emptied every 24 hours into a steel drum in the waste room. This drum is kept in the walk in hood until it is full. The full drum is then removed from the hood and placed on secondary containment in the waste room.
  Teris Profile Number: 50016624
  Onyx Profile WIP Number: 545240

- Waste sodium sulfate. This material is collected from various methods which require the removal of water from solvent which carries the analyte (s) of interest. The solvent is passed through the sodium sulfate and the sodium sulfate plus the water is disposed of. The sodium sulfate is collected in buckets inside the hoods. The material is air dried and disposed of in the municipal waste dumpster.

- Waste sulfuric acid. This material is generated from clean up of PCB extracts for sulfur compounds. The acid is collected in satellite accumulation in the hood. The container is removed to the waste room for neutralization with 50 % sodium hydroxide (Siedler Chemical SC-1824-03), water and sodium bicarbonate (Siedler Chemical SC-0219-25). Ice is used to control temperature in the plastic drums of the neutralization system. When neutralization is complete (pH 6 -9) the material is transferred to the municipal sewer system.

15.0. References / Cross-References

15.1 United States Environmental Protection Agency, “Method SW3510C, Separatory Funnel Extraction”, Test Methods for Evaluating Solid Wastes, SW846 Third Edition, Volume 1B: Laboratory Manual, Physical/Chemical Methods, Revision 3, December 1996.

15.2 TestAmerica Edison SOP No. ED-MSS-002, *Semivolatile Organic Compounds by Gas Chromatography/Mass Spectrometry (GC/MS, SW846 Method 8270C*, current revision.

15.3 TestAmerica Edison SOP No. ED-MSS-009, *Semivolatile Organic Compounds by Gas Chromatography/Mass Spectrometry (GC/MS) SW846 Method 8270D*, current revision

15.4 TestAmerica Edison Document No. ED-QA-LQM, *Laboratory Quality Manual*, current revision.

15.5 TestAmerica Environmental Health and Safety Manual, CW-E-M-001.

15.6 TestAmerica Corporate Quality SOP No. CA-Q-S-001 (*Solvent & Acid Lot Testing & Approval*), current revision.

15.7 TestAmerica Edison SOP No. ED-GEN-023 (*Bulk Solvent Testing and Approval*), current revision.

15.8 TestAmerica Edison SOP No. ED-GEN-022, *Training,* current revision

15.9 TestAmerica Edison SOPs Nos. ED-SPM-007 (*Disposal of Samples and Associated Laboratory Waste, current revision)*

15.10 TestAmerica Edison SOP No. ED-SPM-008 (*Laboratory Waste Disposal Procedures, current revision).*

16.0. Method Modifications:

N/A

17.0. Attachments

N/A

18.0. Revision History

Revision 12, effective 03/26/2018

- Updated throughout to clarify that the lab's standard procdure is to use Reduced Volume Extraction (~250 ml initial volume) with an option to use large volume extraction (~1000 ml initial volume).

Revision 10, effective 11/29/2016:

- Sections 7.2.1 and 7.2.1.1 : updated current sources of standards.
- Section 7.2.1.1: added standards storage information as note at end of section.
- Section 8.0: added option for 250 ml amber sample containers (LVI option)

Revision 9, effective 11/21/2014:

- Section 7.2: updated current sources of all standards.
- Throughout document as required: added option for preparation of leachates by LVI.
- Section 10.1.29 through 10.1.31: clarified the concentration techniques for both full volume and LVI extracts.

Revision 8, effective 11/28/2012

- Throughout document: updated references to Lab Quality Manual section numbers.
- Added references as necessary throughout to TestAmerica Edison SOP No. ED-MSS-009 (*Semivolatile Organic Compounds by GC/MS, SW846 Method 8270C*D, current revision.
- Section 2.2 added describing option for analysis of lower initial volume for subsequent analysis using large volume injection (LVI) technique.
- Section 6.2: added 500 ml separatory funnel.
- Sections 7.2.1 and 7.2.1.1: added '5 Compound BNA Custom Mix' and 'Aromatic Amine Custom Mix' to list of standards and prep instructions table.
- Section 10.1 (Sample Prep for Aqueous Samples): revised throughout to include option for extraction of reduced aqueous volume (250ml) for subsequent analysis by LVI technique. Added note as preface to Section 10.1 alerting analyst to the two available extraction volume options (1000ml and 250ml).

Revision 7, effective 12/6/10

- Section 3: revised to reference new location for definitions

- Sections 7.2.1 and 7.2.1.1: Added 4 compounds and additional details to the description of standard preparation of the Spiking Standard.
- Section 7.2.4: added option to document standards preparation within TALS rather than a laboratory notebook.
- Section 10.3: revised to include TALS as the main repository for raw data associated with sample prep.

Revision 6, effective November, 2008

- Revised SOP format in accordance with TestAmerica Corporate Quality SOP No. CW-QS-002 (Rev 0) Writing a Standard Operating Procedure (SOP).
- Revised title to include 'Leachates'.
- Section 1.3: Added reference to Quality Assurance Manual for method modifications.
- Section 3: revised to reference new location for definitions.
- Section 5: Revised to include most up to date corporate health and safety references and information.
- Section 7: added details of the solvent testing and approval program.
- Section 7.2.1: Added additional details to the description of standards and the preparation of the Spiking Standards and Surrogate Standards. Removed references to the Internal Standard which is now added by the analytical group and is discussed in TestAmerica Edison SOP No. ED-MSS-002, *Semivolatile Organic Compounds by Gas Chromatography/Mass Spectrometry (GC/MS), SW846 Method 8270C*, current revision.
- Section 9: Quality Control: added additional details to the discussion of the various QC sample types
- Section 10: Revised and clarified to reflect current procedures. Removed reference to internal standard addition (now completed by analytical group).
- Section 11: Removed reference to Organic Calculation SOP.
- Section 12: updated and revised the MDL requirements to reflect text in the current revision of the TestAmerica Edison Laboratory Quality Manual (LQM).
- Section 15: References: Expanded to include more specific SOP references
- Section 16: Added Section 16 (Method Modifications).
- Section 18: Added this Revision History section

Title: Extraction of Pesticides and PCBs in Water by Separatory Funnel using SW846 Method 3510C

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Approvals (Signature/Date):

06/27/19		06/27/19	
Mamadou Leye Organic Prep Department Manager	Date	Dan Helfrich Health & Safety Manager	Date
06/27/19			06/27/19
Carl Armbruster Quality Assurance Manager	Date	Mark Acierno Laboratory Director	Date

06/27/19
Sylvanus Klusey Date
Operations Manager - Organics

Facility Distribution No. ELECTRONIC **Distributed To:** FACILITY INTRANET

1.0 Scope and Application

1.1. **Analytes, Matrix(s), and Reporting Limits:** SW846 Method 3510C describes a procedure for isolating organic compounds from aqueous samples, including concentration techniques suitable for preparing the extract for GC and/or GC/MS analysis. This SOP is specifically applicable to the isolation and concentration of water-soluble and slightly water-soluble Pesticides and PCBs using Method 3510C in preparation for analysis GC methods SW846 8081B, 8082A and GC/MS method EPA 680.

1.2. For a complete discussion of analytes and reporting limits (RLs) refer to the SOP for the applicable analytical method:.

- TestAmerica Edison SOP No. ED-GCS-016, *Analysis of Organochlorine Pesticides by SW846 Method 8081B,* current revision.
- TestAmerica Edison SOP No. ED-GCS-017, *Analysis of Polychlorinated Biphenyls by SW846 Method 8082A,* current revision.
- TestAmerica Edison SOP No. ED-MSS-010, *Determination of PCBs in Water and Soil/Sediment by GC/MS, EPA Method 680*, current revision.

1.3. On occasion clients may request modifications to this SOP. These modifications are handled following the procedures outlined in Section 7 (*Review of Work Request*) and 19 (*Test Methods and Method Validation*) of TestAmerica Edison's Quality Assurance Manual (ED-QA-LQM).

2.0 Summary of Method

2.1 A measured volume of aqueous sample is spiked with surrogates and serially extracted with methylene chloride using a separatory funnel. The extract is dried by passing it through activated sodium sulfate, concentrated using a nitrogen blowdown technique and exchanged into hexane prior to analysis by Methods 8081B, 8082A or 680. Cleanup of extracts may be required prior to analysis.

2.2 The preferred option for preparing aqueous samples for analysis by SW8081B/8082A includes use of a reduced initial volume (~250ml) for analysis by a large volume injection (LVI). A technique for extraction of a larger volume (1000 ml) is also described.

3.0 Definitions

For a complete list of definitions refer to Appendix 2 in the most current revision of TestAmerica Edison's Quality Assurance Manual (ED-QA-LQM).

4.0 Interferences

Phthalate esters contaminate many products commonly found in the laboratory. Plastics, in particular, must be avoided, because phthalates are often used as

plasticizers and are easily extracted from plastic material. Phthalate contamination may result at any time if consistent quality control is not practiced.

Method interference may be caused by contaminants in solvents, reagents, glassware and other sample processing hardware that lead to discrete artifacts or elevated baselines in gas chromatograms. All these materials must be routinely demonstrated to be free from interference under the conditions of the analysis, by analyzing method and reagent blanks.

5.0 Safety

Employees must abide by the policies and procedures in the Corporate Environmental Health and Safety Manual (CW-E-M-001) and this document. This procedure may involve hazardous material, operations and equipment. This SOP does not purport to address all of the safety problems associated with its use. It is the responsibility of the user of the method to follow appropriate safety, waste disposal and health practices under the assumption that all samples and reagents are potentially hazardous. Safety glasses, gloves, lab coats and closed-toe, nonabsorbent shoes are a minimum.

5.1. Specific Safety Concerns or Requirements

Nitrile gloves should be used when performing this extraction. Latex and vinyl gloves provide no significant protection against the organic solvents used in this SOP, and should not be used.

The use of separatory funnels to extract aqueous samples with methylene chloride creates excessive pressure very rapidly. Initial venting should be done immediately after the sample container has been sealed and inverted. Vent the funnel into the hood away from people and other samples. This is considered a high-risk activity, and a face shield must be worn over safety glasses or goggle when it is performed.

The use of Kevlar gloves is required for the assembly/disassembly of ground glass joints in addition to those tasks that present the potential risk for injury.

5.2. Primary Materials Used

The following is a list of the materials used in this method, which have a serious or significant hazard rating. **Note: This list does not include all materials used in the method. The table contains a summary of the primary hazards listed in the MSDS for each of the materials listed in the table.** A complete list of materials used in the method can be found in the reagents and materials section. Employees must review the information in the MSDS for each material before using it for the first time or when there are major changes to the MSDS.

Material (1)	Hazards	Exposure Limit (2)	Signs and symptoms of exposure
Methylene Chloride	Carcinogen Irritant	25 ppm-TWA 125 ppm-STEL	Causes irritation to respiratory tract. Has a strong narcotic effect with symptoms of mental confusion, light-headedness, fatigue, nausea, vomiting and headache. Causes irritation, redness and pain to the skin and eyes. Prolonged contact can cause burns. Liquid degreases the skin. May be absorbed through skin.
Acetone	Flammable	1000 ppm-TWA	Inhalation of vapors irritates the respiratory tract. May cause coughing, dizziness, dullness, and headache.
Hexane	Flammable Irritant	500 ppm-TWA	Inhalation of vapors irritates the respiratory tract. Overexposure may cause lightheadedness, nausea, headache, and blurred vision. Vapors may cause irritation to the skin and eyes.
1 – Always add acid to water to prevent violent reactions (if applicable).			
2 – Exposure limit refers to the OSHA regulatory exposure limit.			

6.0 Equipment and Supplies

6.1. Equipment

- Steam Bath - Fisher Scientific 66738 or equivalent.
- Muffle Furnace - Thermolyne Type 6000 or equivalent.
- N-Evap - Meyer Analytical Evaporator Model No. 112 or equivalent
- Separatory Funnel Rotator, APR Machine or equivalent
- Centrifuge, Varifuge F; Hereaus Sepatech

6.2. Supplies

- 400 ml Clear Glass Jar
- 500 ml Separatory Funnel, AMK Glass SFC-0095 or equivalent
- 100 mm o. d. glass funnels, Fisher or equivalent
- 10 ml jacketed, graduated Concentrator Tubes, AMK Glass KD-0018 or equivalent
- 19/22 Ground Glass Stoppers

- 50 ul Gastight Syringe, Hamilton 80900 or equivalent
- 100 ul Gastight Syringe, Hamilton 81000 or equivalent
- 150 ml Centrifuge Tube
- 100 ml Graduated Cylinder
- Pasteur 5¾" Disposable Pipettes, Fisher 13-678-20B or equivalent
- Glass Wool
- Desiccators
- Standard Taper Clamps (Size 19, blue)
- Wax Pencil
- 1 Liter Graduated Cylinder
- Marking Tags
- pH paper

7.0 Reagents and Standards

7.1 Reagents

Note: Each lot of Methylene Chloride and Acetone, is screened for contaminants before being used for analysis as detailed in TestAmerica Corporate Quality SOP No. CA-Q-S-001 (*Solvent & Acid Lot Testing & Approval*) and TestAmerica Edison SOP No. ED-GEN-023 (*Bulk Solvent Testing and Approval*).

- Methylene Chloride - JT Baker Ultra-Resi 9254-03 or equivalent
- Sodium Sulfate Crystals – Mallinckrodt MA8024-06 or equivalent
- Acetone, J. T. Baker Ultra-Resi 9264-03 or equivalent
- Hexane, Pesticide Grade, Baxter 217-4or equivalent
- Organic free reagent water - Reagent water is defined as a water in which an interference is not observed at the MDL of each parameter of interest (ASTM Specification D1193, Type ii). At TestAmerica Edison this water is generated by the Barnstead/Thermolyne Water System (Model # D11991 Serial # 1191020210415).

7.1.1 Reagent preparation

7.1.1.1 Anhydrous sodium sulfate crystals (Mallinckrodt MA8024-06 or equivalent) must be baked in the muffle furnace for four hours at 400^{o}C and serially rinsed with methylene chloride prior to use.

7.2 Standards

7.2.1 Standards are purchased as concentrated solutions (see Section 7.2.2). Most stock solutions are diluted (in volumetric glassware) to working concentration using acetone as the diluent as described in Section 7.3.

7.2.2 **Standard mixes and sources:** Table 1 lists the standard concentrate mix sources. Table 2 details the individual components of these mixes.

Table 1a:
Pesticide/PCB Standard Mixes and Sources (8081/8082)*

Standard Name ("Lab Name")	Concentration in ug/ml (each component)	Source - Catalog #
Pesticide Surrogate Spike Mix	200	RESTEK-32000
Aroclor Spike Mix (1660 Aroclor)	1000	RESTEK-32039
Organochlorine Pesticides Mix ("Pest Spike")	2000	RESTEK-32415
Chlordane (technical) ("Technical Chlordane Spike")	5000	RESTEK-32072
Toxaphene	5000	RESTEK-32071

*May be substituted with equivalent standards from alternate sources.

Table 1b:
PCB Homologue Standard Mixes and Sources (EPA 680)*

Standard Name ("Lab Name")	Concentration in ug/ml (each component)	Source - Catalog #
Retention Time Calibration Standard Mixture	Varies	Ultra-CB682-MN
Concentration Calibration Standard Mix	Varies	Ultra-CB681-MN
$^{13}C_{12}$-Decachlorobiphenyl Surrogate	40	Cambridge-EC1410-3
Lindane13C6	100	Cambridge-CL1282-S

Table 2a:
Components of Pesticide/PCB Standard Mixes (8081/8082)

Parameter	Catalog Nos.	Lab Standard Name	Conc (ug/ml)
Decachlorobiphenyl (DCB)	Pesticide Surrogate Spike Mix	RESTEK-32000	200
2,4,5,6-Tetrachloro-m-xylene (TCMX)	Pesticide Surrogate Spike Mix	RESTEK-32000	200
Aroclor 1016	Aroclor Spike Mix	RESTEK-32039	1000
Aroclor 1260	Aroclor Spike Mix	RESTEK-32039	1000
Aldrin	Organochlorine Pesticides Mix	RESTEK-32415	2000
alpha-BHC	Organochlorine Pesticides Mix	RESTEK-32415	2000
beta-BHC	Organochlorine Pesticides Mix	RESTEK-32415	2000
Lindane	Organochlorine Pesticides Mix	RESTEK-32415	2000
g-BHC	Organochlorine Pesticides Mix	RESTEK-32415	2000
DDD	Organochlorine Pesticides Mix	RESTEK-32415	2000
4,4'-DDE	Organochlorine Pesticides Mix	RESTEK-32415	2000

Table 2a: Components of Pesticide/PCB Standard Mixes (8081/8082)			
Parameter	**Catalog Nos.**	**Lab Standard Name**	**Conc (ug/ml)**
4,4'-DDT	Organochlorine Pesticides Mix	RESTEK-32415	2000
Dieldrin	Organochlorine Pesticides Mix	RESTEK-32415	2000
alpha-Endosulfan	Organochlorine Pesticides Mix	RESTEK-32415	2000
beta-Endosulfan	Organochlorine Pesticides Mix	RESTEK-32415	2000
Endosulfan Sulfate	Organochlorine Pesticides Mix	RESTEK-32415	2000
Endrin	Organochlorine Pesticides Mix	RESTEK-32415	2000
Endrin aldehylde	Organochlorine Pesticides Mix	RESTEK-32415	2000
Endrin ketone	Organochlorine Pesticides Mix	RESTEK-32415	2000
Heptachlor	Organochlorine Pesticides Mix	RESTEK-32415	2000
Hetptachlor epoxide	Organochlorine Pesticides Mix	RESTEK-32415	2000
Methoxychlor	Organochlorine Pesticides Mix	RESTEK-32415	2000
Chlordane (technical)	Technical Chlordane Spike	RESTEK-32072	5000
Toxaphene	Toxaphene Spike	RESTEK-32071	5000

Table 2b: Components of PCB Homologue Standard Mixes (680)			
Parameter	**Catalog Nos.**	**Lab Standard Name**	**Conc (ug/ml)**
PCB-104	Ultra-CB682-MN	Retention Time Calibration Standard Mix	100
PCB-208	Ultra-CB682-MN	Retention Time Calibration Standard Mix	200
PCB-77	Ultra-CB682-MN	Retention Time Calibration Standard Mix	100
Total Nonachlorobiphenyls	Ultra-CB682-MN	Retention Time Calibration Standard Mix	200
DCB Decachlorobiphenyl	Ultra-CB681-MN	Concentration Calibration Standard Mix	250
Total Dichlorobiphenyls	Ultra-CB681-MN	Concentration Calibration Standard Mix	50
Total Heptachlorobiphenyls	Ultra-CB681-MN	Concentration Calibration Standard Mix	150
Total Hexachlorobiphenyls	Ultra-CB681-MN	Concentration Calibration Standard Mix	100
Total Monochlorobiphenyls	Ultra-CB681-MN	Concentration Calibration Standard Mix	50
Total Octachlorobiphenyls	Ultra-CB681-MN	Concentration Calibration Standard Mix	150
Total Pentachlorobiphenyls	Ultra-CB681-MN	Concentration Calibration Standard Mix	100
Total Tetrachlorobiphenyls	Ultra-CB681-MN	Concentration Calibration Standard Mix	100
Total Trichlorobiphenyls	Ultra-CB681-MN	Concentration Calibration Standard Mix	50
Decachlorobiphenyl-13C12	Cambridge-EC1410-3	$^{13}C_{12}$-Decachlorobiphenyl Surrogate	40
Lindane13C6	Cambridge-CL1282-S	Lindane13C6	100

7. 3. Standards Preparation

7.3.1. All standard stock solutions are diluted to the working concentrations with hexane or acetone (as indicated) using Class A volumetric glassware. **Note:** septa on all surrogate and spike vials are to be replaced immediately after use. Additionally, all surrogate and spike vials are to be returned to the standards refrigerator immediately after use.

7.3.2. **Pesticide Spiking Standard (LCS/MS/MSD): For the reduced volume extraction option:** prepare a 4 ug/ml spiking solution by diluting 10 ml of the 20ug/ml standard prepared above to a final volume of 50ml in acetone. For spiking instructions refer to Section 10.

7.3.3. **8082 PCB Spiking Standard (LCS/MS/MSD):**. **For the reduced volume extraction option:** prepare a 20 ug/ml spiking solution by diluting 10 ml of the 100 ug/ml stock standard prepared above to a final volume of 50ml in acetone. For spiking instructions refer to Section 10.

7.3.4. **680 PCB Homologue Spiking Standard (LCS/MS/MSD):** The EPA Method 680 PCB homologue spiking standard is prepared by diluting 1.0 ml of the Retention Time Calibration Standard Mix (Ultra-CB682-MN) and 1.0 ml of the Concentration Calibration Standard Mix (Ultra-CB681-MN) to 100mL of hexane using volumetric glassware. For spiking instructions refer to Section 10.

7.3.5. **Technical Chlordane Spiking Standard (LCS/MS/MSD):** The technical Chlordane spiking solution is prepared at a final concentration of 100 ug/ml by diluting 0.2 ml of the 5000 ug/ml Technical Chlordane Spike solution (RESTEK-32072, see Tables 1 and 2) to a 10 ml final volume using acetone. For spiking instructions refer to Section 10.

7.3.6. **Toxaphene Spiking Standard (LCS/MS/MSD):** A Toxaphene spiking solution is prepared at a final concentration of 100 ug/ml by diluting 0.2 ml of the 5000 ug/ml Toxaphene Spiking Standard (RESTEK-32071, see Tables 1 and 2) to a 10 ml final volume with acetone. For spiking instructions refer to Section 10.

7.3.7. **Pesticide Surrogate Spiking Standard: For the reduced volume extraction option:** prepare a 2 ug/ml spiking solution by diluting 10 ml of the 10 ug/ml stock standard described above to a final volume of 50ml in acetone. For spiking instructions refer to Section 10.

7.3.8. **680 PCB Homologue Surrogate Spiking Standard:** The EPA Method 680 surrogate spiking standard is prepared by diluting 3.125 ml of Decachlorobiphenyl-13C12 Surrogate (Cambridge-EC1410-3) and 1.0 ml of Lindane13C6 (Cambridge-CL1282-S) to 50.0 ml hexane using volumetric glassware. For spiking instructions refer to Section 10.

8.0 Sample Collection, Preservation, Shipment and Storage

Sample container, preservation techniques and holding times may vary and are dependent on sample matrix, method of choice, regulatory compliance, and/or specific contract or client requests. Listed below are the holding times and the references that include preservation requirements.

Matrix	Sample Container	Min. Sample Size	Preservation	Holding Time	Reference
Water	Amber Glass	250 ml (8 oz)	Cool 4 ± 2°C	7 days to extraction; 40 days to analysis	USEPA SW846
Water	Amber glass	1000 ml	Cool 4 ± 2°C	7 days to extraction; 40 days to analysis	USEPA Method 680

8.1. Extracts must be stored under refrigeration (Cool 4 ± 2°C) in the dark and analyzed within 40 days of extraction.

9.0 Quality Control

9.1 Sample QC - The following quality control samples are prepared with each batch of samples.

Quality Controls	Frequency	Control Limit
Method Blank (MB)	1 in 20 or fewer samples	< Rpt. Limit
Laboratory Control Sample (LCS)[1]	1 in 20 or fewer samples	Statistical Limits [4]
Matrix Spike (MS)[2]	1 in 20 or fewer samples	Statistical Limits [4]
MS Duplicate (MSD)[2]	1 in 20 or fewer samples	Statistical Limits [4]
Surrogates	every sample[3]	Statistical Limits [4]

[1] LCS Duplicate (LCD) is performed only when insufficient sample is available for the MS/MSD or when requested by the client/project/contract.
[2] The sample used for MS/MSD is randomly selected by the organic prep lab, unless specifically requested by a client.
[3] Analytical and QC samples (MB, LCS, MS/MSD)
[4] Statistical control limits are updated annually and are updated into TALS (LIMS).

9.1.1. Method Blanks are extracted with each sample batch on each day that samples are extracted. The analytical results for the method blank must fall below the reporting limit for each compound of interest. If a target compound is detected in the blank at a concentration higher than the reporting limit, first the extract is reanalyzed for confirmation. If results are still outside of limits the entire batch of samples extracted with the affected blank must be re-extracted and reanalyzed.

9.1.2. Laboratory Control Sample (LCS): A Laboratory Control Sample (LCS) or blank spike must be extracted and analyzed for with each batch of 20 environmental samples. The LCS data is used to ensure that the analytical system is in control. It is also used to assess performance if the MS/MSD recoveries fall outside of established limits. The recoveries of the LCS must fall within lab generated acceptance criteria. If the spiked sample recovery results fall outside the laboratory generated limits (refer to the current active TALS

method limit group database), the LCS recovery is evaluated. If LCS recovery is within limits the poor sample recovery results are attributed to matrix interference. If the LCS recovery results are outside QC limits, first the extract is reanalyzed and if it is still outside the limits the entire QC batch must be re-extracted and reanalyzed.

9.1.3. Matrix Spike/Matrix Spike Duplicate (MS/MSD): A Matrix Spike/Matrix Spike Duplicate (MS/MSD) pair is extracted and analyzed with every batch of 20 environmental samples. MS/MSD recoveries are evaluated against lab generated limits (refer to refer to the current active TALS method limit group database). If the MS/MSD recovery limits fall outside of lab limits the LCS recovery is evaluated and corrective action is taken as described in the applicable analytical SOP (see Section 15.0, References).

9.1.4. Surrogate Standards: All samples, blanks and QC samples are spiked with surrogate standard solution containing DCB and TCMS (see Section 7.3.6). The percent recovery of the surrogate standards is calculated and compared to lab generated limits (refer to refer to the current active TALS method limit group database). If the surrogate recovery limits are outside of acceptance limits the sample extract is reanalyzed to confirm. If the recoveries are still outside of limits the sample must be re-extracted and reanalyzed or the data flagged as “estimated concentration”.

10.0. Procedure

10.1. Rinse a 500 ml separatory funnels twice each with a 1:1 methylene chloride/acetone mix and once with methylene chloride. Drain the solvents before adding sample to the funnels.

10.2. Place a small amount of glass wool into a 100-mm funnel and fill with pre-baked sodium sulfate crystals. Rinse three times with methylene chloride. Also rinse the outside of the funnel stem three times with methylene chloride, as it is likely to come into contact with the extract. Allow time for all of the rinsate to drain out of the funnel into a waste container.

10.3. Write the lab sample id number for each sample on the separatory funnels using a red wax pencil.

10.4. Make up hangtags for each sample as follows and hang on the corresponding

PCBs	PST
Sample Number: xxx	Sample Number: xxx
Fraction-Matrix:xxx	Fraction-Matrix:xxx
Date of Extraction:xx/xx/xx	Date of Extraction:xx/xx/xx

10.5. Place a 100 ml funnel containing rinsed sodium sulfate crystals onto each flasks

10.6. Mark the fluid level on each sample bottle with a black magic marker.

10.7. Rinse out a 1000ml graduated cylinder two to three times with organic free reagent water (for TCLP/SPLP leachates or reduced volume extraction use a 250 ml graduated cylinder). Obtain 1000ml of organic free reagent water (100 ml for TCLP/SPLP leachates or 250ml for reduced volume extraction) from the Millipore filtering apparatus for each of the method blank and the LCS.

10.8. Measure the initial pH of each sample with wide range pH paper and record the observed value in TALS prep batch as 'Received/initial pH'. If the initial pH of the sample is not between 5-9 adjust the pH to within 5-9 using 1:1(v/v) sulfuric acid or 10N Sodium hydroxide, as appropriate. Record the final adjusted pH value in the TALS batch comment section. Lesser strengths of acid or base solution may be employed, provided that they do not results in a significant change (<1%) in the volume of sample.

10.9. Pour each sample (including the method blank and LCS water) into its corresponding separatory funnel.

10.10. Rinse the spiking syringes 8 to 10 times with Acetone.

10.11. **For methods 8081/8081**: Add 50 ul of Pesticide Surrogate Spike Mix (see Section 7.2) to each sample and QC sample. When spiking the samples, make sure to get all bubbles out of the syringe. In addition, hold the syringe just above the level of the liquid when adding the spike. Don't touch the tip of the syringe to the liquid or the side of the separatory funnel

10.12. **For method 680**: add 1.0 ml of the PCB Homologue Surrogate Spiking Standard (see Section 7.3.8) to each sample and QC sample. When spiking the samples, make sure to get all bubbles out of the syringe. In addition, hold the syringe just above the level of the liquid when adding the spike. Don't touch the tip of the syringe to the liquid or the side of the separatory funnel

10.13. Depending upon the fraction and requested target analytes (Refer to Section 7.3 for information of spiking solutions).

- **PCBs by 8082**: add 50 ul of the 20ug/ml PCB Spike solution (see Section 7.3.3) to each LCS and designated MS/MSD
- **Pesticides (total) and TCLP/SPLP Pesticides by 8081**: 50 ul of the 4 ug/ml Pest Spike solution (see Section 7.3.2) to each LCS and designated MS/MSD
- **Technical Chlordane by 8081:** 20 ul of the 100 ug/ml Technical Chlordane Spike mix (see Section 7.3.5) to each LCS and designated MS/MSD.
- **Toxaphene by 8081:** 20 ul of the 100 ug/ml Toxaphene Spike mix (see Section 7.3.6) to each LCS and designated MS/MSD.

- **PCB Homologues by 680:** 1ml of the 680 PCB Homologue Spiking Standard (see Section 7.3.4) to each LSC and designated MS/MSD).

When spiking the samples, make sure to get all bubbles out of the syringe. In addition, hold the syringe just above the level of the liquid when adding the spike. Don't touch the tip of the syringe to the liquid or the side of the separatory funnel. Add 15 ml of methylene chloride for the reduced volume extraction option.

10.14. Add 15 ml of methylene chloride to each sample bottle. Swirl the solvent in the bottle and add the 15 ml for of methylene chloride to its corresponding separatory funnel.

10.15. Secure the separatory funnels to the rotators. Start the rotators.

10.16. Stop the rotator and vent the funnels after about 10 seconds. Resume rotating for 2 minutes.

10.17. After the rotator stops let the sample settle.

10.18. Drain bottom layer (organic layer) of the sample into the funnel/Concentrator apparatus.

10.19. Repeat steps 10.12 through 10.16 twice, now adding the methylene chloride directly to separatory funnel, rather than rinsing the sample container as in 10.12.

10.20. If an emulsion forms during the extraction, rinse a centrifuge tube well with methylene chloride and then drain the lower layer from the large separatory funnel into the tube. Centrifuge for 3 to 5 minutes at 2000 rpm. Make sure the levels (and weights) of the samples directly opposite one another in the centrifuge are approximately equal. After the centrifuging process is finished, there will be two layers: water on top and the methylene chloride with the desired sample extract on the bottom. A 1-ml disposable pipette should be used to transfer the bottom layer from the centrifuge tube to the appropriate concentration tube. With this method, care must be taken not to transfer any of the top layer. The top layer that remains is poured back into the separatory funnel with the rest of the original sample.

10.21. Find the empty sample bottles previously marked with a black marker with tap water up to the black line. Pour this into the graduated cylinder used for measuring sample volumes. Record each sample volume into the TALs extraction batch database.

10.22. Rinse funnel/sodium sulfate attached to concentration tube, twice with about 20ml of methylene chloride.

10.23. Sample can be concentrated between 30^{o}C-40^{o}C in the N-Vap to 2 ml final volume in the methylene chloride

10.24. Add about 10ml of Hexane to extract to exchange

10.25. Concentrate the hexane extract to 1 ml on the steam bath (temperature set between 30°C-40°C) using the N-EVAP apparatus.

10.26. The extract is now ready for the appropriate clean up.

10.27. If an emulsion forms during extraction, rinse a centrifuge tube well with Methylene Chloride and then drain the lower layer from the large separatory funnel into the tube. Centrifuge for 3 to 5 minutes at 2000 rpm. Make sure the levels (and weights) of the samples directly opposite one another in the centrifuge are approximately equal. After the centrifuging process is finished, there will be two layers: water on top and the Methylene Chloride with the desired sample extract on the bottom. A 1-ml disposable pipette should be used to transfer the bottom layer from the centrifuge tube to the appropriate concentration tube. With this method, care must be taken not to transfer any of the top layer. The top layer that remains is poured back into the 2000-ml separatory funnel with the rest of the original sample.

10.28. Required Documentation:

The organic prep technician is responsible for completing the following items.

10.28.1. The TALS reagent database must be completed in full with all required information whenever standards are logged and/or prepared.

10.28.2. All required data types (sample volumes, reagent and standard volumes, reagent and standard lots, spike witness, etc…) must be entered into the TALS batch database each time an extraction is performed. The department manager is responsible for ensuring that the data is reviewed for completeness and accuracy.

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11.0. Calculations / Data Reduction

None

12.0. Method Performance

12.1. Method Detection Limit Study (MDL)

The method detection limit (MDL) is the lowest concentration that can be detected for a given analytical method and sample matrix with 99% confidence that the analyte is present. The MDL is determined according to the laboratory's MDL procedure in Section 20 (Test Methods and Method Validation) of TestAmerica Edison's Quality Assurance Manual (ED-QA-LQM). MDLs reflect a

calculated (statistical) value determined under ideal laboratory conditions in a clean matrix, and may not be achievable in all environmental matrices. The laboratory maintains MDL studies for analyses performed; these are verified at least annually unless method requirements require a greater frequency.

12.2. Demonstration of Capabilities

For DOC procedure refer to Section 19 in the most current revision of TestAmerica Edison's Quality Assurance Manual (ED-QA-LQM).

12.3. Training Requirements

Refer to the current revision of TestAmerica Edison SOP No. ED-GEN-022, *Training*, for the laboratory's training program.

13.0. Pollution Control

13.1. Pollution prevention encompasses any technique that reduces or eliminates the quantity or toxicity of waste at the point of generation. Numerous opportunities for pollution prevention exist in laboratory operation. The USEPA has established a prevention hierarchy of environmental management techniques that places pollution prevention as the management option of first choice. Whenever feasible, laboratory personnel should use pollution prevention techniques to address their waste generation. When wastes cannot be feasibly reduced at the source, the agency recommends recycling as the next best option.

13.2. The quantity of chemical purchased should be based on expected usage during its shelf life and disposal cost of unused material. Actual reagent preparation volumes should reflect anticipated usage

14.0. Waste Management

14.1. The U.S. Environmental Protection Agency requires that laboratory waste management practices conducted be consistent with all applicable rules and regulations. All waste will be disposed of in accordance with Federal, State and Local regulations. Where reasonably feasible, technological changes have been implemented to minimize the potential for pollution of the environment. Employees will abide by this method and the policies in section 13 of the Corporate Safety Manual for "Waste Management and Pollution Prevention."

14.2. The following waste streams are generated as a result of this analysis:

- Extractions Waste water. This material is created when 50% Acetone and 50% Methylene Chloride are added to 1 liter of sample water. The water is shaken with the solvent. The solvent is collected with the compounds of interest and the water is discarded into the Extractions Waste Water drum. This drum is removed to the walk-in hood in the waste room. A ½

inch PVC pipe is inserted into the bung hole of the drum and air is passed through the solution over night. The solution is then transferred into the first drum of the neutralization system and neutralized to a pH of 6 – 9. This solution is discharged into the municipal sewer system.

- Mixed Solvent Waste. This material is collected from rinsing and other processes into 5 gallon solvent cans as satellite accumulation. These cans are emptied every 24 hours into a steel drum in the waste room. This drum is kept in the walk in hood until it is full. The full drum is then removed from the hood and placed on secondary containment in the waste room.
  Teris Profile Number: 50016624
  Onyx Profile WIP Number: 545240

- Waste sodium sulfate. This material is collected from various methods which require the removal of water from solvent which carries the analyte (s) of interest. The solvent is passed through the sodium sulfate and the sodium sulfate plus the water is disposed of. The sodium sulfate is collected in buckets inside the hoods. The material is air dried and disposed of in the municipal waste dumpster.

- Waste sulfuric acid. This material is generated from clean up of PCB extracts for sulfur compounds. The acid is collected in satellite accumulation in the hood. The container is removed to the waste room for neutralization with 50 % sodium hydroxide (Siedler Chemical SC-1824-03), water and sodium bicarbonate (Siedler Chemical SC-0219-25). Ice is used to control temperature in the plastic drums of the neutralization system. When neutralization is complete (pH 6 -9) the material is transferred to the municipal sewer system.

15.0. References / Cross-References

15.1. United States Environmental Protection Agency, “Method 3510C: Separatory Funnel Liquid-Liquid Extraction”, Test Methods for Evaluating Solid Wastes, SW846 Third Edition, Volume 1B: Laboratory Manual, Physical/Chemical Methods, Revision 3, December 1996.

15.2. *EPA Method 680: Determination of Pesticides and PCBs in Water and Soils/Sediment by Gas Chromatography/Mass Spectrometry*. Physical and Chemical Methods Branch, Environmental Monitoring and Support Laboratory, Office of Research and Development, USEPA, Cincinnati, OH, November 1985.

15.3. TestAmerica Edison SOP No. ED-GCS-016, *Analysis of Organochlorine Pesticides by SW846 Method 8081B,* current revision.

15.4. TestAmerica Edison SOP No. ED-GCS-017, *Analysis of Polychlorinated Biphenyls by SW846 Method 8082A,* current revision.

15.5. TestAmerica Edison SOP No. ED-MSS-010, *Determination of PCBs in Water and Soil/Sediment by GC/MS ,EPA Method 680*, current revision.

15.6. TestAmerica Edison Document No. ED-QA-LQM, *Laboratory Quality Manual*, current revision.

15.7. TestAmerica Corporate Document No. CW-E-M-001, *Environmental Health and Safety Manual,* current revision.

15.8. TestAmerica Corporate Quality SOP No. CA-Q-S-001, *Solvent & Acid Lot Testing & Approval*, current revision.

15.9. TestAmerica Edison SOP No. ED-GEN-023, *Bulk Solvent Testing and Approval*, current revision.

15.10. TestAmerica Edison SOP No. ED-GEN-013, *Glassware Cleaning*, most current revision

15.11. TestAmerica Edison SOP No. ED-GEN-022, *Training,* current revision.

16.0. Method Modifications:

NONE

17.0. Attachments

NONE

18.0. Revision History

- Revison 13, dated Jun 27, 2019:
    - Sections 1 and 15 (and throughout document as applicable): Deleted references to obsolete methods SW8082 and SW8081A as well as references to associated SOPs.
    - Section 2.2: revised wording to clarify that the RVE/LVI technique is the standard procedure.
    - Throughout document: removed references to equipment no longer used in the procedure: Synder columns, Kuderna-Danish concentrators, Erlenmyer flasks.

- Revision 12, dated May 16, 2017:
    - Updated throughout to include aqueous preps for EPA method 680.
- Revision 11, dated Dec 13, 2016:
    - Section 6.2: added pH paper to list of supplies.
    - Section 10.8: added instruction for measuring, adjusting and documenting sample pH values.
- Revison 10, dated Jun 15, 2015:
    - Section 2.1 and throughout document: Revised standard initial volume for Reduced Volume Extraction (RVE) to 250 ml.
    - Section 7.2: Tables 1 and 2 updated with new Restek standards.
    - Sections 7.3: updated spiking standards preparation instructions as necessitated by switch to Restek standards.
    - Section 8.0: updated minimum volume to 250ml.
- Revison 9, dated May 3, 2013:
    - Section 7.2.2, Table 1: corrected concentration of the Chlordane (technical) standard solution to 1000 ug/ml. Deleted 'TCLP Pest Mix 1' and 'TCLP Pest Mix 2'. Added Toxaphene (Supleco 48103-U).
    - Section 7.2.2, Table 2: corrected concentration of the Chlordane (technical) standard solution to 1000 ug/ml. Deleted all TCLP Pest Spike standards. Added Toxaphene (Supleco 48103-U).
    - Deleted Section 7.3.4, TCLP Pesticide Spiking Standard (LSC/MS/MSD). Remaining sections renumbered accordingly.
    - Section 10.11. Deleted last bullet (TCLP Pesticides). Added '…and TCLP/SPLP Pesticides' to the second bullet (Pesticides). Added '(or 20 ul of the 100ug/ml mix for reduced volume extraction)' to the Toxaphene bullet.
- Revision 8, dated February 26, 2013:
    - Throughout document: updated Lab Quality Manual section number references as required.
    - Section 1.2: added references to all applicable analytical SOPs.
    - Section 2.0: added Section 2.1 which describes option for Reduced Volume Extraction.
    - Section 6.2: added a 500 ml separatory funnel.
    - Section 7.0: updated throughout to include additional standards preps for reduced volume extraction.
    - Section 8.0: updated to include smaller sample container for the reduced volume extraction option.
    - Section 10.0: updated to include spiking and concentration instructions for the reduced volume extraction option.

- Revision 7, dated October 31, 2011

    - Section 7.0: added 'Reagent Water' and details concerning it's preparation to the list of reagents.

- Revision 6, dated October 30, 2009

    - Revised SOP format in accordance with TestAmerica Corporate Quality SOP No. CW-QS-002 (Rev 0) Writing a Standard Operating Procedure (SOP).
    - Section 1.1 Added reference to Quality Assurance Manual for method modifications.
    - Section 1.1: Expanded to include references to applicable analytical SOPs.
    - Section 3: revised to reference new location for definitions.
    - Section 5: Revised to include most up to date corporate health and safety references and information.
    - Section 7.2.2: Added tables detailing components found in the various spiking standards mixes.
    - Section 7.3: Updated the instructions for preparation of spiking standards.
    - Section 7.3: Added tables with spiking standards prep details.
    - Section 8: Updated with additional details including a table outlining containers, preservation and holding times.
    - Section 9.1: Expanded QC sample preparation details.
    - References: Expanded to include more specific SOP references
    - Section 18: Added this Revision History section
    - Throughout document: added references to TestAmerica LIMS (TALS).
    - Throughout document: deleted references to organophosphorus pesticides as the Edison lab no longer analyzes for these.

Title: Extraction of Organochlorine Herbicides in Water by SW846 Method 8151A

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Approvals (Signature/Date):

Mamdou Leye Organic Prep Department Manager	10/5/21 Date	Dan Helfrich Health & Safety Manager / Coordinator	10/5/21 Date
Carl Armbruster Quality Assurance Manager	10/5/21 Date	Mark Acierno Laboratory Director	10/5/21 Date
Sylvanus Klusey Operations Manager - Organics	10/5/21 Date		

Facility Distribution No. Electronic **Distributed To: Facility Intranet**

1.0 Scope and Application

1.1. **Analytes, Matrix(s), and Reporting Limits:** SW846 Method 8151A provides extraction and derivatization procedures for the gas chromatographic analysis of chlorinated acid herbicides in water, soil, and waste samples. An option for the hydrolysis of esters is also described. This SOP details the extraction of water samples with diethyl ether follower by esterification with diazomethane. The derivatives are determined by gas chromatography with an electron capture detector (GC/ECD). The results are reported as acid equivalents.

1.2. For a complete discussion of analytes and reporting limits (RLs) please refer to TestAmerica Edison SOP No. ED-GCS-00, *Analysis of Organochlorine Herbicides by SW846 Method 8151A,* current revision.

1.3. On occasion clients may request modifications to this SOP. These modifications are handled following the procedures outlined in Section 7 (*Review of Work*) and 19 (*Test Methods and Method Validation*) of TestAmerica Edison's Quality Assurance Manual (ED-QA-LQM).

2.0 Summary of Method

This SOP provides extraction and derivatization procedures for the determination of chlorinated acid herbicides and their esters in water and aqueous waste samples.

Water samples are hydrolyzed, cleaned with methylene chloride, acidified, extracted with diethyl ether and then esterified with Trimethylsilyl)diazomethane. The derivatives are determined by gas chromatography with an electron capture detector (GC/ECD).

The sensitivity of Method 8151A depends on the level of interferences in addition to instrumental limitations.

3.0 Definitions

For a complete list of definitions refer to Appendix 5 in the most current revision of TestAmerica Edison's Quality Assurance Manual (ED-QA-LQM).

4.0 Interferences

Method interference may be caused by contaminants in solvents, reagents, glassware and other sample processing hardware that lead to discrete artifacts or elevated baselines in gas chromatograms. All these materials must be routinely demonstrated to be free from interference under the conditions of the analysis, by analyzing reagent blanks.

Matrix interference may be caused by contaminants that are co-extracted from the sample. Organic acids, especially chlorinated acids, cause the most direct

interference with the determination by methylation. Phenols, including chlorophenols, may also interfere with this procedure.

The acid forms of the analytes are strong organic acids that react readily with alkaline substances and can be lost during sample preparation. Glassware and glass wool must be acid-rinsed with 1 N hydrochloric acid and the sodium sulfate must be acidified with sulfuric acid prior to use to avoid analyte losses due to adsorption.

Sample extracts must be dry prior to methylation or else poor recoveries will result.

5.0 Safety

Employees must abide by the policies and procedures in the Corporate Environmental Health and Safety Manual (CW-E-M-001) and this document. This procedure may involve hazardous material, operations and equipment. This SOP does not purport to address all of the safety problems associated with its use. It is the responsibility of the user of the method to follow appropriate safety, waste disposal and health practices under the assumption that all samples and reagents are potentially hazardous. Safety glasses, gloves, lab coats and closed-toe, nonabsorbent shoes are a minimum.

5.1. Specific Safety Concerns or Requirements

Ethyl Ether is an extremely flammable solvent. If a mechanical device is used for sample extraction, the device should be equipped with an explosion proof motor and placed in a hood.

Ethyl Ether must be free of peroxides as determined by use of EM Quant Peroxide Test Strips as described in Sections 6 and 7 below.

Diazomethane can explode under certain conditions therefore take the following precautions:

- Use a well-ventilated hood.
- Use a safety screen.
- Do not heat above 90°C.
- Avoid grinding surfaces, ground glass joints, sleeve bearings, and glass stoppers.
- Store away from alkali metals.
- Solutions of diazomethane decompose rapidly in the presence of solid materials such as copper powder, calcium chloride, and boiling chips.

The use of Kevlar gloves is required for the assembly/disassembly of ground glass joints in addition to those tasks that present the potential risk for injury.

Ether creates excessive pressure very rapidly. Therefore, initial venting should be done immediately after the separatory funnel has been sealed and inverted. Vent into hood away from analysts and other samples.

5.2. Primary Materials Used

The following is a list of the materials used in this method, which have a serious or significant hazard rating. **Note: This list does not include all materials used in the method. The table contains a summary of the primary hazards listed in the MSDS for each of the materials listed in the table.** A complete list of materials used in the method can be found in the reagents and materials section. Employees must review the information in the MSDS for each material before using it for the first time or when there are major changes to the MSDS.

Material (1)	Hazards	Exposure Limit (2)	Signs and symptoms of exposure
Methylene Chloride	Carcinogen Irritant	25 ppm-TWA 125 ppm-STEL	Causes irritation to respiratory tract. Has a strong narcotic effect with symptoms of mental confusion, light-headedness, fatigue, nausea, vomiting and headache. Causes irritation, redness and pain to the skin and eyes. Prolonged contact can cause burns. Liquid degreases the skin. May be absorbed through skin.
Sodium Hydroxide	Corrosive Poison	2 ppm, 5 mg/m^3	This material will cause burns if comes into contact with the skin or eyes. Inhalation of Sodium Hydroxide dust will cause irritation of the nasal and respiratory system.
Sulfuric Acid	Corrosive Oxidizer Dehydrator Poison Carcinogen	1 Mg/M3-TWA	Inhalation produces damaging effects on the mucous membranes and upper respiratory tract. Symptoms may include irritation of the nose and throat, and labored breathing. Symptoms of redness, pain, and severe burn can occur. Contact can cause blurred vision, redness, pain and severe tissue burns. Can cause blindness.
Acetone	Flammable	1000 ppm-TWA	Inhalation of vapors irritates the respiratory tract. May cause coughing, dizziness, dullness, and headache.
Ethyl Ether	Flammable Irritant Peroxide Former	400 ppm-TWA	General anesthesia by inhalation can occur. Continued exposure may lead to respiratory failure or death. Early symptoms include irritation of nose and throat, vomiting, and irregular respiration, followed by dizziness, drowsiness, and unconsciousness. May cause irritation, redness and pain to the eyes. Irritating to the skin and mucous membranes by drying effect. Can cause dermatitis on prolonged exposure. May be absorbed through skin. **May form explosive peroxides on long standing or after exposure to air or light. This material must be disposed of within six months.**

Material (1)	Hazards	Exposure Limit (2)	Signs and symptoms of exposure
Hexane	Flammable Irritant	500 ppm-TWA	Inhalation of vapors irritates the respiratory tract. Overexposure may cause lightheadedness, nausea, headache, and blurred vision. Vapors may cause irritation to the skin and eyes.
1 – Always add acid to water to prevent violent reactions.			
2 – Exposure limit refers to the OSHA regulatory exposure limit.			

6.0 Equipment and Supplies

6.1. Equipment

- Top loading Balance - Sargent Welch S2642-25 or equivalent
- Automatic separatory funnel rotator
- Steam Bath - Fisher Scientific 66738 or equivalent.
- Furnace - Thermolyne Type 6000 or equivalent.
- Vacuum Pump - General Electric Model No. 0322-V48-G180X or equivalent.
- N-Evap - Meyer Analytical Evaporator Model No. 112 or equivalent

6.2. Supplies

- 400 ml Clear Glass Jar
- 1 L Erlenmeyer Flask
- 500 ml Erlenmeyer Flask
- 2 L Separatory Funnel
- 500 ml Separatory Funnel
- 500 ml Centrifuge Bottle - Pyrex 1260 or equivalent.
- 10 ml graduated Concentrator Tube - Kontes K570050-1025 or equivalent.
- 10 ml glass vials with Teflon lined screw caps.
- 20 ml glass vials with Teflon lined screw caps.
- 5 ml to 1000 ml Class A Volumetric flasks
- Funnel - 75 mm diameter

- 3 Ball Macro Snyder Column - Baxter K503000-0121 or equivalent.
- 1 L Graduated Cylinder
- 50 ul Gastight Syringe - VWR 60376-241 or equivalent.
- 100 ul Gastight Syringe - Baxter S9662-5 or equivalent.
- 100 ml Graduated Cylinder
- 1 ml Pasteur Disposable Pipet
- Kuderna Danish (K-D) - Kontes 500 ml - Baxter K570001-0500 or equivalent.
- 10 ml Disposable Pipette
- Glass stopper - size 24/40
- Teflon stopper - size 19/22
- Filter paper - 15-cm diameter, Whatman No. 1 or equivalent.
- Phosphoric Acid Washed Glass Wool - Supelco 2-0383 or equivalent.
- Desiccator - Baxter D1420-2 or equivalent.
- Septa, 12 mm - Supelco 2-3273 or equivalent.
- Septa, 8 mm - Supelco 2-3272 or equivalent.
- Standard Taper Clamp (size 19, blue) - Wheaton 297749 or equivalent.
- Silicon Carbide Boiling Stones - Thomas 1590-D33 or equivalent.
- pH paper, Fisher 9590
- EM Quant Peroxide Test Strips, EMD No. 10011-1 or equivalent

Note: All glassware must be pre-rinsed with a 25% Sulfuric Acid: reagent water solution.

7.0 Reagents and Standards

7.1 Reagents

Note: Each lot of Methylene Chloride, Acetone, Methanol and Sulfuric Acid is screened for contaminants before being used for analysis as detailed in TestAmerica Corporate Quality SOP No. CA-Q-S-001 (*Solvent & Acid Lot Testing & Approval*) and TestAmerica Edison SOP No. ED-GEN-023 (*Bulk Solvent Testing and Approval*).

- Organic-free reagent water.
- Trimethylsilyldiazomethane (TMSD) , 2.0 molar solution in hexane, Acros 429211000 or equivalent
- Diethyl Ether (Residue Analysis Grade with 2% Ethanol preservative) - Baker 9259-03 or equivalent (tested for peroxide formation upon opening and monthly thereafter by use of EM Quant Peroxide Test Strips)
- Methylene Chloride (High Purity Grade) - Baker 9266 or equivalent.
- Acetone (High Purity Grade) – Baker 9254 or equivalent.
- Sodium Sulfate Anhydrous Crystals (Reagent Grade) – Malinckrodt 8024 or equivalent. (Baked at 400°C for 4 hours)
- Hexane (High Purity Grade) – Baker 9262 or equivalent.
- Carbitol (diethylene glycol monoethyl ether)
- Potassium Hydroxide (Analytical Reagent Grade) – Aldrich 306568 or equivalent
- Sulfuric Acid (Analytical Reagent Grade) - Mallinckrodt 2876 or equivalent.
- Silicic Acid, Aldrich 30, 636-3 or equivalent

7.1.1 Reagent preparation

7.1.1.1 Anhydrous sodium sulfate crystals must be baked in the muffle furnace at 400° C for 4 hours prior to use. After baking, the sodium sulfate must be acidified. Place 500 grams of anhydrous sodium sulfate into a 500 ml wide mouth Erlenmeyer flask. Add enough diethyl ether to cover the sodium sulfate. Add 0.5 ml concentrated sulfuric acid. Shake vigorously for 1 minute. Evaporate the ether by placing the flask on steam bath. Mix 1 g of the resulting solid with 5 ml of organic-free reagent water and measure the pH of the mixture. It must be below a pH of 4. Prepare fresh daily.

7.1.1.2 37% KOH Solution: Rinse a 1 liter Pyrex flask three times with organic free reagent water. Weigh 148 g KOH into flask and add 400 ml organic free reagent water. Mix until the KOH is completely dissolved and the flask has cooled to room temperature. Transfer the KOH solution to a clean 500-ml amber bottle and label.

7.1.1.3 12N H_2SO_4 : Rinse a 1 liter Pyrex flask three times with organic free reagent water. Add 200 ml organic free reagent water to the flask. Slowly, and while mixing, add 100-ml concentrated H_2SO_4 to flask. Continue mixing until flask has cooled to room temperature. Transfer the H_2SO_4 solution to a clean 500-ml amber bottle and label.

7.1.1.4 1:1 H_2SO_4 : Rinse a 1 liter Pyrex flask three times with organic free reagent water. Add 100 ml organic free reagent water to the flask. Slowly, and while mixing, add 100-ml concentrated H_2SO_4 to flask. Continue mixing until flask has cooled to room temperature. Transfer the H_2SO_4 solution to a clean 500-ml amber bottle and label.

7.1.1.5 25% H_2SO_4 solution (1:3 H_2SO_4 solution): 25 ml Acid and 75 ml reagent water. Add 75 ml organic free reagent water to a graduated cylinder. Slowly, and while mixing, add 25-ml concentrated H_2SO_4 to cylinder. Continue mixing until cylinder has cooled to room temperature. Transfer the H_2SO_4 solution to a clean amber bottle and label.

7.2 Standards

7.2.1 Standards are purchased as concentrated solutions (see Section 7.2.2). The surrogate stock solution is diluted (in volumetric glassware) to working concentration using acetone as the diluent as described in Section 7.3.

7.2.2 **Standard mixes and sources:** Table 1 lists the standard concentrate mix sources. Attachment 1 details the individual components of these mixes.

Table 1:
Herbicide Standard Mixes and Sources*

Standard Name ("Lab Name")	Concentration (ug/ml)	Source
Acid Herbicide LCS RTS ("Spiking Mix")	varies (in Methanol)	Restek Catalog # 567950
DCAA standard (Surrogate Spiking Solution)	1000 (in methanol)	Restek Catalog #567804

*May be substituted with equivalent standards from alternate sources.

Table 2:
Components of Herbicide Standard Mixes

Parameter	Catalog Nos.	Lab Standard Name	Concentration (ug/ml)
2,4-dichlorophenoxyacetic acid (2,4-D)	Restek Catalog # 567950	Spiking Mix	20
2-(2,4,5-Trichlorophenoxy)propionic acid (2,4,5-TP aka Silvex)	Restek Catalog # 567950	Spiking Mix	5
2,4,5-T	Restek Catalog # 567950	Spiking Mix	5
2,4-DB	Restek Catalog # 567950	Spiking Mix	20
Dalapon	Restek Catalog # 567950	Spiking Mix	20

Table 2: Components of Herbicide Standard Mixes			
Parameter	**Catalog Nos.**	**Lab Standard Name**	**Concentration (ug/ml)**
Dicamba	Restek Catalog # 567950	Spiking Mix	10
Dichloroprop	Restek Catalog # 567950	Spiking Mix	20
Dinoseb	Restek Catalog # 567950	Spiking Mix	20
MCPA acid	Restek Catalog # 567950	Spiking Mix	2000
MCPP acid	Restek Catalog # 567950	Spiking Mix	2000
Pentachlorophenol	Restek Catalog # 567950	Spiking Mix	5
Picloram (4-amino-3,5,6-trichloropicolinic acid)	Restek Catalog # 567950	Spiking Mix	20
2,4-Dichlorophenylacetic Acid	Restek Catalog # 567804	Surrogate Spiking Solution	1000

7. 3. Standards Preparation

7.3.1. The surrogate stock solutions are diluted to the working concentrations with acetone as indicated in Attachment1 and 2.

7.3.2. Herbicide Spiking Standard (LCS/MS/MSD):

7.3.2.1 Herbicide QC Spiking Standard Mix (12 compound analysis): The 12 compound spiking standard is ready to spike and used when analyzing for the standard list of herbicides The spiking standard mix is a 25 ml solution (Restek Catalog # 567950; see Tables 1 and 2) of the 12 compounds at the specified concentrations as free acids. The concentrate is used without further dilution prior to the spiking of the following quality control sample types: MS, MSD, and LCS. Spiking instructions are found in Section 10.

7.3.3. Herbicide Surrogate Spiking Standard (2,4-DCAA Acid Spike Mix**):** The herbicide surrogate spiking standard is prepared as detailed in Attachment 1 using volumetric glassware and the concentrated free acid herbicide standards listed in Table 1 above.

8.0 Sample Collection, Preservation, Shipment and Storage

Sample container, preservation techniques and holding times may vary and are dependent on sample matrix, method of choice, regulatory compliance, and/or specific contract or client requests. Listed below are the holding times and the references that include preservation requirements.

Matrix	Sample Container	Min. Sample Size	Preservation	Holding Time	Reference
Waters	Glass	1L amber/ 8oz amber	Cool 4 ± 2°C	7 days to extraction; 40 days to analysis	USEPA SW846

8.1. Extracts must be stored under refrigeration (Cool 4 ± 2°C) in the dark and analyzed within 40 days of extraction.

9.0 Quality Control

9.1 Sample QC - The following quality control samples are prepared with each batch of samples.

Quality Controls	Frequency	Control Limit
Method Blank (MB)	1 in 20 or fewer samples	< Rpt. Limit
Laboratory Control Sample (LCS)[1]	1 in 20 or fewer samples	Statistical Limits [4]
Matrix Spike (MS)[2]	1 in 20 or fewer samples	Statistical Limits [4]
MS Duplicate (MSD)[2]	1 in 20 or fewer samples	Statistical Limits [4]
Surrogates	every sample[3]	Statistical Limits [4]

[1] LCS Duplicate (LCD) is performed only when insufficient sample is available for the MS/MSD or when requested by the client/project/contract.
[2] The sample selection for MS/MSD is randomly selected by the organic prep lab, unless specifically requested by a client.
[3] Analytical and QC samples (MB, LCS, MS/MSD)
[4] Statistical control limits are updated annually and are updated into TALS (LIMS).

9.1.1. Method Blanks are extracted with each sample batch on each day that samples are extracted. The analytical results for the method blank must fall below the reporting limit for each compound of interest. If a target compound is detected in the blank at a concentration higher than the reporting limit, first the extract is reanalyzed for confirmation. If results are still outside of limits the entire batch of samples extracted with the affected blank must be re-extracted and reanalyzed.

9.1.2. Laboratory Control Sample (LCS): A Laboratory Control Sample (LCS) or blank spike must be extracted and analyzed for with each batch of 20 environmental samples. The LCS data is used to ensure that the analytical system is in control. It is also used to assess performance if the MS/MSD recoveries fall outside of established limits. The recoveries of the LCS must fall within lab generated

acceptance criteria. If the spiked sample recovery results fall outside the laboratory generated limits (refer to the current active TALS method limit group database), the LCS recovery is evaluated. If LCS recovery is within limits the poor sample recovery results are attributed to matrix interference. If the LCS recovery results are outside QC limits, first the extract is reanalyzed and if it is still outside the limits the entire QC batch must be re-extracted and reanalyzed.

9.1.3. Matrix Spike/Matrix Spike Duplicate (MS/MSD): A Matrix Spike/Matrix Spike Duplicate (MS/MSD) pair is extracted and analyzed with every batch of 20 environmental samples. MS/MSD recoveries are evaluated against lab generated limits (refer to refer to the current active TALS method limit group database). If the MS/MSD recovery limits fall outside of lab limits the LCS recovery is evaluated and corrective action is taken as described in TestAmerica Edison SOP No. ED-GCS-00, *Analysis of Organochlorine Herbicides by SW846 Method 8151A,* current revision.

9.1.4. Surrogate Standards: All samples, blanks and QC samples are spiked with surrogate standard solution containing 2,4-DCAA (see Section 7.3.3). The percent recovery of the surrogate standards is calculated and compared to lab generated limits (refer to refer to the current active TALS method limit group database). If the surrogate recovery limits are outside of acceptance limits the sample extract is reanalyzed to confirm. If the recoveries are still outside of limits the sample must be re-extracted and reanalyzed or the data flagged as "estimated concentration".

10.0. Procedure

10.1. Sample Extraction (Aqueous Samples)

Note: For TCLP or SPLP leachate extraction procedure proceed to Section 10.6

10.1.1. Rinse enough 2-liter/500ml separatory funnels and 500-ml Erlenmeyer flasks to accommodate the number of samples to be prepped. Each funnel and flask must be rinsed four times: once with a 50:50 mixture of methylene chloride:acetone, twice with methylene chloride and once with a 25% sulfuric acid: reagent water solution.

10.1.2. Mark the lab sample identification numbers on the corresponding separatory funnels with red wax pencil.

10.1.3. Make up tags for each sample with the following information and place on the corresponding 500 ml Erlenmeyer flasks.

HERBICIDES
Sample Number
Matrix
Date of Extraction

10.1.4. Mark the fluid level of sample bottles (volume) with a black magic marker or red wax pencil. Pour each sample into its corresponding separatory funnel. Fill each sample bottle to the black line with tap water. Pour this into the graduated cylinder used for measuring sample volumes. Note the volume for each sample on the Organic Extraction Data Sheet or enter directly into the TALs batch record.

10.1.5. Rinse a 1000 ml graduated cylinder once with methylene chloride and two to three times with lab reagent water. Using the graduated cylinder obtain 1000 ml of lab reagent water from the Millipore filtering apparatus for each of the method blank and the laboratory control sample (LCS) (aka blank spike).

10.1.6. Pour the reagent water for the method blank and LCS into the corresponding, labeled separatory funnels.

10.1.7. Rinse each syringe to be used for adding surrogates and spikes, eight to ten times with acetone.

10.1.8. Add 50 ul of 100 ppm 2,4-Dichlorophenylacetic Acid (DCAA) Surrogate Spiking Solution (see Attachment 2) to each sample and QC sample. NOTE: when spiking the samples with either surrogates or target spiking compounds, ensure that there are no bubbles in the syringe. In addition, hold the syringe just above the level of the water when adding the spike. Do not touch the tip of the syringe to the water or to the side of the separatory funnel. Swirl the separatory funnel after adding surrogate spike. All spikes must be witnessed.

10.1.9. When preparing QC spike samples (MS/MSD/LCS), spike 500ul of 12 compound mix (see Tables 1 and 2).

10.1.10. Add 2 ml of 1:1 Sulfuric Acid/ reagent water to each sample and QC sample. Shake to mix. Check pH with pH paper. The pH must be ≤ 2. Add sulfuric acid as needed to obtain proper pH. Note: pH adjustments must be documented in the extraction log and/or in the TALS batch record.

10.1.11. Add 150 ml of diethyl ether to each sample bottle. Rinse the bottle thoroughly with the ether and transfer the ether to its corresponding separatory funnel.

10.1.12. Extract the samples by vigorously shaking the separatory funnels for 2 minutes, with periodic venting to release excess pressure.

10.1.13. Allow the organic layer to separate from the water phase for a minimum of 10 minutes.

10.1.14. If the emulsion interface between the layers is more than one-third the volume of the solvent layer, the analyst must employ mechanical techniques to complete the phase separation. The optimum technique depends upon the sample, but may include stirring, filtration through glass wool, centrifugation or other physical methods.

10.1.15. Collect the diethyl ether layer for each sample into the corresponding 500-ml Erlenmeyer flask.

10.1.16. Repeat steps 10.11 through 10.15 twice, using 50-ml aliquots of diethyl ether each time.

10.1.17. Discard the aqueous phase.

10.2. Hydrolysis

10.2.1. To each 500-ml Erlenmeyer flask containing the 200-ml ether extract, add 2 ml of the 37% KOH solution, 15 ml of reagent water, and 4-5 boiling chips.

10.2.2. Attach a three-ball macro Snyder and place Erlenmeyer flask on steam bath (100ºC) for 1½ to 2 hours.

10.3. Partitioning

10.3.1. Remove the Erlenmeyer flask from the steam bath and allow it to cool for 5-10 minutes.

10.3.2. Pour the extract into a 500-ml separatory funnel pre-rinsed with diethyl ether.

10.3.3. Add 40 ml of diethyl ether to the separatory funnel and shake for two minutes.

10.3.4. Pour the dried diethyl ether extract through a funnel plugged with acid washed glass wool, collecting the extract in the K-D concentrator. Use a glass rod to crush any caked sodium sulfate during the transfer.

10.3.5. Repeat twice using 20-ml aliquots of diethyl ether.

10.3.6. Discard the ether phase.

10.3.7. Adjust the pH of the extract remaining in the 500-ml separatory funnel to ≤ 2 by adding 2 ml of 1:3 Sulfuric Acid. Check pH with pH paper.

10.3.8. Add 40 ml of diethyl ether to the separatory funnel and shake for 2 minutes with periodic venting to release pressure.

10.3.9. Collect each diethyl ether phase into a single pre-cleaned screw top glass jars each containing approximately 50 g of acidified sodium sulfate crystals and mix well.

10.3.10. Repeat sections10.3.8 and 10.3.9 twice using 20-ml aliquots of diethyl ether.

10.3.11. Allow the diethyl ether extract to remain in contact with the sodium sulfate for approximately 2 hours.

10.3.12. Pour the dried diethyl ether extract through a funnel plugged with acid washed glass wool, collecting the extract in the K-D concentrator. Use a glass rod to crush any caked sodium sulfate during the transfer.

10.3.13. Place the white identification tag on the K-D apparatus.

10.3.14. Rinse the Erlenmeyer flask and funnel with 20 to 30 ml of diethyl ether to complete the quantitative transfer.

10.4. Extract Concentration

10.4.1. Add one or two clean boiling stones to the K-D flask and attach a three-ball Snyder column.

10.4.2. Pre-wet the Snyder column by adding about 1 ml of diethyl ether to the top of the column.

10.4.3. Place the K-D apparatus on a hot water bath (15-20°C above the boiling point of the solvent; set at LOW which is 60ºC) such that the concentrator tube is partially immersed in the hot water and the entire lower rounded surface of the flask is bathed with hot vapor.

10.4.4. Adjust the vertical position of the apparatus and the water temperature, as required, to complete the concentration in 10-20 minutes.

10.4.5. Boil the sample in the K-D flask on the steam bath until the sample volume is approximately 1 ml.

10.4.6. Add 60 ml of hexane and concentrate further to a final volume of approximately 5 ml on a hot water bath (Set at MEDIUM which is approximately 78ºC).

10.4.7. Remove the K-D apparatus from the water bath and allow it to drain and cool for at least 10 minutes.

10.4.8. Remove the Snyder column from the top of the K-D flask. Remove the blue taper clamp from the ground glass joint and dry

the exterior with a Kimwipe. Remove the K-D flask from the concentration tube.

10.4.9. Place the concentration tube onto the N-Evap apparatus. Maintain I.D. tag with concentration

10.4.10. Continue to concentrate under a N2 stream until the sample volume is approximately 1 ml.

10.4.11. To each sample add 500ul of methanol and dilute to a final volume of 10 ml with hexane when the initial volume is 1L , to 3ml final volume when the initial volume is 250ml, and 5 ml final volume for TCLP extracts.

10.5. Esterification

10.5.1. Add approximately 200 uL of methanol to the extract followed by approximately 200 uL of 2.0M Trimethylsilyldiazomethane (TMSD) solution. The extract will turn a yellow color. If this does not occur, add additional (approximately) 200 uL aliquots of 2.0M Trimethylsilyldiazomethane (TMSD) solution until the yellow color persists. Check the sample every 15 minutes for yellow color. If the yellow disappears in this time frame, continue adding an additional aliquot of 2.0M Trimethylsilyldiazomethane (TMSD) solution until the yellow color persists.

10.5.2. Allow the extract to stand for at least one hour at room temperature to allow the methylation reaction to occur. The reaction is halted with the addition of approximately 0.05g (one scoop using a disposable spatula) of silica gel. The extract is then brought up to a final volume of 10 mL with hexane by visually comparing it to a calibrated collection tube.

10.5.3. Adjust the final extract volumes to 10ml with hexane. The extracts ready to be analyzed by GC. Transfer some volume to GC autosampler vials.

10.5.4. Samples must be analyzed as soon as possible (less than 24 hrs. preferably) after esterification. This is to minimize the trans-esterification and other potential reactions that may occur.

10.5.5. Store refrigerated if further processing will not be performed immediately. Extracts should be stored at 4°C away from light.

10.6. Sample Extraction (Leachate Samples)

10.6.1. Rinse enough 60-ml separatory funnels and 500-ml Erlenmeyer flasks to accommodate the number of samples to be prepped. Each funnel and flask must be rinsed four times: once with a 50:50 mixture of methylene chloride:acetone, twice with

methylene chloride and once with a 25% sulfuric acid: reagent water solution.

10.6.2. Mark the lab sample identification numbers on the corresponding separatory funnels with red wax pencil.

10.6.3. Make up tags for each sample with the following information and place on the corresponding 500 ml Erlenmeyer flasks.

HERBICIDES
Sample Number
Matrix
Date of Extraction

10.6.4. Pipette 15 ml of each sample into its corresponding separatory funnel.

10.6.5. Rinse a 100 ml graduated cylinder once with methylene chloride and two to three times with lab reagent water. Using the graduated cylinder obtain 15 ml of lab reagent water from the Millipore filtering apparatus for each of the method blank and the laboratory control sample (LCS) (aka blank spike).

10.6.6. Rinse each syringe to be used for adding surrogates and spikes, eight to ten times with acetone.

10.6.7. Add 50 ul of 100 ppm 2,4-Dichlorophenylacetic Acid (DCAA) Surrogate Spiking Solution (see Attachment 1) to each sample and QC sample. NOTE: when spiking the samples with either surrogates or target spiking compounds, ensure that there are no bubbles in the syringe. In addition, hold the syringe just above the level of the water when adding the spike. Do not touch the tip of the syringe to the water or to the side of the separatory funnel. Swirl the separatory funnel after adding surrogate spike. All spikes must be witnessed.

10.6.8. When preparing QC spike samples (MS/MSD/LCS), spike 500uL of 12 compound mix (see Tables1 and 2). .Swirl the separatory funnel after adding the spike solution(s).

10.6.9. Add 2 ml of 37% KOH solution to each sample.

10.6.10. Add 20 ml of diethyl ether.

10.6.11. Extract the samples by vigorously shaking the separatory funnels for 2 minutes, with periodic venting to release excess pressure.

10.6.12. Allow the organic layer to separate from the water phase for a minimum of 10 minutes.

10.6.13. If the emulsion interface between the layers is more than one-third the volume of the solvent layer, the analyst must employ mechanical techniques to complete the phase separation. The optimum technique depends upon the sample, but may include stirring, filtration through glass wool, centrifugation or other physical methods.

10.6.14. Collect the diethyl ether layer for each sample into the corresponding 250-ml Erlenmeyer flask.

10.6.15. Repeat steps 10.6.10 through 10.14 twice, using 10-ml aliquots of diethyl ether each time.

10.6.16. Discard the ether phase after each extraction.

10.6.17. Adjust the pH to $\leq$ 2 by adding approximately 2 ml of 1:3 Sulfuric Acid/Water to the samples. Check pH with pH paper.

10.6.18. Extract three times for two minutes each with ether, using 20 ml the first time, and 10 ml each the second and third times.

10.6.19. Collect all three ether phases into a screw top 125-ml glass jar containing acidified sodium sulfate. Let the sample dry for 2 hours.

10.6.20. Pour the dried extract through a funnel plugged with acid washed glass wool, and collect the extract into the K-D flask. Use a glass rod to crush any caked sodium sulfate during the transfer.

10.6.21. Place the white I.D. tag on the K-D apparatus.

10.6.22. Rinse the Erlenmeyer flask and funnel with 20 to 30 ml of diethyl ether to complete the quantitative transfer.

10.6.23. Proceed to Section 10.4.1.

10.7. Required Documentation:

The organic prep technician is responsible for completing the following items.

10.7.1. The TALS reagent database must be completed in full with the required information whenever standards are logged and/or prepared.

10.7.2. All required data types (sample volumes, reagent and standard volumes, reagent and standard lots, etc…) must be entered into the TALS batch database each time an extraction is performed.

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11.0. Calculations / Data Reduction

None

12.0. Method Performance

12.1. Method Detection Limit Study (MDL)

A Method Detection Limit (MDL) study, as described in the TestAmerica corporate Detection and Quantitation Limits SOP, CA-Q-S-006, must be performed initially and whenever a significant change affecting sensitivity is made to the analytical system. The MDL must be re-evaluated from quarterly MDL points at least every 12 months..

12.2. Demonstration of Capabilities

For DOC procedure refer to Section 19 in the most current revision of TestAmerica Edison's Quality Assurance Manual (ED-QA-LQM).

12.3. Training Requirements

Refer to the current revision of TestAmerica Edison SOP No. ED-GEN-022, *Training*, for the laboratory's training program.

13.0. Pollution Control

13.1. Pollution prevention encompasses any technique that reduces or eliminates the quantity or toxicity of waste at the point of generation. Numerous opportunities for pollution prevention exist in laboratory operation. The USEPA has established a prevention hierarchy of environmental management techniques that places pollution prevention as the management option of first choice. Whenever feasible, laboratory personnel should use pollution prevention techniques to address their waste generation. When wastes cannot be feasibly reduced at the source, the agency recommends recycling as the next best option.

13.2. The quantity of chemical purchased should be based on expected usage during its shelf life and disposal cost of unused material. Actual reagent preparation volumes should reflect anticipated usage

14.0. Waste Management

14.1. The U.S. Environmental Protection Agency requires that laboratory waste management practices conducted be consistent with all applicable rules and regulations. All waste will be disposed of in accordance with Federal, State and Local regulations. Where reasonably feasible, technological changes have been implemented to minimize the potential for pollution of the environment. Employees will abide by this method and the policies in section

13 of the Corporate Safety Manual for "Waste Management and Pollution Prevention."

14.2. The following waste streams are generated as a result of this analysis:

- Extractions Waste water. This material is created when 50% Acetone and 50% Methylene Chloride are added to 1 liter of sample water. The water is shaken with the solvent. The solvent is collected with the compounds of interest and the water is discarded into the Extractions Waste Water drum. This drum is removed to the walk-in hood in the waste room. A ½ inch PVC pipe is inserted into the bung hole of the drum and air is passed through the solution over night. The solution is then transferred into the first drum of the neutralization system and neutralized to a pH of 6 – 9. This solution is discharged into the municipal sewer system.

- Mixed Solvent Waste. This material is collected from rinsing and other processes into 5 gallon solvent cans as satellite accumulation. These cans are emptied every 24 hours into a steel drum in the waste room. This drum is kept in the walk in hood until it is full. The full drum is then removed from the hood and placed on secondary containment in the waste room.
  Teris Profile Number: 50016624
  Onyx Profile WIP Number: 545240

- Waste sodium sulfate. This material is collected from various methods which require the removal of water from solvent which carries the analyte (s) of interest. The solvent is passed through the sodium sulfate and the sodium sulfate plus the water is disposed of. The sodium sulfate is collected in buckets inside the hoods. The material is air dried and disposed of in the municipal waste dumpster.

- Waste sulfuric acid. This material is generated from clean up of PCB extracts for sulfur compounds. The acid is collected in satellite accumulation in the hood. The container is removed to the waste room for neutralization with 50 % sodium hydroxide (Siedler Chemical SC-1824-03), water and sodium bicarbonate (Siedler Chemical SC-0219-25). Ice is used to control temperature in the plastic drums of the neutralization system. When neutralization is complete (pH 6 -9) the material is transferred to the municipal sewer system.

15.0. References / Cross-References

15.1. United States Environmental Protection Agency, "Method 8151A: Organochlorine Herbicides by Gas Chromatography", Test Methods for Evaluating Solid Wastes, SW846 Third Edition, Volume 1B: Laboratory Manual, Physical/Chemical Methods, Revision 3, December 1996.

15.2. TestAmerica Edison SOP No. ED- ORP-023, *Extraction of Organochlorine Herbicides in Soil by SW846 Method 8151A,* current revision.

15.3. TestAmerica Edison Document No. ED-QA-LQM, *Laboratory Quality Manual*, current revision.

15.4. TestAmerica Corporate Document No. CW-E-M-001, *Environmental Health and Safety Manual,* current revision.

15.5. TestAmerica Corporate Quality SOP No. CA-Q-S-001, *Solvent & Acid Lot Testing & Approval*, current revision.

15.6. TestAmerica Edison SOP No. ED-GEN-023, *Bulk Solvent Testing and Approval*, current revision.

15.7. TestAmerica Edison SOP No. ED-GEN-013, *Glassware Cleaning*, most current revision

15.8. TestAmerica Edison SOP No. ED-GEN-022, *Training,* current revision.

16.0. Method Modifications:

NONE

17.0. Attachments

Attachment 1 – Surrogate Solution Prep

18.0. Revision History

Revision 15, 10/5/2021

- Updated Lab Quality Manual section references as needed throughout document.
- Updated throughout to reflect used of TMSD for esterification (replacing diazald)
- Section 12: updated to reflect current MDL procedures.

Revision 14, April 26, 2019

- Section 10.4.11: methanol was added to extract to help with better herbicide recoveries.

- Revision 13, dated February 22, 2016:
  - Updated Table 2 with the corrected spelling of Picloram(4-amino-3,5,6-trichloropicolinic acid.

- Section 7.1.1.3: corrected the preparation instructions for 12N sulfuric acid. The correct volume of water is 200ml (was 300 ml). 100ml concentrated sulfuric diluted into 200 ml water is 12N.
- Section 7.1.1.5: clarified to indicate that a 25% H2SO4 solution is a 1:3 solution.

- Revision 12, dated January 28, 2016:

  - Sections 7.2.1 and 7.3.1: updated info to reflect prep using new standard solutions.
  - Tables 1 and 2 updated with new standard mixes and sources.
  - Attachment 1: deleted. Attachment 2 now becomes Attachment 1.
  - Section 7.3.2.1: revised to reflect new standards and procedures.
  - Section 7.3.3: updated to revise from Attachment 2 to Attachment 1.
  - Section 8.0: added 8 oz amber glass container as option.
  - Section 10.1.1: added 500 ml sep funnel as option.
  - Section 10.1.9 and 10.6.8: updated spiking instructions.
  - Section 10.4.11: updated and clarified final extract volumes.

- Revision 11, dated January 27, 2014::

  - Minor editorial changes throughout document.
  - Section 7.2.2: Update standard sources and mixes (Table 1).
  - Section 7.3.2 and 7.3.3: Updated standards prep information.
  - Section 10.1.9: updated QC spiking procedures (LCS/MS/MSD).
  - Section 10.6.8: updated leachate QC spiking procedures.
  - Added Attachment 1 which details the individual target compounds and their concentrations in the new standards.
  - Added Attachment 2 which details the individual surrogate compounds and their concentrations in the new standards.

- Revision 10, dated January 6, 2012:

  - Section 7.2.2, Table 2: updated concentration of MCPP spiking mix.
  - Section 7.2.2, Tables 1 and 2: Updated source of Picloram spiking standard.
  - Section 7.3.2.4: revised instructions for preparation of Picloram spiking solution.
  - Corrected number of Sections 7.3.2.1 thru 7.3.2.4.
  - Revised Section 10.6.8: corrected typos and added detailed procedures dependent upon spiking scenarios.

- Revision 9, dated December 21 2011:

  - Revised the following sections to reflect the requirement that all glassware must be rinsed with sulfuric acid prior to use in the extraction of herbicides: Section 6.2, Section 10.1.1 and Section 10.6.1.
  - Added Section 7.1.1.5 which describes the preparation of a 25% sulfuric acid: reagent water solution for use in acid-rinsing glassware.

- Throughout document: updated LQM section references as appropriate as some have changed with the latest LQM revision

- Revision 8, dated August 26 2011:

  - Section 5.1: revised to include following statement: “Ethyl Ether must be free of peroxides as determined by use of EM Quant Peroxide Test Strips as described in Sections 6 and 7 below.”
  - Section 6.2: added EM Quant Peroxide Test Strips to supplies list.
  - Section 7.1: added requirement to test Ether for peroxide formation.
  - Revised Section 10.3.4 to include a requirement to pour extract through a funnel plugged with acid washed glass wool. Also revised collection container (was Erlenmeyer flask; now K-D concentrator).
  - Section 10.3.9 was revised to clarify that the ether extracts from each sample are collected into a single container (not multiple separate containers).
  - Section 10.6.8: corrected the spike volume used for the 10 Compound Spike mix. Reworded the section to make the instructions clearer.

- Revision 7, dated October 30, 2009
  - Revised SOP format in accordance with TestAmerica Corporate Quality SOP No. CW-QS-002 (Rev 0) Writing a Standard Operating Procedure (SOP).
  - Section 1.1 Added reference to Quality Assurance Manual for method modifications.
  - Section 1.1: Expanded to include references to applicable prep and cleanup SOPs.
  - Section 3: revised to reference new location for definitions.
  - Section 5: Revised to include most up to date corporate health and safety references and information.
  - Section 7.2.2: Added tables detailing components found in the various standards mixes.
  - Section 7.3: Updated the instructions for preparation of standards.
  - Section 7.3: Added tables with calibration standards prep details.
  - Section 8: Updated with additional details including a table outlining containers, preservation and holding times for waters and soils.
  - Section 9.1: Expanded QC sample preparation details.
  - References: Expanded to include more specific SOP references
  - Section 18: Added this Revision History section
  - Throughout document: added references to TestAmerica LIMS (TALS).

Attachment 1 – Surrogate Standards Preparation

TestAmerica
THE LEADER IN ENVIRONMENTAL TESTING

Reagent ID: OP_HERB_SU_00020

		Expiration Date:	06/10/2014
Description:	HERBICIDE Surrogate	Laboratory:	TestAmerica Edison
No. of Bottles:	10	Prepared By:	Patel, Hemex
Storage Location:	ORP-STDS & SPIKES	Solvent:	Acetone
Reagent Volume:	100.000 mL	Solvent Lot:	33214
Creation Date:	12/10/2013		
Container(s):	2644797, 2644798, 2644799, 2644800, 2644801, 2644802, 2644803, 2644804, 2644805, 2644806		
Comment:			

Reagent Analyte Information

Analyte	Source ID	Source Exp. Date	Source Conc.	Source Conc. Units	Final Conc.	Final Conc. Units
2,4-Dichlorophenylacetic acid	OP_2,4DCAA_00016	09/30/2014	1000.00000	ug/mL	100.00000	ug/mL

Source Reagents

Reagent	Description	Type	Expiration	Vendor	Vendor Lot #	Vendor Cat Lot #	Volume Used	Volume Units
OP_2,4DCAA_00016	2,4 - DCAA Acid Spike Mix	ASTD	09/30/14	RESTEK	A093820	567804	10.00000	mL

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Title: Extraction of Organochlorine Herbicides in Soil by SW846 Method 8151A

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Approvals (Signature/Date):

Mamdou Leye	10/8/20 Date	Dan Helfrich	10/8/20 Date
Organic Prep Department Manager		Health & Safety Manager / Coordinator	
Carl Armbruster	10/8/20 Date	Mark Acierno	10/8/20 Date
Quality Assurance Manager		Laboratory Director	

Sylvanus Klusey 10/8/20 Date
Operations Manager - Organics

Facility Distribution No. ELECTRONIC **Distributed To: FACILITY INTRANET**

1.0 Scope and Application

1.1. **Analytes, Matrix(s), and Reporting Limits:** SW846 Method 8151A provides extraction and derivatization procedures for the gas chromatographic analysis of chlorinated acid herbicides in water, soil, and waste samples. An option for the hydrolysis of esters is also described. This SOP details the extraction of soil samples with diethyl ether follower by esterification with diazomethane. The derivatives are determined by gas chromatography with an electron capture detector (GC/ECD). The results are reported as acid equivalents.

1.2. For a complete discussion of analytes and reporting limits (RLs) please refer to TestAmerica Edison SOP No. ED-GCS-00, *Analysis of Organochlorine Herbicides by SW846 Method 8151A,* current revision.

1.3. On occasion clients may request modifications to this SOP. These modifications are handled following the procedures outlined in Section 7 (*Review of Work Request*) and 19 (*Test Methods and Method Validation*) of TestAmerica Edison's Quality Assurance Manual (ED-QA-LQM).

2.0 Summary of Method

This SOP provides extraction and derivatization procedures for the determination of chlorinated acid herbicides and their esters in solid samples.

Soil samples are serially extracted with diethyl ether. The extracts are hydrolyzed, dried and concentrated and then esterified with (Trimethylsilyl)diazomethane. The derivatives are determined by gas chromatography with an electron capture detector (GC/ECD).

The sensitivity of Method 8151 depends on the level of interferences in addition to instrumental limitations.

3.0 Definitions

For a complete list of definitions refer to Appendix 2 in the most current revision of TestAmerica Edison's Quality Assurance Manual (ED-QA-LQM).

4.0 Interferences

Method interference may be caused by contaminants in solvents, reagents, glassware and other sample processing hardware that lead to discrete artifacts or elevated baselines in gas chromatograms. All these materials must be routinely demonstrated to be free from interference under the conditions of the analysis, by analyzing reagent blanks.

Matrix interference may be caused by contaminants that are co-extracted from the sample. Organic acids, especially chlorinated acids, cause the most direct

interference with the determination by methylation. Phenols, including chlorophenols, may also interfere with this procedure.

The acid forms of the analytes are strong organic acids that react readily with alkaline substances and can be lost during sample preparation. Glassware and glass wool must be acid-rinsed with 1 N hydrochloric acid and the sodium sulfate must be acidified with sulfuric acid prior to use to avoid analyte losses due to adsorption.

Sample extracts must be dry prior to methylation or else poor recoveries will result.

5.0 Safety

Employees must abide by the policies and procedures in the Corporate Environmental Health and Safety Manual (CW-E-M-001) and this document. This procedure may involve hazardous material, operations and equipment. This SOP does not purport to address all of the safety problems associated with its use. It is the responsibility of the user of the method to follow appropriate safety, waste disposal and health practices under the assumption that all samples and reagents are potentially hazardous. Safety glasses, gloves, lab coats and closed-toe, nonabsorbent shoes are a minimum.

5.1. Specific Safety Concerns or Requirements

Ethyl Ether is an extremely flammable solvent. If a mechanical device is used for sample extraction, the device should be equipped with an explosion proof motor and placed in a hood.

Ethyl Ether must be free of peroxides as determined by use of EM Quant Peroxide Test Strips as described in Sections 6 and 7 below.

Diazomethane can explode under certain conditions therefore take the following precautions:

- Use a well-ventilated hood.
- Use a safety screen.
- Do not heat above 90°C.
- Avoid grinding surfaces, ground glass joints, sleeve bearings, and glass stoppers.
- Store away from alkali metals.
- Solutions of diazomethane decompose rapidly in the presence of solid materials such as copper powder, calcium chloride, and boiling chips.

The use of Kevlar gloves is required for the assembly/disassembly of ground glass joints in addition to those tasks that present the potential risk for injury.

Ether creates excessive pressure very rapidly. Therefore, initial venting should be done immediately after the separatory funnel has been sealed and inverted. Vent into hood away from analysts and other samples.

5.2. Primary Materials Used

The following is a list of the materials used in this method, which have a serious or significant hazard rating. **Note: This list does not include all materials used in the method. The table contains a summary of the primary hazards listed in the MSDS for each of the materials listed in the table.** A complete list of materials used in the method can be found in the reagents and materials section. Employees must review the information in the MSDS for each material before using it for the first time or when there are major changes to the MSDS.

Material (1)	Hazards	Exposure Limit (2)	Signs and symptoms of exposure
Methylene Chloride	Carcinogen Irritant	25 ppm-TWA 125 ppm-STEL	Causes irritation to respiratory tract. Has a strong narcotic effect with symptoms of mental confusion, light-headedness, fatigue, nausea, vomiting and headache. Causes irritation, redness and pain to the skin and eyes. Prolonged contact can cause burns. Liquid degreases the skin. May be absorbed through skin.
Sodium Hydroxide	Corrosive Poison	2 ppm, 5 mg/m^3	This material will cause burns if comes into contact with the skin or eyes. Inhalation of Sodium Hydroxide dust will cause irritation of the nasal and respiratory system.
Sulfuric Acid	Corrosive Oxidizer Dehydrator Poison Carcinogen	1 Mg/M3-TWA	Inhalation produces damaging effects on the mucous membranes and upper respiratory tract. Symptoms may include irritation of the nose and throat, and labored breathing. Symptoms of redness, pain, and severe burn can occur. Contact can cause blurred vision, redness, pain and severe tissue burns. Can cause blindness.
Acetone	Flammable	1000 ppm-TWA	Inhalation of vapors irritates the respiratory tract. May cause coughing, dizziness, dullness, and headache.
Ethyl Ether	Flammable Irritant Peroxide Former	400 ppm-TWA	General anesthesia by inhalation can occur. Continued exposure may lead to respiratory failure or death. Early symptoms include irritation of nose and throat, vomiting, and irregular respiration, followed by dizziness, drowsiness, and unconsciousness. May cause irritation, redness and pain to the eyes. Irritating to the skin and mucous membranes by drying effect. Can cause dermatitis on prolonged exposure. May be absorbed through skin. **May form explosive peroxides on long standing or after exposure to air or light. This material must be disposed of within six months.**

Material (1)	Hazards	Exposure Limit (2)	Signs and symptoms of exposure
Hexane	Flammable Irritant	500 ppm-TWA	Inhalation of vapors irritates the respiratory tract. Overexposure may cause lightheadedness, nausea, headache, and blurred vision. Vapors may cause irritation to the skin and eyes.
1 – Always add acid to water to prevent violent reactions.			
2 – Exposure limit refers to the OSHA regulatory exposure limit.			

6.0 Equipment and Supplies

6.1. Equipment

- Top loading Balance - Sargent Welch S2642-25 or equivalent
- Steam Bath - Fisher Scientific 66738 or equivalent.
- Furnace - Thermolyne Type 6000 or equivalent.
- Vacuum Pump - General Electric Model No. 0322-V48-G180X or equivalent.
- N-Evap - Meyer Analytical Evaporator Model No. 112 or equivalent
- Explosion Proof Hood
- Desiccator - Baxter D1420-2 or equivalent.

6.2. Supplies

- 250 ml Clear Glass Jar
- 500 ml Erlenmeyer Flask
- 125 ml Erlenmeyer Flask
- 125 ml Separatory Funnel
- 500 ml Centrifuge Bottle - Pyrex 1260 or equivalent.
- 10 ml graduated Concentrator Tube - Kontes K570050-1025 or equivalent.
- 10 ml glass vials with Teflon lined screw caps.
- 20 ml glass vials with Teflon lined screw caps.
- 5 ml to 1000 ml Class A Volumetric flasks
- Funnel - 75 mm diameter

- 3 Ball Macro Snyder Column - Baxter K503000-0121 or equivalent.
- 50 ul Gastight Syringe - VWR 60376-241 or equivalent.
- 100 ul Gastight Syringe - Baxter S9662-5 or equivalent.
- 100 ml Graduated Cylinder
- 1 ml Pasteur Disposable Pipette
- Kuderna Danish (K-D) - Kontes 500 ml - Baxter K570001-0500 or equivalent.
- 10 ml Disposable Pipette
- Glass stopper - size 24/40
- Teflon stopper - size 19/22
- Phosphoric Acid Washed Glass Wool - Supelco 2-0383 or equivalent.
- Septa, 12 mm - Supelco 2-3273 or equivalent.
- Septa, 8 mm - Supelco 2-3272 or equivalent.
- Standard Taper Clamp (size 19, blue) - Wheaton 297749 or equivalent.
- Silicon Carbide Boiling Stones - Thomas 1590-D33 or equivalent.
- Explosion Shield – Nalgene 6350 or equivalent
- Mini Stir Plate
- pH Paper
- Tongue Depressors
- EM Quant Peroxide Test Strips, EMD No. 10011-1 or equivalent

Note: All glassware must be pre-rinsed with a 25% Sulfuric Acid: reagent water solution.

7.0 Reagents and Standards

7.1 Reagents

Note: Each lot of Methylene Chloride, Acetone, Methanol and Sulfuric Acid is screened for contaminants before being used for analysis as detailed in TestAmerica Corporate Quality SOP No. CA-Q-S-001 (*Solvent & Acid Lot Testing & Approval*) and TestAmerica Edison SOP No. ED-GEN-023 (*Bulk Solvent Testing and Approval*).

- Trimethylsilyldiazomethane (TMSD) , 2.0 molar solution in hexane, Acros 429211000 or equivalent.
- Diethyl Ether (Residue Analysis Grade with 2% Ethanol preservative) - Baker 9259-03 or equivalent (tested for peroxide formation upon opening and monthly thereafter by use of EM Quant Peroxide Test Strips)
- Methylene Chloride (High Purity Grade) - Baker 9266 or equivalent.
- Acetone (High Purity Grade) – Baker 9254 or equivalent.
- Sodium Sulfate Anhydrous Crystals (Reagent Grade) – Malinckrodt 8024 or equivalent. (Baked at 400°C for 4 hours)
- Hexane (High Purity Grade) – Baker 9262 or equivalent.
- Carbitol (diethylene glycol monoethyl ether)
- Potassium Hydroxide (Analytical Reagent Grade) – Aldrich 306568 or equivalent
- Sulfuric Acid (Analytical Reagent Grade) - Mallinckrodt 2876 or equivalent.
- Silicic Acid, Aldrich 30, 636-3 or equivalent
- Peroxide Test Strips - Aldrich Z10, 168-0 or equivalent
- Organic free reagent water - Reagent water is defined as a water in which an interference is not observed at the MDL of each parameter of interest (ASTM Specification D1193, Type ii). At TestAmerica Edison this water is generated by the Barnstead/Thermolyne Water System (Model # D11991 Serial # 1191020210415).

7.1.1 Reagent preparation

7.1.1.1 Anhydrous sodium sulfate crystals must be baked in the muffle furnace at 400° C for 4 hours prior to use. After baking, the sodium sulfate must be acidified. Place 500 grams of anhydrous sodium sulfate into a 500 ml wide mouth Erlenmeyer flask. Add enough diethyl ether to cover the sodium sulfate. Add 0.5 ml concentrated sulfuric acid. Shake vigorously for 1 minute. Evaporate the ether by placing the flask on steam bath. Mix 1 g of the resulting solid with 5 ml of organic-free reagent water and measure the pH of the mixture. It must be below a pH of 4. Prepare fresh daily.

7.1.1.2 37% KOH Solution: Rinse a 1 liter Pyrex flask three times with organic free reagent water. Weigh 148 g KOH into flask and add 400 ml organic free reagent water. Mix until the KOH is completely dissolved and the flask has cooled

to room temperature. Transfer the KOH solution to a clean 500-ml amber bottle and label.

7.1.1.3 12N H2SO4: Rinse a 1 liter Pyrex flask three times with organic free reagent water. Add 200 ml organic free reagent water to the flask. Slowly, and while mixing, add 100-ml concentrated H2SO4 to flask. Continue mixing until flask has cooled to room temperature. Transfer the H2SO4 solution to a clean 500-ml amber bottle and label.

7.1.1.4 25% H2SO4 solution: 25 ml Acid and 75 ml reagent water. Add 75 ml organic free reagent water to a graduated cylinder. Slowly, and while mixing, add 25-ml concentrated H2SO4 to cylinder. Continue mixing until cylinder has cooled to room temperature. Transfer the H2SO4 solution to a clean amber bottle and label.

7.2 Standards

7.2.1 Standards are purchased as concentrated solutions (see Section 7.2.2). Most stock solutions are diluted (in volumetric glassware) to working concentration using hexane as the diluent as described in Section 7.3.

7.2.2 **Standard mixes and sources:** Table 1 lists the standard concentrate mix sources. Table 2 details the individual components of these mixes.

Table 1:
Herbicide Standard Mixes and Sources*

Standard Name ("Lab Name")	Concentration (ug/ml)	Source
Acid Herbicide LCS RTS ("Spiking Mix")	varies (in Methanol)	Restek Catalog # 567950
DCAA standard	1000 (in methanol)	Restek Catalog #567804

*May be substituted with equivalent standards from alternate sources.

Table 2:
Components of Herbicide Standard Mixes

Parameter	Catalog Nos.	Lab Standard Name	Concentration (ug/ml)
2,4-dichlorophenoxyacetic acid (2,4-D)	Restek Catalog # 567950	Spiking Mix	20
2-(2,4,5-Trichlorophenoxy)propionic acid (2,4,5-TP aka Silvex)	Restek Catalog # 567950	Spiking Mix	5
2,4,5-T	Restek Catalog # 567950	Spiking Mix	5
2,4-DB	Restek Catalog # 567950	Spiking Mix	20
Dalapon	Restek Catalog #	Spiking Mix	20

Table 2: Components of Herbicide Standard Mixes			
Parameter	**Catalog Nos.**	**Lab Standard Name**	**Concentration (ug/ml)**
	567950		
Dicamba	Restek Catalog # 567950	Spiking Mix	10
Dichloroprop	Restek Catalog # 567950	Spiking Mix	20
Dinoseb	Restek Catalog # 567950	Spiking Mix – 10 compound	20
MCPA acid	Restek Catalog # 567950	Spiking Mix – 10 compound	2000
MCPP acid	Restek Catalog # 567950	Spiking Mix – 10 compound	2000
Pentachlorophenol	Restek Catalog # 567950	Pentachlorophenol spike	5
Picloram (4-amino-3,5,6-trichloropicolinic acid)	Restek Catalog # 567950	Picloram Acid Spiking Standard	20
2,4-Dichlorophenylacetic Acid	Restek Catalog # 567804	Surrogate Spiking Solution	1000

7. 3. Standards Preparation

7.3.1. All standard stock solutions are diluted to the working concentrations as required with acetone (as indicated) using Class A volumetric glassware.

7.3.2. Herbicide Spiking Standard (LCS/MS/MSD):

7.3.3.1 Herbicide QC Spiking Standard Mix (12 compound analysis): The (12) compound spiking standard is ready to spike and used when analyzing for the standard list of herbicides The spiking standard mix is a 25 ml solution (Restek Catalog # 567950; see Tables 1 and 2) of the 12 compounds at the specified concentrations as free acids (see Table 2). The concentrate is used without further dilution prior to the spiking of the following quality control sample types: MS, MSD, and LCS. Spiking instructions are found in Section 10.

7.3.3. Herbicide Surrogate Spiking Standard (2,4-DCAA Acid Spike Mix**):** The herbicide surrogate spiking solution is a 1 ml solution of 2,4-DCAA at a concentration of 1000 ug/ml. (Supelco Catalog # 861271, see Tables 1 and 2). The concentrate is used after 10X dilution in acetone prior to the spiking of samples (final concentration of spiking standard is 100 ug/ml).

8.0 Sample Collection, Preservation, Shipment and Storage

Sample container, preservation techniques and holding times may vary and are dependent on sample matrix, method of choice, regulatory compliance, and/or specific contract or client requests. Listed below are the holding times and the references that include preservation requirements.

Matrix	Sample Container	Min. Sample Size	Preservation	Holding Time	Reference
Soil	Glass	8oz clear glass	Cool 4 ± 2ºC	14 days to extraction; 40 days to analysis	USEPA SW846

8.1. Extracts must be stored under refrigeration (Cool 4 ± 2ºC) in the dark and analyzed within 40 days of extraction.

9.0 Quality Control

9.1 Sample QC - The following quality control samples are prepared with each batch of samples.

Quality Controls	Frequency	Control Limit
Method Blank (MB)	1 in 20 or fewer samples	< Rpt. Limit
Laboratory Control Sample (LCS)[1]	1 in 20 or fewer samples	Statistical Limits [4]
Matrix Spike (MS)[2]	1 in 20 or fewer samples	Statistical Limits [4]
MS Duplicate (MSD)[2]	1 in 20 or fewer samples	Statistical Limits [4]
Surrogates	every sample[3]	Statistical Limits [4]

[1] LCS Duplicate (LCD) is performed only when insufficient sample is available for the MS/MSD or when requested by the client/project/contract.
[2] The sample selection for MS/MSD is randomly selected by the organic prep lab, unless specifically requested by a client.
[3] Analytical and QC samples (MB, LCS, MS/MSD)
[4] Statistical control limits are updated annually and are updated into TALS (LIMS).

9.1.1. Method Blanks are extracted with each sample batch on each day that samples are extracted. The analytical results for the method blank must fall below the reporting limit for each compound of interest. If a target compound is detected in the blank at a concentration higher than the reporting limit, first the extract is reanalyzed for confirmation. If results are still outside of limits the entire batch of samples extracted with the affected blank must be re-extracted and reanalyzed.

9.1.2. Laboratory Control Sample (LCS): A Laboratory Control Sample (LCS) or blank spike must be extracted and analyzed for with each batch of 20 environmental samples. The LCS data is used to ensure that the analytical system is in control. It is also used to assess

performance if the MS/MSD recoveries fall outside of established limits. The recoveries of the LCS must fall within lab generated acceptance criteria. If the spiked sample recovery results fall outside the laboratory generated limits (refer to the current active TALS method limit group database), the LCS recovery is evaluated. If LCS recovery is within limits the poor sample recovery results are attributed to matrix interference. If the LCS recovery results are outside QC limits, first the extract is reanalyzed and if it is still outside the limits the entire QC batch must be re-extracted and reanalyzed.

9.1.3. Matrix Spike/Matrix Spike Duplicate (MS/MSD): A Matrix Spike/Matrix Spike Duplicate (MS/MSD) pair is extracted and analyzed with every batch of 20 environmental samples. MS/MSD recoveries are evaluated against lab generated limits (refer to refer to the current active TALS method limit group database). If the MS/MSD recovery limits fall outside of lab limits the LCS recovery is evaluated and corrective action is taken as described in TestAmerica Edison SOP No. ED-GCS-00, *Analysis of Organochlorine Herbicides by SW846 Method 8151A,* current revision.

9.1.4. Surrogate Standards: All samples, blanks and QC samples are spiked with surrogate standard solution containing 2,4-DCAA (see Section 7.3.3). The percent recovery of the surrogate standards is calculated and compared to lab generated limits (refer to refer to the current active TALS method limit group database). If the surrogate recovery limits are outside of acceptance limits the sample extract is reanalyzed to confirm. If the recoveries are still outside of limits the sample must be re-extracted and reanalyzed or the data flagged as “estimated concentration”.

10.0. Procedure

10.1. Sample Extraction (Soil Samples)

10.1.1. Rinse enough graduated cylinders and flasks once with acetone, twice with diethyl ether and once with 25% sulfuric acid: reagent water.

10.1.2. Weigh out 30g (+/- 0.1g) of soil sample into a clean clear glass 8oz sample container. Mark the lab sample identification numbers on the corresponding sample containers with red wax pencil.

10.1.3. Make up tags for each sample with the following information and place on the corresponding 500 ml Erlenmeyer flasks.

HERBICIDES
Sample Number
Matrix
Date of Extraction

10.1.4. Weigh out approximately 30 g of sodium sulfate into two clean glass jars. These will serve as the blank and LCS samples respectively. Label with sample identification using red wax pencil.

10.1.5. Rinse each syringe to be used for adding surrogates and spikes, eight to ten times with acetone.

10.1.6. Add 50 ul of 100 ppm 2,4-Dichlorophenylacetic Acid (DCAA) Surrogate Spiking Solution (see Table 1) to each sample and QC sample. NOTE: when spiking the samples with either surrogates or target spiking compounds, ensure that there are no bubbles in the syringe. In addition, hold the syringe just above the level of the soil when adding the spike. Do not touch the tip of the syringe to the soil or to the side of the glass jar. Swirl the separatory funnel after adding surrogate spike. All spikes must be witnessed.

10.1.7. When preparing QC spike samples (MS/MSD/LCS/LCSD), spike with 1000ul of the Herbicide QC Spiking Standard Mix (Restek 569750,see Section 7.3.3.1). Swirl the glass jar after adding the spike. All spikes must be witnessed.

10.1.8. Adjust the pH of each sample and QC sample to 2 with concentrated HCl (about 5ml) and monitor the pH for 15 minutes with occasional stirring. If necessary, add additional HCl until the pH remains at 2.

10.1.9. Add 20 ml of acetone to each sample. Mix each sample with the wrist shaker for 20 minutes.

10.1.10. Add 80 ml of diethyl ether to the each sample and shake again for 20 minutes. Decant the extract and measure the volume of solvent recovered.

10.1.11. Extract the sample twice more using 20 ml of acetone followed by 80 ml of diethyl ether. After addition of each solvent, the mixture should be shaken with the wrist shaker for 10 minutes and the acetone-ether extract decanted.

10.1.12. After the third extraction, the volume of extract recovered should be at least 75% of the total volume of solvent added. If solvent recovery is less than 75%, extract once more with 20 ml of diethyl ether.

10.1.13. Check the pH of the extract. If it is not at or below pH 2, add more concentrated HCl until stabilized at pH of 2.

10.1.14. Into a pre-rinsed 500ml separatory funnel add 250ml of organic free reagent water.

10.1.15. Add 5ml of H2SO4 to the reagent water and check with pH paper to insure pH is<2.

10.1.16. Add the solvent extract to separatory funnel and shake slowly for one minute. Let stand for 5 minutes.

10.1.17. Discard the aqueous phase (bottom layer) and collect the extract (top layer) into a pre-rinsed 500ml Erlenmeyer flask for hydrolysis.

10.2. Hydrolysis

10.2.1. To each 500-ml Erlenmeyer flask containing the ether extract, add 2 ml of the 37% KOH solution, 15 ml of reagent water, and 4-5 boiling chips.

10.2.2. Attach a three-ball macro Snyder and place Erlenmeyer flask on steam bath (100ºC) for 1½ to 2 hours.

10.3. Partitioning

10.3.1. Remove the Erlenmeyer flask from the steam bath and allow it to cool for 5-10 minutes.

10.3.2. Pour the extract into a 500-ml separatory funnel pre-rinsed with diethyl ether.

10.3.3. Add 40 ml of diethyl ether to the separatory funnel and shake for two minutes.

10.3.4. Pour the dried diethyl ether extract through a funnel plugged with acid washed glass wool, collecting the extract in the K-D concentrator. Use a glass rod to crush any caked sodium sulfate during the transfer.

10.3.5. Repeat twice using 20-ml aliquots of diethyl ether.

10.3.6. Discard the ether phase.

10.3.7. Adjust the pH of the extract remaining in the 500-ml separatory funnel to ≤ 2 by adding 5 ml of 12N Sulfuric Acid. Check pH with pH paper.

10.3.8. Add 40 ml of diethyl ether to the separatory funnel and shake for 2 minutes with periodic venting to release pressure.

10.3.9. Collect each diethyl ether phase into one pre-cleaned screw top glass jars containing approximately 10 g of acidified sodium sulfate crystals and mix well.

10.3.10. Repeat sections10.3.8 and 10.3.9 twice using 20-ml aliquots of diethyl ether.

10.3.11. Allow the diethyl ether extract to remain in contact with the sodium sulfate for approximately 2 hours.

10.3.12. Pour the dried diethyl ether extract through a funnel plugged with acid washed glass wool, collecting the extract in the K-D concentrator. Use a glass rod to crush any caked sodium sulfate during the transfer.

10.3.13. Place the white identification tag on the K-D apparatus.

10.3.14. Rinse the Erlenmeyer flask and funnel with 20 to 30 ml of diethyl ether to complete the quantitative transfer.

10.4. Extract Concentration

10.4.1. Add one or two clean boiling stones to the K-D flask and attach a three-ball Snyder column.

10.4.2. Pre-wet the Snyder column by adding about 1 ml of diethyl ether to the top of the column.

10.4.3. Place the K-D apparatus on a hot water bath (15-20°C above the boiling point of the solvent; set at LOW which is 60ºC) such that the concentrator tube is partially immersed in the hot water and the entire lower rounded surface of the flask is bathed with hot vapor.

10.4.4. Adjust the vertical position of the apparatus and the water temperature, as required, to complete the concentration in 10-20 minutes.

10.4.5. Boil the sample in the K-D flask on the steam bath until the sample volume is approximately 1 ml.

10.4.6. Add 60 ml of hexane and concentrate further to a final volume of approximately 5 ml.

10.4.7. Remove the K-D apparatus from the water bath and allow it to drain and cool for at least 10 minutes.

10.4.8. Remove the Snyder column from the top of the K-D flask. Remove the blue taper clamp from the ground glass joint and dry the exterior with a Kimwipe. Remove the K-D flask from the concentration tube.

10.4.9. Place the concentration tube onto the N-Evap apparatus. Maintain I.D. tag with concentration

10.4.10. Continue to concentrate under a N2 stream until the sample volume is approximately 1 ml.

10.4.11. To each sample add 500ul of methanol and dilute to a final volume of 10 ml with hexane.

10.5. Esterification

10.5.1. Add approximately 200 uL of methanol to the extract followed by approximately 200 uL of 2.0M Trimethylsilyldiazomethane (TMSD) solution. The extract will turn a yellow color. If this does not occur, add additional (approximately) 200 uL aliquots of 2.0M Trimethylsilyldiazomethane (TMSD) solution until the yellow color persists. Check the sample every 15 minutes for yellow color. If the yellow disappears in this time frame, continue adding an additional aliquot of 2.0M Trimethylsilyldiazomethane (TMSD) solution until the yellow color persists.

10.5.2. Allow the extract to stand for at least one hour at room temperature to allow the methylation reaction to occur. The reaction is halted with the addition of approximately 0.05g (one scoop using a disposable spatula) of silica gel. The extract is then brought up to a final volume of 10 mL with hexane by visually comparing it to a calibrated collection tube.

10.5.3. Adjust the final extract volumes to 10ml with hexane. The extracts ready to be analyzed by GC. Transfer some volume to GC autosampler vials.

10.5.4. Samples must be analyzed as soon as possible (less than 24 hrs. preferably) after esterification. This is to minimize the trans-esterification and other potential reactions that may occur.

10.5.5. Store refrigerated if further processing will not be performed immediately.

10.5.6. Extracts should be stored at 4°C away from light.

10.6. Required Documentation:

The organic prep technician is responsible for completing the following items.

10.6.1. The TALS reagent database must be completed in full with the required information whenever standards are logged and/or prepared.

10.6.2. All required data types (sample volumes, reagent and standard volumes, reagent and standard lots, etc…) must be entered into the TALS batch database each time an extraction is performed.

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11.0. Calculations / Data Reduction

None

12.0. Method Performance

12.1. Method Detection Limit Study (MDL)

The method detection limit (MDL) is the lowest concentration that can be detected for a given analytical method and sample matrix with 99% confidence that the analyte is present. The MDL is determined according to the laboratory's MDL procedure in Section 19 (Test Methods and Method Validation) of TestAmerica Edison's Quality Assurance Manual (ED-QA-LQM). MDLs reflect a calculated (statistical) value determined under ideal laboratory conditions in a clean matrix, and may not be achievable in all environmental matrices. The laboratory maintains MDL studies for analyses performed; these are verified at least annually unless method requirements require a greater frequency.

12.2. Demonstration of Capabilities

For DOC procedure refer to Section 19 in the most current revision of TestAmerica Edison's Quality Assurance Manual (ED-QA-LQM).

12.3. Training Requirements

Refer to the current revision of TestAmerica Edison SOP No. ED-GEN-022, *Training*, for the laboratory's training program.

13.0. Pollution Control

13.1. Pollution prevention encompasses any technique that reduces or eliminates the quantity or toxicity of waste at the point of generation. Numerous opportunities for pollution prevention exist in laboratory operation. The USEPA has established a prevention hierarchy of environmental management techniques that places pollution prevention as the management option of first choice. Whenever feasible, laboratory personnel should use pollution prevention techniques to address their waste generation. When wastes cannot be feasibly reduced at the source, the agency recommends recycling as the next best option.

13.2. The quantity of chemical purchased should be based on expected usage during its shelf life and disposal cost of unused material. Actual reagent preparation volumes should reflect anticipated usage

14.0. Waste Management

14.1. The U.S. Environmental Protection Agency requires that laboratory waste management practices conducted be consistent with all applicable rules and regulations. All waste will be disposed of in accordance with Federal, State and Local regulations. Where reasonably feasible, technological changes have been implemented to minimize the potential for pollution of the environment. Employees will abide by this method and the policies in section 13 of the Corporate Safety Manual for "Waste Management and Pollution Prevention."

14.2. The following waste streams are generated as a result of this analysis:

- Extractions Waste water. This material is created when 50% Acetone and 50% Methylene Chloride are added to 1 liter of sample water. The water is shaken with the solvent. The solvent is collected with the compounds of interest and the water is discarded into the Extractions Waste Water drum. This drum is removed to the walk-in hood in the waste room. A ½ inch PVC pipe is inserted into the bung hole of the drum and air is passed through the solution over night. The solution is then transferred into the first drum of the neutralization system and neutralized to a pH of 6 – 9. This solution is discharged into the municipal sewer system.

- Mixed Solvent Waste. This material is collected from rinsing and other processes into 5 gallon solvent cans as satellite accumulation. These cans are emptied every 24 hours into a steel drum in the waste room. This drum is kept in the walk in hood until it is full. The full drum is then removed from the hood and placed on secondary containment in the waste room.
  Teris Profile Number: 50016624
  Onyx Profile WIP Number: 545240

- Waste sodium sulfate. This material is collected from various methods which require the removal of water from solvent which carries the analyte (s) of interest. The solvent is passed through the sodium sulfate and the sodium sulfate plus the water is disposed of. The sodium sulfate is collected in buckets inside the hoods. The material is air dried and disposed of in the municipal waste dumpster.

- Waste sulfuric acid. This material is generated from clean up of PCB extracts for sulfur compounds. The acid is collected in satellite accumulation in the hood. The container is removed to the waste room for neutralization with 50 % sodium hydroxide (Siedler Chemical SC-1824-03), water and sodium bicarbonate (Siedler Chemical SC-0219-25). Ice is used to control temperature in the plastic drums of the neutralization system. When neutralization is complete (pH 6 -9) the material is transferred to the municipal sewer system.

15.0. References / Cross-References

15.1. United States Environmental Protection Agency, "Method 8151A: Organochlorine Herbicides by Gas Chromatography", Test Methods for Evaluating Solid Wastes, SW846 Third Edition, Volume 1B: Laboratory Manual, Physical/Chemical Methods, Revision 3, December 1996.

15.2. TestAmerica Edison SOP No. ED- ORP-015, *Extraction of Organochlorine Herbicides in Water by SW846 Method 8151A,* current revision.

15.3. TestAmerica Edison Document No. ED-QA-LQM, *Laboratory Quality Manual*, current revision.

15.4. TestAmerica Corporate Document No. CW-E-M-001, *Environmental Health and Safety Manual,* current revision.

15.5. TestAmerica Corporate Quality SOP No. CA-Q-S-001, *Solvent & Acid Lot Testing & Approval*, current revision.

15.6. TestAmerica Edison SOP No. ED-GEN-023, *Bulk Solvent Testing and Approval*, current revision.

15.7. TestAmerica Edison SOP No. ED-GEN-013, *Glassware Cleaning*, most current revision

15.8. TestAmerica Edison SOP No. ED-GEN-022, *Training,* current revision.

16.0. Method Modifications:

NONE

17.0. Attachments

NONE

18.0. Revision History

- Revision 12, dated Oct 08 2020:
  - Section 7.1: added Trimethylsilyldiazomethane (TMSD) , 2.0 molar solution in hexane, Acros 429211000 or equivalent. Removed Diazald.
  - Section10.5.1: Replaced the Diazald esterification procedure with the TMSD procedure.

- Revision 11, dated April 26, 2019
  - Section 10.4.11: methanol was added to extract to help with better herbicide recoveries.

- Revision 10, dated February 22, 2016:

- Revised throughout to correct the spelling of Picloram (4-amino-3,5,6-trichloropicolinic acid
- Section 7.1.1.3: corrected the preparation instructions for 12N sulfuric acid. The correct volume of water is 200ml (was 300 ml). 100ml concentrated sulfuric diluted into 200 ml water is 12N.

- Revision 9, dated January 06, 2016:

  - Tables 1 and 2: updated sources and concentrations of standards.
  - Section 7.3.1 revised to specify acetone as the diluent.
  - Section 7.3.3.1 (spiking solution prep): updated to reflect current standards and procedures. Sections 7.3.3.2, 7.3.3.3 and 7.3.3.4 deleted (obsolete).
  - Section 7.3.3 (surrogate spiking standard prep): updated to reflect current standards and procedures.
  - Section 8.0: updated minimum sample size (8 oz. glass container).
  - Section 10.1.7 (spiking procedures): updated to reflect current practice and new standard mixes.

- Revision 8, dated December 21, 2011

  - Revised the following sections to reflect the requirement that all glassware must be rinsed with sulfuric acid prior to use in the extraction of herbicides: Section 6.2 and Section 10.1.1.
  - Added Section 7.1.1.4 which describes the preparation of a 25% sulfuric acid: reagent water solution for use in acid-rinsing glassware.
  - Throughout document: updated LQM section references as appropriate as some have changed with the latest LQM revision

- Revision 7, dated August 26, 2011

  - Section 5.1: revised to include following statement: “Ethyl Ether must be free of peroxides as determined by use of EM Quant Peroxide Test Strips as described in Sections 6 and 7 below.”
  - Section 6.2: added EM Quant Peroxide Test Strips to supplies list.
  - Section 7.1: definition and source of Organic-Free Water added.
  - Section 7.1: added requirement to test Ether for peroxide formation.
  - Section 10.1.2: added detail regarding the initial weight of sample to be measured (i.e. ‘Weigh out 30g +/- 0.1g)
  - Revised Section 10.3.4 to include a requirement to pour extract through a funnel plugged with acid washed glass wool. Also revised collection container (was Erlenmeyer flask; now K-D concentrator).
  - Revised Section 10.3.9 to include a procedure for mixing well after adding ethyl ether phase to sodium sulfate.
  - Section 10.6 deleted. Section 10.6 was essentially a duplicate of Section 10.5 that had mistakenly been incorporated into the SOP text.

- Revision 6, dated October 30, 2009

  - Revised SOP format in accordance with TestAmerica Corporate Quality SOP

No. CW-QS-002 (Rev 0) Writing a Standard Operating Procedure (SOP).

- Section 1.1 Added reference to Quality Assurance Manual for method modifications.
- Section 1.1: Expanded to include references to applicable prep and cleanup SOPs.
- Section 3: revised to reference new location for definitions.
- Section 5: Revised to include most up to date corporate health and safety references and information.
- Section 7.2.2: Added tables detailing components found in the various standards mixes.
- Section 7.3: Updated the instructions for preparation of standards.
- Section 7.3: Added tables with calibration standards prep details.
- Section 8: Updated with additional details including a table outlining containers, preservation and holding times for waters and soils.
- Section 9.1: Expanded QC sample preparation details.
- References: Expanded to include more specific SOP references
- Section 18: Added this Revision History section
- Throughout document: added references to TestAmerica LIMS (TALS).

Title: Procedure for the Microwave Extraction of Solids, SW846 3546

Once printed, this is considered an uncontrolled document

Approvals (Signature/Date):

Sylvanus Klusey Organic Operations Manager	3/12/18 Date	Dan Helfrich Health & Safety Manager	3/12/18 Date
Carl Armbruster Quality Assurance Manager	3/12/18 Date	Mark Acierno Laboratory Director	3/12/18 Date

1.0 Scope and Application

The procedure uses microwave energy to produce elevated temperature and pressure conditions (i.e., 100^{o} -115^{o}C and 50 - 175 psi) in a closed vessel containing the sample (soil, sediment and sludge) and organic solvent(s) to achieve analyte recoveries equivalent to those from Automated Soxhlet extraction (Method 3541), using less solvent and taking significantly less time than the Automated Soxhlet procedure.

This method is applicable to the extraction of semi-volatile petroleum products in soil, sediment and sludge which may then be analyzed by a variety of chromatographic procedures. The method may also be applicable for the extraction of additional target analytes, provided that the analyst demonstrates adequate performance for the intended application as further described in TestAmerica Edison's Quality Assurance Manual (ED-QA-LQM).

This method only is applicable to solid samples with small particle sizes. If practical the sample may be air dried and ground to a fine powder prior to extraction.

1.1 **Analytes, Matrix(s), and Reporting Limits:** This standard operating procedure details the procedures used by TestAmerica Edison for the microwave extraction of soils, sediments, sludges, and waste solids for semivolatile organic compounds by SW846 Method 3546 for subsequent analysis of:

- Diesel Range Organics (C10-C28 & C8-C44) by SW846 Methods 8015B, 8015C and 8015D (see TestAmerica Edison SOP Nos. ED-GCS-009 and ED-GCS-020);
- TPH by NJDEP-OQA-QAM-025 (see TestAmerica Edison SOP No. ED-GCS-011);
- Organochlorine Pesticides and PCBs by SW846 8081A/B and 8082/A (see TestAmerica Edison SOPs No.ED-GCS-003, ED-GCS-004, ED-GCS-016 and ED-GCS-017)
- Semivolatile Organic Compounds by Gas Chromatography/ Mass Spectrometry (GC/MS) by SW846 8270 and 8270D (TestAmerica Edison SOPs ED-MSS-002 and ED-MSS-009)
- PCBs by GC/MS using EPA Method 680 (TestAmerica Edison SOP ED-MSS-010)

1.2 The microwave prep for NJDEP Extractable Petroleum Hydrocarbons (EPH) (10/08), current revision, is addressed separately in TestAmerica Edison SOP ED-GCS-012 (current revision).

1.3 For a complete discussion of analytes and reporting limits (RLs) please refer to the applicable TestAmerica Edison analytical SOPs referenced in Section 1.1 and in References (Section 14.0).

1.4 On occasion clients may request modifications to this SOP. These modifications are handled following the procedures outlined in Section 7 (*Review of Work Request*) and 19 (*Test Methods and Method Validation*) of TestAmerica Edison's Quality Assurance Manual (ED-QA-LQM).

2.0 Summary of Method

A representative soil, clay, sediment, sludge, solid or waste sample is weighed and placed into a 75mL Teflon vessel and extracted using an appropriate solvent in a microwave extractor. The extraction vessel containing the sample and solvent system is heated to the extraction temperature and extracted for 10 minutes. The mixture is allowed to cool. The vessel is opened and the contents are filtered. The solid material is rinsed and the various solvent fractions are combined. The extract is then dried, concentrated using a nitrogen blow down device, and as necessary, solvent exchanged for use in clean up, fractionation, or determinative methods.

3.0 Definitions

For a complete list of definitions refer to Appendix 2 in the most current revision of TestAmerica Edison's Quality Assurance Manual (ED-QA-LQM).

4.0 Interferences

4.1 Solvents, reagents, glassware, and other sample processing hardware may contribute to interferences with sample analysis. All materials must be demonstrated to be free from interferences under the conditions of the analysis by analyzing a method blanks.

4.2 Phthalate esters are commonly extracted from laboratory items. Plastics in particular, must be avoided because phthalates are easily extracted from these materials. The presence of these contaminants in extracts is problematic because it will give false positive results for the methods that list phthalates as target compounds or it will interfere with the target compounds of other methods that are co-extracted with them.

4.3 All glassware used must be scrupulously cleaned before use in trace analysis. Refer to TestAmerica Edison SOP No. ED-GEN-013 (*Glassware Cleaning*), current revision.

4.4 Use caution if sodium sulfate is used for this method. Salts are known to super heat when used in microwave. Typically samples are analyzed with the addition of 5.0 grams sodium sulfate.

5.0 Safety

Employees must abide by the policies and procedures in the Corporate Safety Manual, Radiation Safety Manual and this document.

This procedure may involve hazardous material, operations and equipment. This SOP does not purport to address all of the safety problems associated with its use. It is the responsibility of the user of the method to follow appropriate safety, waste disposal and health practices under the assumption that all samples and reagents are potentially hazardous. Safety glasses, gloves, lab coats and closed-toe, nonabsorbent shoes are a minimum.

5.1 Specific Safety Concerns or Requirements

Nitrile gloves should be used when performing this extraction. Latex and vinyl gloves provide no significant protection against the organic solvents used in this SOP, and should not be used. During Kuderna-Danish (KD) concentration, do not allow the extract to boil to dryness. The solvent vapors remaining in the KD apparatus may superheat and create an explosion or fire hazard.

The use of the microwave to extract samples creates excessive pressure and temperature within the digestion vessels very rapidly. Cooling the sample after digestion is extremely important.

5.2 Primary Materials Used

The following is a list of the materials used in this method, which have a serious or significant hazard rating. NOTE: This list does not include all materials used in the method. The table contains a summary of the primary hazards listed in the MSDS for each of the materials listed in the table. A complete list of materials used in the method can be found in the reagents and materials section. Employees must review the information in the MSDS for each material before using it for the first time or when there are major changes to the MSDS.

Material	Hazards	Exposure Limit (2)	Signs and symptoms of exposure
Acetone	Flammable	1000 ppm-TWA	Inhalation of vapors irritates the respiratory tract. May cause coughing, dizziness, dullness, and headache.
Hexane	Flammable Irritant	500 ppm-TWA	Inhalation of vapors irritates the respiratory tract. Overexposure may cause lightheadedness, nausea, headache, and blurred vision. Vapors may cause irritation to the skin and eyes.
Methylene Chloride	Carcinogen Irritant	25 ppm-TWA 125 ppm-STEL	Causes irritation to respiratory tract. Has a strong narcotic effect with symptoms of mental confusion, light-headedness, fatigue, nausea, vomiting and headache. Causes irritation, redness and pain to the skin and eyes. Prolonged contact can cause burns. Liquid degreases the skin. May be absorbed through skin.
1 – Always add acid to water to prevent violent reactions.			
2 – Exposure limit refers to the OSHA regulatory exposure limit.			

6.0 Equipment and Supplies

6.1 Instrumentation

- Zymark Turbovap II concentrator
- Electronic Balance, capable of weighing to 0.01g.
- N-Evap Analytical Evaporator
- Fisher 8 Position Steam Bath
- MARS 5 microwave oven with temperature sensor (CEM Corp) capable of sensing

temperature within 2.5°C and adjusting microwave field output within 2 sec of sensing.
- MARS 40 position carousel (CEM Corp)
- Visiprep DL column holder

6.2 Supplies

- 10, 50 and 200 mL Concentrator tubes.
- Vials, - 1ml, 4ml, and 10ml with PTFE lined snapcaps.
- Glass syringes, 25ul, 50ul, 100ul, 500ul, 1000ul
- Pastuer pipets
- Glass Buchner funnels (stainless steel funnels may be substituted) – Fisher Scientific.
- 500 ml Kuderna-Danish (KD) flask
- Stainless steal spatulas
- Glass wool- Contaminant-free (silane treated or oven baked at 400^{O}C)
- 75mL Teflon Express vessels with stopper and cap (CEM Corp.)
- Glass fiber filter paper - #1 185mm (Whatman)
- Weigh boats
- Tongue depressors, wood

7.0 Reagents and Standards

Note: Each solvent lot is screened for contaminants before being used for analysis as detailed in TestAmerica Corporate Quality SOP No. CA-Q-S-001 (*Solvent & Acid Lot Testing & Approval*) and TestAmerica Edison SOP No. ED-GEN-023 (*Bulk Solvent Testing and Approval*).

7.1. Reagents

7.1.1 Organic free reagent water - Reagent water is defined as a water in which an interference is not observed at the MDL of each parameter of interest (ASTM Specification D1193, Type ii). At TestAmerica Edison this water is generated by the Barnstead/Thermolyne Water System (Model # D11991 Serial # 1191020210415).

7.1.2 Methylene Chloride, JT Baker Resi-analyzed, P/N 9254-03 (or equivalent)

7.1.3 Hexane – JT Baker Ultra-Resi 73513-42-5 or equivalent

7.1.4 Acetone, J.T. Baker Ultra- ResiAnalyzed, Catalog No. 9264-03 (or equivalent)

7.1.5 Nitrogen, high-purity

7.1.6 Construction Sand (Home Depot or Lowes), free from organic compounds. (Baked in the muffle furnace for four hours at 400^{O}C and stored in a dessicator prior to use.)

7.1.7 Sodium Sulfate Anhydrous Powder, Mallinckrodt MA8020-06 or equivalent (Must be baked in the muffle furnace for four hours at 400^{O}C and stored in a dessicator prior to use.)

7.2. Standards

7.2.1 Stock target analyte standard spiking solutions are purchased as prepared solutions (see table below). Stock solutions are diluted (in volumetric glassware) to a working concentration using methylene chloride ($MeCl_2$) hexane or Acetone as the diluent as indicated below below. Stock standards of similar quality from other suppliers may be substituted as required.

NOTE: Second sources (from separate lots) are used for quanitation standards and spiking.

Stock Standards (Methods 8015B,8015C and 8015D DRO & NJDEP OQA-QAM-025)				
Standard Mix	**Concentration**	**Source**	**Catalog #**	**Used in method(s):**
TPH Mix 3	1000 ppm	Sigma Aldrich	861394U	*NJDEP OQA-QAM-025
o-Terphenyl	10000 ppm	Sigma Aldrich	47580U	*SW846 8015B, 8015C &8015D DRO *NJDEP OQA-QAM-025
Chlorobenzene	10000 ppm	Sigma Aldrich	4 6860U	*NJ DEP OQA-QAM-025
Diesel Fuel #2	50000 ppm	Restek	31259	*SW846 8015B, 8015C &8015D DRO *NJDEP OQA-QAM-025

Stock Standards - Pesticides/PCBs* (Methods SW846 8081A/B and 8082/A)				
Standard Mix	**Concentration**	**Source**	**Catalog #**	**Used in method(s):**
Pesticide Surrogate Spike Mix ("Pest/PCB Surrogate")	10 ug/ml	Supelco	861275	SW846 8081/8082
Aroclor Spike Mix ("PCB Spike")	100 ug/ml	Supelco	861274	SW846 8082
SS CLP Organochlorine Pesticides Mix ("Pest Spike")	2000 ug/ml	Supelco	4S7426-U	SW846 8081
Chlordane (technical) ("Technical Chlordane Spike")	1000 ug/ml	Supelco	48065-U	SW846 8081
Toxaphene	1000 ug/ml	Supelco	48065-U	SW846 8081

*May be substituted with equivalent standards from alternate sources.

Components of Pesticide/PCB Standard Mixes (Methods SW846 8081A/B and 8082/A)			
Parameter	**Catalog Nos.**	**Lab Standard Name**	**Concentration (ug/ml)**
Decachlorobiphenyl (DCB)	Supelco – 4S8913	Pest/PCB Surrogate	10.0
2,4,5,6-Tetrachloro-m-xylene (TCMX)	Supelco – 4S8913	Pest/PCB Surrogate	10.0
Aroclor 1016	Supelco - 861274	PCB Spike	100

Components of Pesticide/PCB Standard Mixes (Methods SW846 8081A/B and 8082/A)			
Parameter	**Catalog Nos.**	**Lab Standard Name**	**Concentration (ug/ml)**
Aroclor 1260	Supelco - 861274	PCB Spike	100
Aldrin	Supelco - 4S7426-U	Pest Spike	2000
Alpha-BHC	Supelco - 4S7426-U	Pest Spike	2000
Alpha-Chlordane	Supelco - 4S7426-U	Pest Spike	2000
Beta-BHC	Supelco - 4S7426-U	Pest Spike	2000
Delta-BHC	Supelco - 4S7426-U	Pest Spike	2000
Dieldrin	Supelco - 4S7426-U	Pest Spike	2000
Endosulfan I (Alpha)	Supelco - 4S7426-U	Pest Spike	2000
Endosulfan II (Beta)	Supelco - 4S7426-U	Pest Spike	2000
Endosulfan Sulfate	Supelco - 4S7426-U	Pest Spike	2000
Endrin Aldehyde	Supelco - 4S7426-U	Pest Spike	2000
Endrin Ketone	Supelco - 4S7426-U	Pest Spike	2000
Endrin	Supelco - 4S7426-U	Pest Spike	2000
Gamma-BHC (Lindane)	Supelco - 4S7426-U	Pest Spike	2000
Gamma-Chlordane	Supelco - 4S7426-U	Pest Spike	2000
Heptachlor	Supelco - 4S7426-U	Pest Spike	2000
Heptachlor Epoxide (Isomer B)	Supelco - 4S7426-U	Pest Spike	2000
Methoxychlor	Supelco - 4S7426-U	Pest Spike	2000
4,4’-DDD	Supelco - 4S7426-U	Pest Spike	2000
4,4’DDE	Supelco - 4S7426-U	Pest Spike	2000
4,4’-DDT	Supelco - 4S7426-U	Pest Spike	2000
Chlordane (technical)	Supleco – 48065-U	Technical Chlordane Spike	1000
Toxaphene	Supelco 48103	Toxaphene Spike	1000

Stock Standards – BNAs (Method 8270x)			
Standard Mix	**Concentration (ug/ml)**	**Lab Vendor**	**Catalog #**
8270 List 1/ Std#1 MegaMix	500/1000/2000	RESTEK	567672
8270 List 1/ Std#5 N-Nitrosodiphenylamine	2000	RESTEK	567676
8270 List 1/ Std#2 Amines	2000	RESTEK	567673
8270 List 1/ Std#3 Benzoic Acid	2000	RESTEK	567674
Custom SVO Mix	2000	SPEX	SVO-TANJ-16-5
Bisphenol A	1000	SPEX	S-509-MC
8270 List 1/ Std#8	2000	RESTEK	568724
8270 Surrogate Standard	5000	RESTEK	567685
Aromatic Amines Custom Mix	2000	Supelco	21467482

Stock Standards – PCB Homologues (Method 680)			
Standard Mix	**Concentration (ug/ml)**	**Lab Vendor**	**Catalog #**
Retention Time Calibration Standard Mixture	Varies by component (100 to 200)	Ultra	CB-682MN
Concentration Calibration Standard Mix	Varies by component (50 to 250)	Ultra	CB-681MN
13C12-Decachlorobiphenyl Surrogate	40	Cambridge	EC-1410-3
Lindane13C6	100	Cambridge	CLM-1282-S

Components of PCB Homologue Mixes (Methods 680)			
Parameter	**Catalog Nos.**	**Lab Standard Name**	**Concentration (ug/ml)**
PCB-104	Ultra CB-682MN	Retention Time Calibration Standard Mixture	100
PCB-208	Ultra CB-682MN	Retention Time Calibration Standard Mixture	200
PCB-77	Ultra CB-682MN	Retention Time Calibration Standard Mixture	100
Total Nonachlorobiphenyls	Ultra CB-682MN	Retention Time Calibration Standard Mixture	200
DCB Decachlorobiphenyl	Ultra CB-681MN	Concentration Calibration Standard Mix	250
Total Dichlorobiphenyls	Ultra CB-681MN	Concentration Calibration Standard Mix	50
Total Heptachlorobiphenyls	Ultra CB-681MN	Concentration Calibration Standard Mix	150
Total Hexachlorobiphenyls	Ultra CB-681MN	Concentration Calibration Standard Mix	100
Total Monochlorobiphenyls	Ultra CB-681MN	Concentration Calibration Standard Mix	50
Total Octachlorobiphenyls	Ultra CB-681MN	Concentration Calibration Standard Mix	150
Total Pentachlorobiphenyls	Ultra CB-681MN	Concentration Calibration Standard Mix	100
Total Tetrachlorobiphenyls	Ultra CB-681MN	Concentration Calibration Standard Mix	100
Total Trichlorobiphenyls	Ultra CB-681MN	Concentration Calibration Standard Mix	50

7.2.2 Spiking Standards Prep for GC FID (DRO/QAM): The table below provides instruction in preparation of the various working spiking standards for use in this preparation method using the applicable standards listed in 7.2.1. Use volumetric glassware. Dilute to the volume marker with the indicated solvent.

Working Spiking Standards Preparation					
Standard Name	**Vendor/ Cat #**	**Initial Conc. (ug/ml)**	**Vol. of Standard (ml)**	**Vol. of solvent mls (solvent)**	**Final Conc. (ug/ml)**
DRO Spike/ QAM-025 LCS Spike Diesel Fuel #2	Restek/31259	50000	4	100 ($MeCl_2$)	2000
DRO Surrogate -o-terphenyl	Supelco/47580U	10000	1.0	500 (Acetone)	20
QAM-025 MS/MSD Spike TPH Mix 3	Sigma/861394-U	1000	1.47	10 ($MeCl_2$)	147
QAM-025 Surrogate -Chlorobenzene -o-terphenyl	Supelco/46860-U Supelco/47580-U	10000 10000	1.0 1.0	500 500	20 20

7.2.3 Spiking Standards Prep for GC ECD(8081/8082):

7.2.3.1 All standard stock solutions are diluted to the working concentrations with hexane or acetone (as indicated) using Class A volumetric glassware. **Note:** septa on all surrogate and spike vials are to be replaced immediately after use. Additionally, all surrogate and spike vials are to be returned to the standards refrigerator immediately after use.

7.2.3.2 Pesticide Spiking Standard (LCS/MS/MSD): The Pesticide Mix BS/MS/MSD solution with 20 single component pesticides is prepared at a final concentration of 20 ug/ml by diluting 500 ul (0.50 ml) of the 2000 ppm Pest Spike stock solution (Supelco 4S7426-U, see tables in Section 7.2.1) to a 50 ml final volume with acetone. For spiking instructions refer to Section 10.

7.2.3.3 PCB Spiking Standard (LCS/MS/MSD): The PCB spiking solution (Supelco – 861274, see Tables 1 and 2) is a custom mix received from the vendor at its working concentration of 100 ug/ml in Acetone. For spiking instructions refer to Section 10.

7.2.3.4 Technical Chlordane Spiking Standard (LCS/MS/MSD): A technical Chlordane spiking solution is prepared at a final concentration of 100 ug/ml by diluting 1 ml of the 1000 ug/ml Technical Chlordane Spike solution (Supelco 48065-U, see tables in Section 7.2.1) to a 10 ml final volume using acetone. For spiking instructions refer to Section 10.

7.2.3.5 Toxaphene Spiking Standard (LCS/MS/MSD): A Toxaphene spiking solution is prepared at a final concentration of 100 ug/ml by diluting 1 ml of the 1000 ug/ml Toxaphene Spike solution (Supelco 48103, see tables in

Section 7.2.1) to a final volume 10 mL.using acetone. For spiking instructions refer to Section 10.

7.2.3.6 Pesticide/PCB Surrogate Spiking Standard: The Pesticide/PCB surrogate spiking solution is a concentrated 10 ml solution of DCB/TCMX at a concentration of 10 ug/ml. (Supelco Catalog # 861275, see tables in Section 7.2.1). The concentrate is used without further dilution prior to the spiking of samples.

7.2.4 Spiking Standard for BNA: For use in spiking Soils samples prepared for BNA analysis by SW846 Method 8270. Prepare the spiking solution for MS/MSD/Blank Spike (LCS) as detailed in the following table using the applicable standards listed in 7.2.1. Use volumetric glassware. Dilute to the volume marker with methanol

Standard Name	Concentration	Volume of Standard added to final volume of 200ml (solvent)	Final Concentration
8270 List 1/ Std#1 MegaMix	500/1000/2000pmm	20 ml (methanol)	50/100/200 ppm
8270 List 1/ Std#5 N-Nitrosodiphenylamine	2000ppm	10 ml (methanol)	100 ppm
8270 List 1/ Std#2 Amines	2000ppm	10 ml (methanol)	100 ppm
8270 List 1/ Std#3 Benzoic Acid	2000ppm	10 ml (methanol)	100 ppm
Custom SVO Mix	2000ppm	10 ml (methanol)	100 ppm
Bisphenol A	1000ppm	10ml (methanol)	50ppm
8270 List 1/ Std#8	2000ppm	20ul (methanol) 100ul(methanol)	Used neat for LVI Used neat for non-LVI
Aromatic Amines Custom Mix**	2000ppm	10 ml (methylene chloride)	100 ppm

** *As needed based on clients requirement.*

7.2.4.1 BNA Surrogate Standard Spiking Solution: For use in spiking all blanks, samples and associated QC prior to extraction. Prepare as detailed in the following table using the applicable standards listed in 7.2.1. Use volumetric glassware. Dilute to the volume marker with methanol.

Standard Name	Concentration	Volume of Standard added to final volume of 1000 ml methanol	Final Concentration
8270 Surrogate Standard	5000 ppm	20 ml	100 m

7.2.5 Spiking Standard for PCB Homologues (EPA 680): For use in spiking LCS and MS/MSD prior to extraction. Prepare by adding 1.0ml

of the Retention Time Calibration Standard and 1.0ml of the Concentration Calibration Standard Mix to hexane in volumetric glassware and dilute to final volume of 100ml. 1.0mL of this solution is spiked into LCS/MS/MSD prior to extraction.

7.2.5.1 PCB Homologue Surrogate Standard Spiking Solution (EPA 680): (2.0ug/mL and 2.5ug/mL) – Add 3.125mL of $^{13}C_{12}$-Decachlorobiphenyl and 1.0mL of Lindane13C6 and dilute to a final volume of 50mL in hexane in volumetric glassware. 1.0mL of this solution is spiked into all blanks, samples and QC items prior to extraction.

7.2.6 The preparation of all standards must be documented in a standard preparation logbook or in the TALS reagent module. Information such as standard supplier, lot number, original concentration, and a description of how standard was prepared are required along with a laboratory lot number, analyst's initials, date prepared and verification signature. Standards must be made every 6 months or sooner if signs of degradation appear.

8.0 Sample Collection, Preservation, Shipment and Storage

Sample container, preservation techniques and holding times may vary and are dependent on sample matrix, method of choice, regulatory compliance, and/or specific contract or client requests. Listed below are the holding times and the references that include preservation requirements.

Matrix	Sample Container	Min. Sample Size	Preservation	Holding Time [1]	Reference
Soils	Glass	4 oz	Cool 4 ± 2°C	14 Days	a) NJDEP-OQA-QAM-025; b)SW846 Method 8000

Samples are collected in amber jars with Teflon lined caps.
[1]All sample extracts must be analyzed within forty (40) days of extraction.

9.0 Quality Control

9.1. Sample QC – The following quality control samples are prepared with each batch of samples.

QC Analysis	Frequency	Acceptance Criteria	Corrective Action
Laboratory Control Sample (LCS/LCSD)[1]	1 in 20 or fewer samples	See applicable analytical SOPs	See applicable analytical SOPs for control criteria
Method Blank (MB)	1 in 20 or fewer samples	See applicable analytical SOPs	See applicable analytical SOPs for control criteria

QC Analysis	Frequency	Acceptance Criteria	Corrective Action
MS\MSD[2] (Client specified)	1 in 20 or fewer samples	See applicable analytical SOPs	See applicable analytical SOPs for control criteria
Surrogates[3]	All Samples including QC	See applicable analytical SOPs	See applicable analytical SOPs for control criteria

[1] LCS Duplicate (LCSD) is performed only when insufficient sample is available for the MS/MSD or when requested by the client/project/contract except for NJDEP EPH 10/09 where LCS/LCSD is required.
[2] The sample selections for MS/MSD are randomly selected, unless specifically requested by a client. For NJDEP EPH 10/09, only MS is required (MSD for NJDEP EPH 10/09 is by client request only).
[3] Analytical and QC samples (MB, LCS, MS/MSD)
[4] Statistical control limits are updated annually and are updated into LIMS.

9.2. Instrument QC

N/A

10.0 Procedure

10.1. Sample Preparation

10.1.1 Decant and discard any water layer on a sediment sample. Discard any foreign objects such as rocks, leaves and sticks. Mix sample thoroughly with wooden tongue depressor prior to subsampling

10.1.2 The initial amount and treatment of soil samples is method dependent:

- For QAM-025, DRO (8015): 15 grams of soil is weighed into a 75mL Teflon microwave vessel (the weight is recorded to two significant figures). Where practical samples should be air dried and ground prior to extraction. Drying should be performed in a hood to avoid contamination.
- For 8270/ 8081/8082: 15 grams of soil is weighed into an aluminum weighing dish (the weight is recorded to two significant figures). 15 mls of acetone is added to the dish and the soil/acetone mixture is stirred well with a wooden tongue depressor to create a slurry. The soil/acetone slurry is transferred to a 75 ml Teflon microwave vessel. The dish is then rinsed with 15 ml of methylene chloride which is also added to the microwave vessel.
- For EPA 680: 30 grams of soil is weighed into a 75mL Teflon microwave vessel (the weight is recorded to two significant figures).

10.1.3 For laboratory blanks and control spikes, an equal portion of clean sand is prepped along with samples.

10.1.4 All samples, blanks, and spikes are fortified with an appropriate amount of the applicable method surrogates. All matrix spikes and lab

control spikes are fortified with an appropriate amount of the applicable method spiking solution. See Attachment 1 for general spiking instructions. (Refer to Section 7.2 for information on preparation of spiking solutions):

- PCBs (8082): add 50 ul of the PCB Spike solution to each LCS and designated MS/MSD;
- Pesticides (total, 8081): 100 ul of the Pest Spike solution to each LCS and designated MS/MSD;
- Technical Chlordane: 100 ul of the Technical Chlordane Spike mix to each LCS and designated MS/MDS;
- Toxaphene: 100 ul of the Toxaphene Spike mix to each LCS and designated MS/MSD;
- BNA (8270): 500ul of spike/Surrogate solution to each LCS and designated MS/MSD);
- PCB Homolgues (EPA 680): 1.0 ml each of Spiking solution (see Sec.7.2.5) and Surrogate solution (see Sec. 7.2.5.1) to each LCS and designated MS/MSD.

NOTE: When spiking the samples, it is critical to remove all bubbles from the syringe.

10.1.5 Add the following solvent to each sample vessel depending upon the fraction (**note**: for Pesticides and PCBs by methods 8081 and 8082 the solvent addition is addressed in Section 10.1.2 above)

- 30mL of methylene chloride for DRO (8015)/QAM-025;
- 30 mL of Methylene Chloride/Acetone blend (50:50) for BNA (8270)

30 mL of Hexane for PCB homologues by EPA 680

10.1.6 A stopper is placed over the opening along with the vessel cap. The vessel is then tightened using the MARS capping wrench. This capping wrench tightens the cap to the appropriate torque.

10.1.7 The sample vessels are placed into the 40-position carousel. Each position has an insulator sleeve that the vessel slides into – slide the vessel into the appropriate sleeve – and press firmly down to ensure a complete connection.

10.1.8 Place the carousel (with samples and vessels) into the microwave unit.

Start Run: (load method – using key pad on face of unit)
Press: Home

Press Select: load method
Press Select: from user directory
Press Select: 3546 Microwave – Xpress method
Press Start.

Microwave Operating Parameters				
Power Max	**Power %**	**Oven Ramp**	**Degrees Celsius**	**Hold Time**
1200W	100	10.0 minutes	110	10.0 minutes
1600W[1]	100	20.0 minutes	115	10.0 minutes

[1] For pesticide/PCB and BNA samples only

10.1.9 A 5-minute cool down will close out the run.

10.1.10 Remove vessels from carousel and allow cooling (either at room temperature or in a cold water bath).

10.1.11 When cool down is complete, remove carousel from microwave.

10.1.12 Pre-wash a funnel filled with sodium sulfate with three successive washings of 30 ml acetone. Pour the extract carefully through the sodium sulfate funnel (to remove any moisture) into the 200mL concentrator tube. Rinse the 75mL vessel with 10 mL methylene chloride three times to ensure quantitative transfer.

10.1.13 Set the water temperature in the concentrator to 35°C and adjust the pressure to around 5psi of nitrogen.

10.1.14 Place the tube into the apparatus and slowly raise the pressure to 20psi. If splashing occurs, start with a lower pressure and raise it when the solvent gets lower.

10.1.15 Concentrate to just under the 1mL mark on the nipple of the tube. Remove from the bath and transfer to an auto sampler vial with a glass Pasteur pipette..

10.1.16 Add a few drops of clean methylene chloride to the tube, rinsing the narrow part and add to the vial to adjust to 1mL or other method specific final volume. If solvent exchange to hexane is required, add approximately 30mL (mix well) and return to concentrator set at 35°C. See Attachment 2.

10.1.17 Total concentration time should be approximately 20 minutes for methylene chloride. If a sample takes a longer than usual amount of time to concentrate or if the extract becomes viscous the final volume should be adjusted to a larger volume and documented.

10.1.18 The extract is now ready for the appropriate clean up and or analysis.

10.2. Calibration

10.2.1 The balance calibration is checked daily prior to use. Refer to TestAmerica Edison SOPs ED-GEN-010, *Calibration of Analytical Balances*, current revision and ED-GEN-006, *Standard Operating Procedure for Preventive Maintenance and Calibration Procedures for All Analytical Instruments and Ancillary Equipment*, current revision.

11.0 Calculations / Data Reduction

N/A

12.0 Method Performance

12.1. Method Detection Limit Study (MDL)

The method detection limit (MDL) is the lowest concentration that can be detected for a given analytical method and sample matrix with 99% confidence that the analyte is present. The MDL is determined according to the laboratory's MDL procedure in Section 19 (Test Methods and Method Validation) of TestAmerica Edison's Quality Assurance Manual (ED-QA-LQM). MDLs reflect a calculated (statistical) value determined under ideal laboratory conditions in a clean matrix, and may not be achievable in all environmental matrices. The laboratory maintains MDL studies for analyses performed; these are verified at least annually unless method requirements require a greater frequency.

12.2. Demonstration of Capabilities

For demonstration of capability procedure refer to Section 19 in the most current revision of TestAmerica Edison's Quality Assurance Manual (ED-QA-LQM).

12.3. Training Requirements

Refer to TestAmerica SOP No. ED-GEN-022, *Training*, for the laboratory's training program.

13.0 Pollution Control

13.1. It is TestAmerica's policy to evaluate each method and look for opportunities to minimize waste generated (i.e., examine recycling options, ordering chemicals based on quantity needed, preparation of reagents based on anticipated usage and reagent stability). Employees must abide by the policies in Section 13 of the Corporate Environmental Health and Safety Manual (CW-E-M-001) for "Waste Management and Pollution Prevention."

14.0 Waste Management

14.1 Waste management practices are conducted consistent with all applicable rules and regulations. Excess reagents, samples and method process wastes are disposed of in an accepted manner. Waste description rules and land disposal restrictions are followed. Waste disposal procedures are incorporated by reference to TestAmerica Edison SOPs No. ED-SPM-008 (*Laboratory Waste Disposal Procedures, current revision).* The following waste streams are produced when this method is carried out:

- *Mixed Solvent Waste*: Mixed solvent waste is collected in a small beaker inside the bench top hood. This waste is then transferred into the satellite accumulation container in the Organic Prep. Lab. on a daily basis. This material is transferred into 5 gallon solvent cans as satellite accumulation. These cans are emptied every 24 hours into a steel drum in the waste room. This drum is kept in the walk in hood until it is full. The full drum is then removed from the hood and placed on secondary containment in the waste room.

  Teris Profile Number: 50016624
  Onyx Profile WIP Number: 545240

- *Auto sampler vials and expired standards*: These vials are collected in satellite accumulation within the instrument laboratory. The vials are then placed into a 55 steel open top drum in the waste room. When the drums are full, the drum will be collected by the waste vendor for disposal. This waste is treated for incineration.

  Teris Profile Number: 50016652
  Onyx Profile WIP Number: 282493

15.0 References / Cross-References

15.1 United States Environmental Protection Agency, “Method SW3546, Microwave Extraction”, Test Methods for Evaluating Solid Wastes, SW846 Laboratory Manual, Physical/Chemical Methods, February 2007.

15.2 United States Environmental Protection Agency, “Method SW8015B, Non-Halogenated Organics, Test Methods for Evaluating Solid Wastes, SW846 Third Edition,Volume 1B: Laboratory Manual, Physical/Chemical Methods, Revision 3, December 1996.

15.3 United States Environmental Protection Agency, Method SW8015C, Test Methods for Evaluating Solid Wastes, SW846 Third Edition, Volume 1B: Laboratory Manual, Physical/Chemical Methods, Revision 3, February 2007.

15.4 United States Environmental Protection Agency, Method SW8015D, Test Methods for Evaluating Solid Wastes, SW846 Third Edition, Volume 1B: Laboratory Manual, Physical/Chemical Methods, Revision 4, June 2003.

15.5 NJDEP Method No. OQA-QAM-025, “Quantitation of Semivolatile Petroleum Products in Water, Soil, Sediment and Sludge” (current revision)

15.6 United States Environmental Protection Agency, “Method 8081A, Organochlorine Pesticide by Gas Chromatography”, Test Methods for Evaluating Solid Wastes, SW846 Third Edition, Volume 1B: Laboratory Manual, Physical/Chemical Methods, Revision 3, December 1996.

15.7 United States Environmental Protection Agency, “Method 8082, Polychlorinated Biphenyls PCBs) by Gas Chromatography”, Test Methods for Evaluating Solid Wastes, SW846 Third Edition, Volume 1B: Laboratory Manual, Physical/Chemical Methods, Revision 3, December 1996.

15.8 United States Environmental Protection Agency, “Method 8081B, Organochlorine Pesticide by Gas Chromatography”, Test Methods for Evaluating Solid Wastes, SW846 Third Edition, Volume 1B: Laboratory Manual, Physical/Chemical Methods, Revision 2, February 2007.

15.9 United States Environmental Protection Agency, “Method 8082A, Organochlorine Pesticide by Gas Chromatography”, Test Methods for Evaluating Solid Wastes, SW846 Third Edition, Volume 1B: Laboratory Manual, Physical/Chemical Methods, Revision 1, February 2007.

15.10 United States Environmental Protection Agency, “Method SW8270C, Semivolatile Organic Compounds by Gas Chromatography/Mass Spectrometry”, Test Methods for Evaluating Solid Wastes, SW846 Third Edition, Volume 1B: Laboratory Manual, Physical/Chemical Methods, Revision 3, December 1996.

15.11 United States Environmental Protection Agency, “Method SW8270D, Semivolatile Organic Compounds by Gas Chromatography/Mass Spectrometry”, Test Methods for Evaluating Solid Wastes, SW846 Third Edition, Volume 1B: Laboratory Manual, Physical/Chemical Methods, Revision 4, February 2007.

15.12 TestAmerica Edison SOP No. ED-GCS-008, *Preparation and Analysis of Diesel Range Organics (DRO C10-C28) in Soil and Water Samples by SW846 Methods 3510C (Separatory Funnel), 3541 (Automated Soxhlet Extraction) and 8015B(GC/FID),* current revision.

15.13 TestAmerica Edison SOP No. ED-GCS-009, *Preparation and Analysis of Diesel Range Organics (DRO C10-C44) in Soil and Water Samples by SW846 Methods 3510C (Sep Funnel), 3541 (Automated Soxhlet) and 8015B (GC/FID),* current revision.

15.14 TestAmerica Edison SOP No. ED-GCS-011,*NJDEP OQA-QAM-025 Quantitation of Semivolatile Petroleum Products in Water Soil Sediment and Sludge,* current revision.

15.15 TestAmerica Edison SOP No. ED-GCS-003, *Analysis of Organochlorine Pesticides by Gas Chromatograph, SW846 Method 8081A*, current revision.

15.16 TestAmerica Edison SOP No. ED-GCS-004, *Analysis of Polychlorinated Biphenyls by Gas Chromatograph, SW846 Method 8082*, current revision

15.17 TestAmerica Edison SOP No. ED-GCS-016, *Analysis of Organochlorine Pesticides by Gas Chromatograph, SW846 Method 8081B*, current revision.

15.18 TestAmerica Edison SOP No. ED-GCS-017, *Analysis of Polychlorinated Biphenyls by Gas Chromatograph, SW846 Method 8082A*, current revision

15.19 TestAmerica Edison SOP No. ED-MSS-010, *Determination of PCBs in Water and Soil/Sediment by GC/MS, EPA Method 680,* current revision.

15.20 TestAmerica Edison Document No. ED-QA-LQM, *Laboratory Quality Manual*, current revision.

15.21 TestAmerica Environmental Health and Safety Manual, CW-E-M-001.

15.22 TestAmerica Corporate Quality SOP No. CA-Q-S-001, *Solvent & Acid Lot Testing& Approval*, current revision.

15.23 TestAmerica Edison SOP No. ED-GEN-023, *Bulk Solvent Testing and Approval*, current revision.

15.24 TestAmerica Edison SOP No. ED-GEN-013, *Glassware Cleaning,* current revision

15.25 TestAmerica Edison SOP No. ED-GEN-022, *Training,* current revision

15.26 TestAmerica Edison SOP No. ED-SPM-008, *Laboratory Waste Disposal Procedures,* current revision.

15.27 TestAmerica Edison SOP No. ED-GEN-006, *Standard Operating Procedure for Preventive Maintenance and Calibration Procedures for All Analytical Instruments and Ancillary Equipment,* current revision.

15.28 TestAmerica Edison SOP No. ED-GEN-010, *Calibration of Analytical Balances,* current revision.

15.29 TestAmerica Edison SOP No. ED-GEN-007, *Subsampling*, current revision.

15.30 Operation Manuals for the Zymark Turbo Vap II Concentrator.

15.31 Operating manual for the MARS 5 Microwave unit.

16.0 Method Modifications:

N/A

17.0 Attachments

Attachment 1: Spiking information
Attachment 2: Specific Extraction Conditions

18.0 Revision History

Rev 11, March 12, 2018

- Updated Section 10.1 to revise procedures for the microwave extraction of solids for subsequent analysis by EPA methods 8081, 8082 and 8270.
- Updated to remove all references to addition of sodium sulfate to samples prior to microwave extraction.
- Fixed section numbering and formatting errors.

Rev 10, May 16, 2017

- Updated throughout to add procedures for microwave extractions of solids for subsequent analysis by EPA Method 680 (PCBs by GC/MS).

Rev 9, Dec 15, 2016

- Updated throughout to remove procedures for microwave extraction of solids for NJ EPH method. Those procedures are fully covered in TestAmerica Edison SOP ED-GCS-012, Preparation and Analysis of Extractable Petroleum Hydrocarbons (EPH) in Solid and Water Samples using NJDEP EPH Method 10/08, August 2010 (Rev. 3): Analysis of Extractable Petroleum Hydrocarbon Compounds (EPH) in Aqueous and Soil/Sediment/Sludge Matrices (current revision)
- Added following text to Section 1.2: The microwave prep for NJDEP Extractable Petroleum Hydrocarbons (EPH) (10/08), current revision, is addressed separately in TestAmerica Edison SOP ED-GCS-012 (current revision).
- All references to NJDEP EPH methodology removed.

Rev 8, Jan 22, 2015

- Updated to include procedures for microwave extraction of solids for analysis my SW846 Methods 8270C and 8270D (Semivolatile Organic Compounds by Gas Chromatography/ Mass Spectrometry (GC/MS). Specifically
    - Added 8270 method references and SOP numbers to Sections 1.1 and 15.0
    - Added a table to Section 7.2.1 detailing the BNA standard mixes.
    - Added new Section 7.2.4 detailing the preparation of BNA spiking standards

- Added Section 7.2.4.1 detailing the preparation of BNA surrogate spiking standards.
- Added details of 8270 spiking and extraction to Section 10.0 as appropriate.
- Added BNA Spiking details to Attachments 1 and 2. COAs for BNA spiking not included in SOP but are on-file in the department.

Rev 7, June 14, 2013

- Section 1.1: updated the DRO method references to include SW8015C and SW8015D. Also update the SOP references for the DRO analytical methods.
- Section 7.2.1: updated stock standards table to include SW8015C and 8015D. Revised source for o-Terphenyl stock. Removed TPH Mix 1 from list. Removed 8015x DRO from the list of methods for which TPH Mix 3 is used. Added 8015x DRO to the list of methods for which Diesel Fuel #2 is used.
- Section 7.2.2.1: Revised DRO surrogate spike source and prep instructions. Revised DRO and QAM-025 spiking standard source and prep instructions.
- Section 15.0: References: added references for methods SW8015C and 8015D.
- Attachment 1: updated spiking information for DRO.

Rev 6, October 10, 2012

- Throughout document:: Revised LQM reference sections to reflect the most current revision
- Throughout document: updated as detailed below to include prep instructions for solid samples undergoing analysis by SW846 8081A/B and 8082/A.
- Section 1.1: added references to the 8081/8082 SOPs: TestAmerica Edison SOPs No.ED-GCS-003, ED-GCS-004, ED-GCS-016 and ED-GCS-017.
- Section 6.2: added weigh boats and wooden tongue depressors.
- Section 7.2.1: added acetone as a diluent and added tables detailing the Pestcide and PCB standards.
- Section 7.2.2: added “GCFID (DRO/EPH/QAM)” to section header.
- Section 7.2.3: added instructions for prep of Pest/PCB spiking standards.
- Section10.1.4: Added Pest/PCB spiking instructions.
- Section 10.1.5: added instructions for addition of extraction solvent (Methylene Chloride:Acetone for Pest/PCB).
- Section 10.1.8: updated table to include Microwave Parameters for Pest/PCB prep.
- Section 10.1.12: added a methylene chloride rinse.

- Section 15: added method and SOP references for SW846 8081A/8081B and SW846 8082/8082A.
- Attachments: updated to include Pest/PCB information.

Rev 5, August 26, 2011

- Section .6.2: added vendor and part number details for silica gel columns (Phenomenex Part No:8L-S012-JCH)
- Section 10.1.2: Added details for the pre-washing of sodium sulfate funnels with three successive 30 ml volumes of acetone. Added details for the rinsing of the 75 ml vessel with three successive volumes of methylene chloride.

Rev 4, April 21, 2010

- Section 7.2.1: Corrected the footnotes in the tables 'Stock Standards – Aliphatics (Method NJDEP EPH 10/08)' and 'Stock Standards-Aromatics (Method NDJEP EPH 10.08)'.
- Attachment 3: Added this attachment which consists of select certificates of analysis for Method NJDEP EPH 10/08.
- Section 7.2.2.1: Table 'Working Spiking Standards Preparation'" replaced asterisks (*) with dashes (-) in order to clarify that these are simply bullets rather than referencing footnotes.

Rev 3, April 19, 2010

- Section 7.2.2.1: added procedure for preparation of a 500 ppm Aliphatic Neat Spike Mix.
- Section 7.2.2: Spiking Standards Prep: revised the procedure for prep of the EPH Aliphatic Spike in the 'Working Spiking Standards Preparation' table as follows:
    - Replaced 'MA EPH-n-Hydrocarbons (Absolute/91488) with the 'Florida TRPH Standard (Restek/31266)'
    - Included used of the intermediate 500 Aliphatic Neat Spike Mix.

Rev 2, February 9, 2009

- Section 3.0, Definitions – updated to reflect current location of definitions (Appendix 2 of Lab Quality Manual
- Section 5.2, Primary Materials Used – added 'hexane' to table (used in the NJDEP EPH method).
- Section 7.2.1: added stock standards associated with NJDEP EPH 10/08 method.
- Section 7.2.2: Spiking Standards Prep: updated 'Working Spike Standards' table to include revised NJDEP EPH 10/09 spiking standards; revised other as required.
- Section 9.1, Sample QC: updated with revise NJDEP EPH 10/09 QC requirements.
- Section 10.1, Sample Preparation: revised initial sample weight to 15 grams.
- Section 10.1.8: added Microwave Operating Parameters for the NJDEP EPH 10/09 method.
- Section 10.2.1: changed the specifications of the silica column.

- Section 10.2.5: changed the volume of the hexane rinsate.
- Section 10.2.7: changed the volume of the methylene chloride rinsate.
- Section 10.2.11: Added text describing procedures for NJDEP EPH 10/09 Demonstration of Fractionation Capability.
- Section 14.1 and Section 15.0 : Removed reference to TestAmerica Edison SOP No. ED-SPM-007 (*Disposal of Samples and Associated Laboratory Waste, current revision)* (SOP is now retired).
- Section 15.0: updated references to NJDEP EPH method as well as TestAmerica Edison SOP for NJDEP EPH analysis.
- Section 17.0, Attachments: corrected descriptions of attachments.

Rev 1, June 29, 2009

- Section 1.0, Scope and Application – corrected typo in the temperature range 100 – 115^{o}C
- Section 5.2, Primary Materials Used – removed ‘sodium hydroxide’ and ‘sulfuric acid’ from table as they are not used in this method.
- Section 6.1, Instrumentation – added Visiprep DL column holder to the instrumentation list
- Section 6.2, Supplies – added 5 g silica gel column (20 ml capacity)
- Sections 7.2.1, 7.2.2, Attachment 1 and Attachment 2: corrected a typo in the abbreviation for Methylene Chloride ($MeCl_2$).
- Section 10.1.1.2 - sentence correction to show actual process and typo error correction. Phrase ‘may also’ changed to ‘shall’.
- Section 10.1.12 – replaced the word ‘salt’ with ‘Sodium Sulfate’ to show actual reagent being used.
- Section 10.1.17 – typo error correction to ’20 minutes’
- Section 10.2.5 – Added the following text to the end of the section: “The amount on hexane will need to be adjusted with every lot of silica columns. The Hexane amount should never exceed 20 mls.”
- Corrected carbon ranges for the reference DRO SOPs throughout the document.

Rev 0, March 27, 2009: New

Attachment 1

Spiking Information – Microwave Extraction

Method	Surrogate Amount (all samples and QC)	Spike Amount (LCS/MS/ MSD)	Final volume	Initial Solvent	Final Solvent
DRO (8015)	1 mL	1 mL	1 mL	$MeCl_2$	$MeCl_2$
QAM-025	1 mL	1 mL	1 mL	$MeCl_2$	$MeCl_2$
Pest/PCB (8081/8082)	0.05 mL	0.1/0.05 mL	10 mL	MeCl2/Acetone (50:50)	Hexane Exchange from MeCl2/Acetone
BNA (8270)	500uL	500uL	1 mL	MeCl2/Acetone (50:50)	$MeCl_2$
PCB Homologues (EPA 680)	1.0	1.0	1 mL	Hexane	Hexane

Attachment 2

Specific Extraction Conditions

Method	Initial extraction pH	Secondary extraction pH	Final Solvent	Volume of extract required for cleanup(mL)	Final extract volume for analysis (mL)
DRO (8015)	as received	NA	$MeCL_2$	NA	1.0
QAM-025	as received	NA	$MeCL_2$	NA	1.0
Pest/PCB (8081/8082)	as received	NA	Hexane	3	10.0
BNA (8270)	as received	NA	$MeCL_2$	NA	1.0
PCBs (EPA 680)	as received	NA	Hexane	NA	1.0

Title: SW846 Method SW8081B, Analysis of Organochlorine Pesticides by Gas Chromatography

Approvals (Signature/Date):

Catalina Dalangin SVOA GC Manager	10/17/2022 Date	Dan Helfrich Health & Safety Manager	10/17/2022 Date
Carl Armbruster Quality Assurance Manager	10/17/2022 Date	Mark Acierno Laboratory Director	10/17/2022 Date
Sylvanus Klusey Operations Manager - Organics	10/17/2022 Date		

1.0 Scope and Application

1.1 Analytes, Matrix(s), and Reporting Limits

Method 8081B is used to determine the concentrations of various organochlorine pesticides in extracts from solid and liquid matrices, using dual fused-silica, open-tubular, capillary columns with electron capture detectors (ECD). The list of analytes and their corresponding reporting limits are as follows:

Parameter		Soil	Water	Leachate
	CAS Registry No.	Reporting Limits (ug/Kg)	Reporting Limits (ug/L)	Reporting Limits (mg/L)
Aldrin	309-00-2	6.7	0.02	---
Alpha-BHC	319-84-6	2.0	0.02	---
Beta-BHC	319-85-7	2.0	0.02	---
Delta-BHC	319-86-8	2.0	0.02	---
Gamma-BHC (Lindane)	58-89-9	6.7	0.02	0.00050
Chlordane	57-74-9	67	0.50	0.0050
4,4' –DDD	72-54-8	6.7	0.02	---
4,4' –DDE	72-55-9	6.7	0.02	---
4.4' –DDT	50-29-3	6.7	0.02	---
Dieldrin	60-57-1	2.0	0.02	---
Endosulfan I	959-98-8	6.7	0.02	---
Endosulfan II	33213-65-9	6.7	0.02	---
Endosulfan sulfate	1031-07-8	6.7	0.02	---
Endrin	72-20-8	6.7	0.02	0.00050
Endrin aldehyde	7421-93-4	6.7	0.02	---
Endrin ketone	53494-70-5	6.7	0.02	---
Heptachlor	76-44-8	6.7	0.02	0.00050
Heptachlor epoxide	1024-57-3	6.7	0.02	0.00050
Methoxychlor	72-42-5	6.7	0.02	0.00050
Toxaphene	8001-35-2	67	.50	0.0050
Gamma- Chlordane	5103-74-2	6.7	0.02	---
Alpha-Chlordane	5103-71-9	6.7	0.02	---
Mirex	2385-85-5	6.7	0.010	---

The most current MDLs and RLs for this method can be found in the active Eurofins LIMS (TALS) SW846 8081B Method Limit Group (MLG) database.

1.2 On occasion clients may request modifications to this SOP. These modifications are handled following the procedures outlined in Section 7 (*Review of Work Request*) and Section 19 (*Test Methods and Method Validation)* of Eurofins Edison's Quality Assurance Manual (ED-QA-LQM).

2.0 Summary of Method

2.1. Samples undergo a preparation step prior to analysis by SW846 Method 8081B. A measured volume or weight of sample (15g for soil, 1 g for waste, 250 ml for water, and 250 ml for TCLP) is extracted using the appropriate matrix-specific sample extraction technique. (Reference the applicable Organic Sample Prep SOPs listed below). The effective final volume is usually between 5 and 20 ml in hexane.

2.1.1. Aqueous samples are extracted using SW846 Method 3510C (SOP No. ED-ORP-014: *Extraction of Pesticides and PCBs in Water by Separatory Funnel*).

2.1.2. Solid samples are extracted using SW846 Method 3550B: Sonication (*SOP No. ED-ORP-018: Extraction of Pesticides/PCBs in Soil Using Low-Level Extraction*) or SW846 Method 3546: Microwave (SOP No. ED-ORP-044: *Procedure for the Microwave Extraction of Solids,SW846 3546).*

2.1.3. Organic liquids are prepared using SW846 Method 3580A (*SOP No. ED-ORP-019: Waste Dilution for Pesticides and PCBs*).

2.1.4. Extract cleanup steps are employed depending on the nature of the matrix interferences. Suggested cleanups include SW846 Method 3620B (*SOP No. ED-ORP-020: Florisil Cleanup for Pesticide/PCB Sample Extracts*) and SW846 Method 3660B (*SOP No. ED-ORP-021: The Removal of Elemental Sulfur from Pesticide/PCB Sample Extracts*).

2.2. After cleanup, a small aliquot of the extract is injected into a gas chromatograph (GC) equipped with a capillary column. The GC is temperature programmed to separate the compounds which were recovered during the extraction step by boiling point. The effluent of the gas chromatograph is interfaced to an Electron Capture Detector (ECD) which is used to detect the compounds eluting from the GC.. Quantitation is accomplished by comparing the area response of each target analyte relative to an internal standard established through a five-point calibration (six points for second order regression). Specific calibration and quality control steps are included in the method that must be performed and must meet the specifications of SW846 Method 8081B.

2.3. For pesticide analysis a system performance check (DDT/Endrin breakdown) and a calibration verification standard must be run prior to analysis. Failure of either will generally indicate the need for injection port/column maintenance and/or recalibration.

2.4. Samples are analyzed after all the necessary checks have been performed. Samples analyzed for pesticides require an additional post analysis Quant Report to be printed and attached to the chromatographic report.

2.5. All samples are then manually reviewed. Secondary column confirmation of target compounds and quantitation are conducted by the analyst as required.

3.0 Definitions

For a complete list of definitions refer to Appendix 5 in the most current revision of Eurofins Edison's Quality Assurance Manual (ED-QA-LQM).

4.0 Interferences

4.1. Interferences by phthalate esters introduced during sample preparation can pose a major problem in pesticide determinations.

4.1.1. Interferences from phthalate esters can be minimized by avoiding contact with any plastic materials and checking all solvents and reagents for phthalate contamination. Exhaustive cleanup of solvents, reagents and glassware may be required to eliminate background phthalate ester contamination.

4.2. The presence of elemental sulfur will result in broad peaks that interfere with the detection of early-eluting organochlorine pesticides. Sulfur contamination should be expected with sediment samples. If sulfur is encountered, employ the sulfur removal procedures detailed in SW846 Method 3660B (*SOP No. ED-ORP-021: The Removal of Elemental Sulfur from Pesticide/PCB Sample Extracts*). Note that the recover of Endrin Aldehyde is adversely affected by the TBA cleanup procedure detailed in this method. Accordingly, this compound must be determined prior to sulfur cleanup.

4.3. Co-eluting chlorophenols are eliminated by using SW846 Method 3620B (*SOP No. ED-ORP-020: Florisil Cleanup for Pesticide/PCB Sample Extracts*).

4.3.1. Check Florisil prior to use to assure quantitative recovery of targeted analytes. Duplicate checks are required for each new lot or every three hundred samples whichever is more frequent.

4.3.2. Check Florisil by spiking 1ml of the Pest Std Mix A midpoint (Supelco Catalog No. 47977) and 0.5 ml of trichlorophenol (Absolute Standardsl Catalog No. 20024) onto the cartridge and concentrating to final volume of 1 ml. Inject 1 ul onto a capillary column, conducting the elution and analyzing the extract. Recovery is acceptable if all pesticides are recovered at 80 - 110% and the recovery of trichlorophenol is <5% and co-eluting interfering peaks are absent from the extract.

5.0 Safety

Employees must abide by the policies and procedures in the Corporate Environmental Health and Safety Manual (CW-E-M-001) and this document. This procedure may involve hazardous material, operations and equipment. This SOP does not purport to address all of the safety problems associated with its use. It is the responsibility of the user of the method to follow appropriate safety, waste disposal and health practices under the assumption that all samples and reagents are potentially hazardous. Safety glasses, gloves, lab coats and closed-toe, nonabsorbent shoes are a minimum.

5.1. Specific Safety Concerns or Requirements

The gas chromatograph contains zones that have elevated temperatures. The analyst needs to be aware of the locations of those zones, and must cool them to room temperature prior to working on them.

There are areas of high voltage in the gas chromatograph. Depending on the type of work involved, either turn the power to the instrument off, or disconnect it from its source of power.

5.2. Primary Materials Used

The following is a list of the materials used in this method, which have a serious or significant hazard rating. **Note: This list does not include all materials used in the method. The table contains a summary of the primary hazards listed in the MSDS for each of the materials listed in the table.** A complete list of materials used in the method can be found in the reagents and materials section. Employees must review the information in the MSDS for each material before using it for the first time or when there are major changes to the MSDS.

Material (1)	Hazards	Exposure Limit (2)	Signs and symptoms of exposure
Acetone	Flammable	1000 ppm-TWA	Inhalation of vapors irritates the respiratory tract. May cause coughing, dizziness, dullness, and headache.
Hexane	Flammable Irritant	500 ppm-TWA	Inhalation of vapors irritates the respiratory tract. Overexposure may cause lightheadedness, nausea, headache, and blurred vision. Vapors may cause irritation to the skin and eyes.
Methanol	Flammable Poison Irritant	200 ppm-TWA	A slight irritant to the mucous membranes. Toxic effects exerted upon nervous system, particularly the optic nerve. Symptoms of overexposure may include headache, drowsiness and dizziness. Methyl alcohol is a defatting agent and may cause skin to become dry and cracked. Skin absorption can occur; symptoms may parallel inhalation exposure. Irritant to the eyes.
Methylene Chloride	Carcinogen Irritant	25 ppm-TWA 125 ppm-STEL	Causes irritation to respiratory tract. Has a strong narcotic effect with symptoms of mental confusion, light-headedness, fatigue, nausea, vomiting and headache. Causes irritation, redness and pain to the skin and eyes. Prolonged contact can cause burns. Liquid degreases the skin. May be absorbed through skin.
1 – Always add acid to water to prevent violent reactions.			
2 – Exposure limit refers to the OSHA regulatory exposure limit.			

6.0 Equipment and Supplies

6.1. Instrumentation:

6.1.1. Gas Chromatograph: The system used is an HP and an Agilent Technologies (Avondale, PA) model 5890/6890 Gas Chromatograph (GC). Each GC is equipped for simultaneous quantitation and confirmation columns using two separate detector channels on dual megabore capillary columns that are suitable for the analysis of organochlorine pesticides. All operations are as automated as possible with the equipment utilized.

6.1.2. Injection system: Sample injection is accomplished by a single auto injector. The auto injector is serviced by a robot arm that shuttles samples between the sample tray and the injector turret.

6.1.2.1. The samples are injected into a split/splitless injection port equipped with electronic pressure control (EPC). The injection port is normally operated in splitless mode during injection. The EPC is operated in the ramp pressure mode.

6.1.2.2. Liners: The injection port is each fitted with replaceable, heavy-walled siltek-coated glass double gooseneck liner. The liner contains a plug of silanized glass wool approximately 1 cm in length. The glass wool is positioned in the liner between the double gooseneck. The liner is replaced on a regular maintenance schedule.

6.1.2.3. Oven and Columns: Temperature programmable gas chromatograph ovens are required, capable of integrated temperature control between 35^{O}C and 350^{O}C.

6.1.2.3.1. Two dissimilar columns are used for analysis. A Restek RtxCLPesticides, 30m x 0.53mm ID x 0.5um film thickness column is used for sample quantitation. The secondary confirmation column is a Restek RtxCLPesticides II, 30m x 0.53mm ID x 0.42um film thickness column.

6.1.2.4. Detectors: Sample detection is by electron capture. The GC is equipped with dual Electron Capture Detectors (ECD), one for each column.

6.1.2.4.1. Each detector is supplemented with make-up gas to provide sufficient detector flow for maintaining the electron plasma. This is in addition to the gas exiting the column. The make-up gas (P-5 & Nitrogen) is fed from a supply other than the injection port.

7. Reagents and Standards

7.1. Reagents

7.1.1. Gases: Hydrogen is used as the carrier and injection port purge gas. It is introduced to the GC at the injection port. P-5 and Nitrogen) is used as make-up gas. It is introduced to the GC via the make-up gas adapter at the end of the capillary column. Hydrogen is supplied via a Parker Balston H2 Generator. Nitrogen & P-5 is supplied by Air Gas

7.1.1.1. The gas streams are polished using three traps or filters before introduction to the GC The traps are as follows:

7.1.1.1.1. Hydrocarbon trap
7.1.1.1.2. H_2O trap
7.1.1.1.3. O_2 scrubber

7.1.1.2. Both the moisture trap and the Oxygen scrubber are of the indicating type. They require either replacement or reconditioning upon color change of the active agents. Refer to the instructions for the individual traps to determine if it is still active. The hydrocarbon trap is a simple activated carbon trap. With high quality gas, it should last for an extended period of time (1-yr. minimum).

7.1.2. Solvents used in the extraction and cleanup procedures include n-hexane, methylene chloride, and acetone that are exchanged to n-hexane prior to analysis.

7.1.3. Hexane is required in this procedure. All solvents must be pesticide quality or equivalent. Each lot of solvent is screened for contaminants before being used for analysis as detailed in Eurofins Corporate Quality SOP No. CA-Q-S-001 (*Solvent & Acid Lot Testing & Approval*) and Eurofins Edison SOP No. ED-GEN-023 (*Bulk Solvent Testing and Approval*).

7.2. Standards

7.2.1. Standards are purchased as concentrated solutions (see Section 7.2.2).

NOTE: Independent sources are used for quantitation standards and spiking standards

7.2.1.1. Most stock solutions are diluted (in volumetric glassware) to working concentration using hexane as the diluent as described in Section 7.2.2.1.

7.2.2. Standard mixes and sources *.

Standard Name	Source
Organochlorine Pesticide Mix AB #3	Restek Catalog No. 32415
Organochlorine Pesticide Mix AB #3.sec	Restek Catalog No. 32415.sec (second source)
Pesticide Surrogate Mix	Restek Catalog No. 32000
Endrin/DDT	Supelco Catalog No 48282
Chlordane	Restek Catalog No. 32021
Toxaphene	Restek Catalog No. 32005
Toxaphene (different pattern)	Restek Catalog No. 32071
Mirex	Accustandard Cat. No. P-066S-10X
1-Bromo-2-nitrobenzene (internal standard)	Restek Cat. No. 32279

*Suppliers with equivalent standards may be substituted..

The components of each standard mix are as follows (note: 'sec' indicates second source):

Parameter	Supplier	Catalog No.	Concentration of Standard (ug/ml)
Aldrin	Restek	32415 & 32415.sec	2000
Alpha-BHC	Restek	32415 & 32415.sec	2000
Beta-BHC	Restek	32415 & 32415.sec	2000
Delta-BHC	Restek	32415 & 32415.sec	2000
Gamma-BHC (Lindane)	Restek	32415 & 32415.sec	2000
Alpha -Chlordane	Restek	32415 & 32415.sec	2000
Gamma -Chlordane	Restek	32415 & 32415.sec	2000
Technical Chlordane	Restek	32021	1000
4,4' –DDD	Restek	32415 & 32415.sec	2000
4,4' –DDE	Restek	32415 & 32415.sec	2000
4.4' –DDT	Restek	32415 & 32415.sec	2000
Dieldrin	Restek	32415 & 32415.sec	2000
Endosulfan I	Restek	32415 & 32415.sec	2000
Endosulfan II	Restek	32415 & 32415.sec	2000
Endosulfan sulfate	Restek	32415 & 32415.sec	2000
Endrin	Restek	32415 & 32415.sec	2000
Endrin aldehyde	Restek	32415 & 32415.sec	2000
Endrin ketone	Restek	32415 & 32415.sec	2000
Heptachlor	Restek	32415 & 32415.sec	2000
Heptachlor epoxide	Restek	32415 & 32415.sec	2000
Methoxychlor	Restek	32415 & 32415.sec	2000
Toxaphene	Restek	32005	1000
Toxaphene	Restek	32071	5000
Mirex	Accustandard	861428-U	1000
4,4-DDT	Supelco	18282	500
Endrin	Supelco	48282	500
Decachlorobiphenyl (DCB)	Restek	32000	200
Tetrachloro-m-xylene (TCmX)	Restek	32000	200
1-Bromo-2-nitrobenzene (internal standard)	Restek	32279	1000

7.2.2.1. Standards Preparation

7.2.2.1.1. Calibration Mix (Organochlorine Pesticide Mix)

The 5 point calibration standards are prepared as detailed in the following table using volumetric glassware and hexane as the diluent:

Initial Calibration Standards Prep (Organochlorine Pesticide Mix)					
Stock Std	**2.5 ppb**	**50 ppb**	**100 ppb**	**250 ppb**	**500 ppb**
Organochlorine Mix AB#3 ***(2000 ug/ml)*** *Restek (Cat No..32415)in Hexane:Toluene*	Volume brought up to 200 ml hexane: *0.25 ul*	Volume brought up to 200 ml hexane: *5.0 ul*	Volume brought up to 500 ml hexane: *25 ul*	Volume brought up to 200 ml hexane: *25 ul*	Volume brought up to 200 ml hexane: *50 ul*
Initial Calibration Standards Prep (TCMX/DCB Surrogate Mix)					
Stock Std	**25 ppb***	**50 ppb**	**100 ppb**	**150 ppb**	**200 ppb**
Tetrachloro-m-xylene/ Decachlorobiphenyl Surrogates Mix (200 ug/ml) *Restek (Cat, No, 32000) in Acetone*	Volume brought up to 200 ml hexane: *6.25 ul*	Volume brought up to 200 ml hexane: *50 ul*	Volume brought up to 500 ml hexane: *250 ul*	Volume brought up to 200 ml hexane: *150 ul*	Volume brought up to 200 ml hexane: *200 ul*
Initial Calibration Standards Prep (Mirex)					
Stock Std	**2.5 ppb**	**50 ppb**	**100 ppb**	**250 ppb**	**500 ppb**
Mirex ***(1000 ug/ml)*** *Accustandard (Cat. No. P-066S-10x) in Methanol*	20x dilution of 50 ppb	10x dilution of 500 ppb	5x dilution of 500 ppb	2x dilution of 500 ppb	Volume brought up to 100 ml hexane: *50 ul*

Note: 20 ul of 5 ug/ml Internal Standard solution is added to all calibration standards prior to analysis.

7.2.2.2. **Pesticide Surrogate Spike Mix (10 ug/ml**) : The Pesticide 10ug/ml surrogate spiking solution is prepared by diluting 10 ml of 200 ug/ml of the Pesticide Surrogate Mix (Restek-32000) in to 200 ml of Acetone. For spiking instructions refer to the applicable prep SOP.

7.2.2.3. **Pesticide Surrogate Spike Mix (2 ug/ml) for the reduced volume extraction option:** prepare a 2 ug/ml spiking solution by diluting 10 ml of the 10 ug/ml stock standard described in 7.2.2.2 to a final volume of 50ml in acetone. For spiking instructions refer to the applicable prep SOP.

7.2.2.4. **Pesticide Internal Standard Spike Mix (5 ug/ml):** The Pesticide 5 ug/ml internal standard spike mix is prepared by

dilution 1 ml of 1000 ug/ml of the 1-Bromo-2-Nitrobenzene standard (Restek 32279) in to 200 ml of Hexane. 20 ul of this solution is added to all standards, QC samples and field sample extracts prior to analysis.

7.2.2.5. **Pesticide Spiking Standard (20 ug/ml):** The Pesticide Spiking Mix containing the single component pesticides is prepared at a final concentration of 20 ug/ml by diluting 500 ul of the Organochlorine Pesticide Mix AB#3 (Restek 32415 to a 50 ml final volume with acetone. For spiking instructions refer to the applicable prep SOP.

7.2.2.6. **Pesticide Spiking Standard (4 ug/ml) for the reduced volume extraction option**: prepare a 4 ug/ml spiking solution by diluting 10 ml of the 20ug/ml standard prepared in Section 7.2.2.4 above to a final volume of 50ml in acetone. For spiking instructions refer to the applicable prep SOP.

7.2.2.7. **System Performance Solution (Breakdown Check)** 3,3-DDT and Endrin at 0.25 ug/ml): The breakdown check is prepared by taking 250 ul of 500 ug/ml DDT/Endrin Mix (Supelco Catalog No 48282) and bringing it up to a volume of 500 ml with hexane

7.2.2.8. **Technical Chlordane Calibration Solution** (1.0 ug/ml solution w/ surrogates TCMX & DCB at 0.10 ug/ml): 100 ul of 1000 ug/ml Technical Chlordane Standard (Restek Catalog No 32021) and 50 ul of 200 ug/ml Surrogate Mix (Restek Catalog No. 32000). Dilute to 100 ml in Hexane. ***NOTE:*** ICAL consists of single midpoint calibration standard for multiple responders Technical Chlordane and Toxaphene.

7.2.2.9. **Toxaphene Calibration Solution** (1.0 ug/ml solution w/surrogates TCMX & DCB at 0.1 ug/ml) : 20 ul of 5000 ug/ml Toxaphene stock (Restek Catalog No 32071) and 50 ul of 200 ug/ml Surrogate Mix (Restek Catalog No. 32000). Dilute to 100 ml in Hexane. ***NOTE:*** ICAL consists of single midpoint calibration standard for multiple responders Technical Chlordane and Toxaphene.

7.2.2.10. **Initial Calibration Verification (ICV)** Preparation: follow the instructions above for midpoint standards substituting the second source standard (catalog no. suffix = sec) for the primary standards.

8. Sample Collection, Preservation, Shipment and Storage

8.1. Extracts must be stored under refrigeration in the dark and analyzed within 40 days of extraction.

8.2. Samples from chlorinated water sources must be treated with sodium thiosulfate (0.008% solution) at the time of collection to remove chlorine. NOTE: containers pre-preserved with sodium thiosulfate must be requested in bottle orders for samples from chlorinated water sources.

Matrix	Sample Container	Min. Sample Size	Preservation	Holding Time	Reference
Waters	Amber glass, 250 ml	250 ml	Cool 4 ± 2ºC	7 days to extraction; Analyze within 40 days of extraction	SW846
Soils	Glass, 2 or 4 oz	100 g	Cool 4 ± 2ºC	14 days to extraction; Analyze within 40 days of extraction	SW846

Sample container, preservation techniques and holding times may vary and are dependent on sample matrix, method of choice, regulatory compliance, and/or specific contract or client requests. Listed below are the holding times and the references that include preservation requirements.

9. Quality Control

9.1. **Sample QC** - The following quality control samples are prepared with each batch of samples.

Quality Controls	Frequency	Control Limit
Method Blank (MB)	1 in 20 or fewer samples	< Rpt. Limit
Laboratory Control Sample (LCS)[1]	1 in 20 or fewer samples	Statistical Limits [4]
Matrix Spike (MS)[2]	1 in 20 or fewer samples	Statistical Limits [4]
MS Duplicate (MSD)[2]	1 in 20 or fewer samples	Statistical Limits [4]
Surrogates	every sample[3]	Statistical Limits [4]
Internal Standard [1]	Every sample[3]	Response within -50% to +100% of most recent cal standard

LCS Duplicate (LCSD) is performed only when insufficient sample is available for the MS/MSD or when requested by the client/project/contract.
[2] The sample selection for MS/MSD are randomly selected, unless specifically requested by a client….predetermined by the extraction lab.
[3] Analytical and QC samples (MB, LCS, MS/MSD)
[4] Statistical control limits are updated annually and are updated into LIMS.

9.1.1. **Method Blanks** are extracted with each sample batch on each day that samples are extracted. The analytical results for the method blank must fall

below the reporting limit for each compound of interest. If a target compound is detected in the blank at a concentration higher than the reporting limit, first the extract is reanalyzed for confirmation. If results are still outside of limits the entire batch of samples extracted with the affected blank must be re-extracted and reanalyzed.

9.1.2. Laboratory Control Sample (LCS): A Laboratory Control Sample (LCS) or blank spike must be extracted and analyzed for with each batch of 20 environmental samples. The recoveries of the LCS must fall within lab generated acceptance criteria (refer to the current TALS Method Limit Group database). If the LCS recovery results are outside of these limits, the extract is reanalyzed. If LCS recoveries are still outside of QC limits after extract reanalysis but recoveries for the Matrix Spike/ Matrix spike Duplicate (MS/MSD) are within QC limits, the data is reported and a Non Conformance Memo (NCM) is written.

9.1.3. Matrix Spike/Matrix Spike Duplicate (MS/MSD): A Matrix Spike/Matrix Spike Duplicate (MS/MSD) pair is extracted and analyzed with every batch of 20 environmental samples. MS/MSD recoveries are evaluated against lab generated limits (refer to the current TALS Method Limit Group database). If the MS/MSD recovery limits fall outside of lab limits the LCS recovery is evaluated. If the LCS recoveries meet criteria the data is reported and a Non-Conformance Memo (NCM) is written.

9.1.4. Surrogate Standards: All samples, blanks and QC samples are spiked with a 2 component surrogate standard mix containing TCMX & DCB (see Section 7.2). The percent recovery of the surrogate standards is calculated and compared to lab generated limits (refer to the current TALS Method Limit Group database*)*. Minimum requirements for surrogate evaluation:

- Both surrogates must have reportable results that meet the acceptance criteria;
- Reported surrogates must be from a column with a passing CCV;
- At least one surrogate must pass on any column from which target analytes are identified and reported.

If both TCMX and DCB recovery are outside of acceptance limits the sample extract is reanalyzed to confirm. If the recoveries are still outside of limits the sample must be re-extracted and reanalyzed or the data flagged as “estimated concentration”.

9.1.5. Internal Standard: The internal standard (1-bromo-2-nitrobenzene) must elute within 30 seconds of and have an area response of 50 to 100% as compared to the most recent preceding calibration standard.

9.1.6. Lower Limit of Quantitation (LLOQ) (aka Reporting Limit) Verification: The LLOQ is the lowest concentration at which the laboratory has demonstrated target analytes can be reliably measured and reported with a certain degree of confidence, which must be ≥ the lowest point in the calibration curve. The lab verifies the LLOQ annually to

demonstrate the capability of quantiation at lower analyte concentrations. The verification is performed by the extraction and analysis of an MDL spike at a concentration of 0.5-2 times the established LLOQ. Recovery of target analytes in the LLOQ verification should be within established in-house limits or within other such project-specific acceptance limits to demonstrate acceptable method performance at the LLOQ. Until the laboratory has sufficient data to determine acceptance limits, the LCS criteria ± 20% (i.e., lower limit minus 20% and upper limit plus 20%) will be used for the LLOQ acceptance criteria. The annual LLOQ verification is completed and documented with the required annual MDL evaluation.

9.2. Instrument QC

9.2.1. GC System Performance Check

9.2.1.1. **Endrin/4,4'-DDT Breakdown:** Prior to performing any standards or sample analysis, a daily check is made on the chromatographic performance of the system. This performance check is made by injecting a standard of Endrin and DDT, each at a 250-ppb level (see Section 7.2), and calculating the percentage breakdown for each compound.

9.2.1.2. Ideally, only two peaks will be seen (one for Endrin and one for DDT). As a rule, this is not the case. It is normal to observe up to six peaks. Three peaks are attributable to Endrin and its degradation products: Endrin Aldehyde (EA) and Endrin Ketone (EK). Three peaks are attributable to DDT and its degradation products: DDE and DDD. Calculate the percentage breakdown as follows:

Endrin:

$$\frac{\text{(Areas of EA + EK)}}{\text{(Areas of EA + EK + Endrin)}} \times 100 = \%\ \text{breakdown Endrin}$$

DDT:

$$\frac{\text{(Areas of DDE + DDD)}}{\text{(Areas of DDE + DDD + DDT)}} \times 100 = \%\ \text{breakdown DDT}$$

9.2.1.3. If the percentage breakdown for either Endrin or DDT is greater than 15%, the system CANNOT be used for pesticide analysis. If the Endrin/DDT performance check fails, injection port/column maintenance must be performed. Usually, changing the glass wool/liner will cure most breakdown problems in the injection port. Depending upon the nature of the samples, the entire injection port will occasionally need to be cleaned. This cleaning is best done with 1:1 Acetone: Hexane. Another routine maintenance operation to improve column performance is the removal of the

first 3 cm of the column. (Note: the septa should be changed each time the injection port is opened).

9.2.1.4. After injection port/column maintenance has been performed, and the columns have been given time to equilibrate (baseline back down to normal) the Endrin/DDT must be re-injected and the system performance re-evaluated.

9.2.2. Initial Calibration Range and Initial Calibration Verification (ICV)

9.2.2.1. ***Initial Calibration Range*:** Single component pesticides are calibrated using a five-point calibration range. Multi-component pesticides are calibrated using a single point calibration at the anticipated midpoint of the calibration range. Standards are prepared following the instructions in Section 7.2.

9.2.2.2. Single response Pesticide Calibration: All single component pesticides and two surrogates are calibrated with a minimum of 5 concentrations. Single component pesticides are analyzed at 10, (5 ppb for the 125 ml initial volume method) 5, 100, 250 and 500 ppb. Surrogate standards are analyzed at 25, 50, 100, 150 and 200 ppb. See Section 7.2 for details on standard prep.

9.2.2.3. Multi-response Pesticide Calibration: Chlordane (technical) and Toxaphene initial calibration is accomplished by analysis of a single point at 1000 ppb (see Section 7.2 for details on standard prep).

9.2.2.4. ***Initial Calibration Verification (ICV):*** An Initial Calibration Verification (ICV) standard is analyzed immediately after the Initial Calibration Range and before any samples are analyzed. The ICV is prepared as detailed in Section 7.2 and must be from a source separate from the standards used in the Initial Calibration Range.

9.2.3. Continuing Calibration Verification (CCV): For single component pesticides, a mid-point Continuing Calibration Verification (CCV) must be analyzed every 12-hours or 20 samples (whichever is more frequent). 'Samples' here are defined as field samples and batch QC (MB, LCS, MS, MSD) and do not include CCVs, PEMs, instrument blanks, etc… For multi-response pesticides a CCV must be analyzed within 12 hours of any multi-response pesticide detects.

9.2.3.1 Analysis of Replicate CCVs: Occasionally dual, sequential CCVs may (for a variety of reasons) be included in an analytical sequence. When such replicate CCVs are injected both must be evaluated as detailed in the table below (reference Eurofins Document CA-Q-W-008, 'Technical Guidance on the Use and

Evaluation of Replicate Continuing Calibration Verification (CCV)').

Dual CCV Evaluation Decision Tree		
Inejection	**QC Acceptance**	**Action**
CCV1 **CCV2**	**Pass** **Pass**	Continue analytical sequence. Data acceptable based on calibration.
CCV1 **CCV2**	**Pass** **Fail**	Analyses before CCV1 may be accepted. Re-analyze all samples that were analyzed after failed CCV2.
CCV1 **CCV2**	**Fail** **Pass**	Re-analyzed all samples that were analyzed before CCV1 and after the previous compliant CCV. Analyses after CCV2 may continue.
CCV1 **CCV2**	**Fail** **Fail**	Re-analyze all samples since the last acceptable CCV. Perform maintenance and/or recalibration prior to re-analysis.

9.2.3.2 In the event that replicate CCVs are included in a sample injection sequence, the acceptance of the associated sample analyses must be properly evaluated and the evaluation process must be documented. Under no circumstances is it allowable to accept sample data based on the evaluation of only 1 of the CCV replicates.

9.2.3.3 Clear documentation of the evaluation of the CCV must be included in the analytical sequence run log (i.e., 'Pass' or 'Fail' in the comment section for each CCV). Additionally, clear documentation of corrective action taken in the event of CCV failures must be included in the run log (see table above for actions that need documentation).

Note: It is acceptable to use the CCV solution as a primer at the beginning of an analytical sequence however the injection should be documented as a 'Primer' in the sequence rather than as a CCV.

9.2.4. Calibration Acceptance Summary

9.2.4.1. Retention Time Windows: Retention time windows must be established to compensate for minor shifts in absolute retention times as a result of sample loading and normal chromatographic variability (for more detail on the following procedures refer to

Eurofins Corporate Technical Services Policy No. CA-T-P-005, “Policy for Determining RT Windows for GC/ECD Tests” and Eurofins Corporate Technical Services Work Instruction No. CA-T-WI-010, “Further Guidance on the RT Window Policy No. CA-T-P-005”).

9.2.4.1.1. Initial determination of RT windows.

9.2.4.1.1.1. The center of the retention time (RT) window shall be updated based on the middle level in the initial calibration of the first CCV in the daily sequence, whichever is most recent.

9.2.4.1.1.2. Use a default RT window of 0.01 minutes. At the end of the batch evaluate all CCVs and LCSs in the batch. If necessary, widen the window such that all analytes fall within the RT window. Reprocess the batch using the new RT windows.

9.2.4.1.1.3. Multiply the maximum deviation by 1.5. This is the retention time window, unless the result is less than 0.01 minutes, in which case the window is set at 0.01 minutes. For example, if the maximum RT deviation for a specific analyte is 0.008 minutes, then the RT window is set at ±0.012 minutes.

NOTE: For the multi-component analytes, for example Aroclors, Toxaphene and Technical Chlordane, the maximum deviation must be evaluated for each of the 3 to 6 major peaks used for sample calculations.

9.2.4.1.1.4. Retention time windows for analytes of interest must not overlap for GC analysis.

9.2.4.1.2. Ongoing evaluation of retention time windows

9.2.4.1.2.1. Evaluate the retention time windows on an ongoing basis. The center of the RT window is updated on the first CCV of the day. All analytes for all subsequent CCVs, LCS and matrix spikes must fall within the retention time window (except as discussed below).

9.2.4.1.2.2. Matrix spike analytes may fall outside of the retention time window if there is a large non-target peak coeluting with the analyte in the matrix spike.

9.2.4.1.2.3. If any analytes fall outside of the retention time window in CCVs, LCS or matrix spikes (except as discussed above for matrix spikes) then the RT windows for those analytes shall be widened to the minimum degree required for the analyte to fall within the RT window. All samples in the batch shall be reprocessed with the new RT window, and the wider RT window shall remain in place for subsequent batches.

9.2.4.1.2.4. Retention time windows should be reliably narrower than ±0.03 minutes. If RT windows wider that this are necessary, the instrument should be evaluated and maintenance performed as needed. Subsequent to maintenance, RT windows shall be narrowed to the extent that is consistent with the data obtained.

9.2.4.2. **Initial Calibration Range:** Internal standard calibration is employed for this method. A response factor is calculated for each analyte at each calibration concentration.

Response factor = $((A_x)\ (C_{is}))/((A_{is})\ (C_x))$

Where:
A_x = area of the compound
C_x = Concentration of the compound
A_{is} = area of the internal standard
C_{is} = Concentration of the internal standard

9.2.4.2.1. Calculate the percent Relative Standard Deviation of the response factors for each compound at each level:

% RSD = (RF Standard Deviation/RF Mean) X 100

Where:
RF = Response Factor

9.2.4.2.2. If the % RSD across the 5 point range is <20% for any given compound the calibration can be assumed to be linear and the average response factor can be used to calculate concentrations of target compounds in samples.

9.2.4.2.3. If the % RSD is >20% for any given compound, a first order linear regression may be applied to the data to calculate the calibration curve and determine sample concentration. If this method is employed, the r^1 (Correlation Coefficient) value must be $\geq$ 0.990 for the calibration to be acceptable. Calibration is checked every

12 hours or after every twenty (20) samples, whichever comes first, by injecting a calibration verification standard for all single component pesticide standards.

9.2.4.2.4. Chlordane and Toxaphene Calibration: Chlordane and Toxaphene are multiple response pesticides and are calibrated with a minimum of 5 points as required (i.e, within 12 hours of either analyte being detected in a sample). Three to eight peaks are used for calculation of response factors and the same criteria detailed above is applied to determine acceptability of calibration.

9.2.4.2.5. Resolution: All single component analyte peaks must exhibit at least 80% chromatographic resolution. The analyst performs a visual check of the mid-level initial calibration standard and all subsequent calibration checks (ICV/CCV). If the resolution requirement is not met instrument maintenance should be performed followed by re-calibration. Percent resolution can be calculated when necessary as follows:

$$\% \text{ Resolution} = V/H \times 100$$

Where:

V= Depth of the valley between two peaks. The depth of the valley is measured along a vertical line from the level of the apex of the shorter peak to the floor of the valley between the two peaks

H = Height of the shorter of the adjacent peaks

9.2.4.3. **Relative Standard Error:** The percent relative standard error (% RSE) from the calibration curve is used to evaluate the initial calibration. This provides a measure of how much error is associated with using the calibration curve for quantitation. The average Relative Standard Error (RSD for average response factors) of the calibration points from the curve used must be ≤ 20%.

Some data systems will not measure the %RSE from a linear or quadratic fit. For the linear case, the correlation coefficient may be used as an alternative to the %RSE, and must be greater than or equal to 0.990. For the quadratic case the Coefficient of Determination may be used, and must be greater or equal to 0.990.

Note: The Relative Standard Error (RSE) is superior to the Correlation Coefficient (r) and Coefficient of Determination (r_2) for

testing the fit of a set of calibration points to a line. The lower points on a curve have little effect on *r.* As a result, a curve may have a very good correlation coefficient (>0.990) while also having > 100% error at the low point

9.2.4.4. Initial Calibration Verification (ICV): An ICV will consist of a second source standard at or near the midpoint of the Initial Calibration Range analyzed at the frequency specified in Section 9.2.2.4. The calculated concentration of the ICV must be within ±20%D of the expected concentration. Should the %D exceed 20% the analyst should take corrective action (check standard solution, perform instrument maintenance, etc.) and re-inject the ICV. If the %D still exceeds 20% after a single ICV reinjection, a new Initial Calibration Range must be analyzed. All single component analyte peaks must exhibit 80% resolution (see Section 9.2.4.2.5)

9.2.4.5. Continuing Calibration Verification (CCV): A CCV will consist of a second source standard at or near the midpoint of the Initial Calibration Range analyzed at the frequency specified in Section 9.2.3.. The calculated concentration of the CCV must be within ±20%D of the expected concentration. Should the %D exceed 20% the analyst should take corrective action (check standard solution, perform instrument maintenance, etc.) and re-inject the CCV. If the %D still exceeds 20% after a single CCV reinjection, a new Initial Calibration Range must be analyzed. All single component analyte peaks must exhibit 80% resolution (see Section 9.2.4.2.5).

Step	***Standards***	***Type***	***Control Limit***	***Frequency***
Method # 8081B				
GC System Performance Check	*Endrin/DDT, 250 ppb*	*Performance check*	*15% breakdown*	*At beginning of 12 hour clock and after system maintenance*
*Initial Calibration**	2.5, 50, 100, 250 and 500 ppb for single response, 1000 ppb for multi response	*Average response factor or 1st order linear regression*	*For average RF: <20%RSD all analytes. For linear regression: r ≥0.990 or <20%RSE*	*As required when ICV or CCV do not meet requirements*
ICV	*100 ppb*	*Average*	*± 20%D*	*Once after each initial calibration*
CCV	*100 ppb*	*Average*	*± 20%D*	*Every 12 hrs or 20 samples, whichever is more frequent*

10. Procedure

10.1. Gas Chromatograph Operation

10.1.1. The sequence of events for GC analysis involves many steps. First the injection system and column performance and calibration must be verified. Maintenance operations are performed as needed. Then samples must be run on the instrument. Chromatograms and reports must be evaluated for content, integration and concentration. Re-runs and dilutions must be made based on the calibrations that were in effect at the time the sample was run. Lastly, a detailed analysis and calculations must be performed to determine the concentration of all the parameters for which the sample was analyzed.

10.1.2. General Operating Conditions

10.1.2.1. Injection System: A split/splitless injection port with electronic pressure control (EPC) is used. Thirty seconds after sample injection, the purge valve is turned on to facilitate the sweeping of any remaining residual solvent/sample from the injection port.

10.1.2.2. The EPC is used in the ramp pressure mode. The ramp pressure program is as follows:

Initial Pressure	InitialTime	Rate	Final Pressure	Hold
12 psi	2.5 min	7 psi/min	4 psi	1.50 min
		5 psi/min	9 psi	1.40 min
		9 psi/min	13 psi	2.00 min

10.1.2.3. For pesticide analysis the normal operating conditions of the injection port are as follows:

Injection port Temperature:	250^{o}C
Column flow:	12.3 ml/minute
Split vent flow:	5 ml/minute
EPC:	Pressure Ramp
Detector temperature	330C

10.1.2.4. In addition to the EPC, the injection port is also equipped with a siltek-coated glass double goose neck liner that contains a 1 cm glass wool plug. The plug of glass wool is located in the liner between the double goose neck.

10.1.2.5. This liner/glass wool combination provides many functions. The glass wool serves as a heat sink rapidly vaporizing solvent and samples resulting in higher response factors. The liner also protects the column head from accumulation of high boiling residuals and particulates.

10.1.2.6. The glass wool will be changed when changing the liner. The changing of the glass wool/liner is based upon the breakdown of

an Endrin/DDT standard. This is covered in further detail in section 10.2.1.

10.1.2.7. Regular maintenance is performed on the injection port. When the glass wool/liner is changed, the septa also must be changed. Injection port, oven and detector temperatures are lowered to ambient prior to "cracking" the system. This is so as to introduce a minimum of damaging oxygen molecules into the system.

10.1.2.8. After the system has cooled, the old liner is removed. The injection port should be checked for particulate residues and cleaned as needed. A flashlight is usually required for this. After a new liner has been prepared it is placed into the injection port. A graphite seal is placed around the liner. The edges of the seal must be flat, not knife-edged, and free of nicks or burrs. If any of these conditions are not met, the graphite seal must be replaced as well. The graphite seal is critical to proper operation of the injection port. If in doubt, replace it.

10.1.2.9. The locking ring on the top of the injection port should be turned, with the wrench, about 1/8 turn past finger tight. The septum nut should never be tightened more than finger tight. After the injection port is reassembled, all column nuts inside the oven should be checked for leaks using Snoop (Supelco) or another suitable leak tester.

10.1.2.10. Once the signal from both detectors has stabilized, it is time to re-heat the zones. The zones should be heated in the order of detectors, oven and then injectors. This is to ensure that volatilized contaminants do not condense on the column or detector.

10.1.2.11. Oven: With the megabore columns installed, temperature programming is employed to achieve higher resolution of compounds and shorter run times than could be accomplished using isothermal methods.

10.1.2.11.1. A standard oven program for pesticide analysis is employed for all columns as follows:

Initial Temp	**Hold Time1**	**Rate1**	**Temp1**
160°C	0.62 min	30°/min	244°C
Hold Time2	**Rate 2**	**Final Temp**	**Final Time**
2.5min	21°/min	315°C	3.0min

10.1.2.12. Detectors: Detectors operate at 330°C and need to be supplied with 60 ml/min total flow. They are essentially maintenance free

on a day-to-day basis. They are routinely baked out at 330°C to remove persistent contaminants. On occasion the detectors may be baked out at a higher temperature to remove contaminants with an extremely high boiling point (CAUTION: Do not exceed the maximum detector temperature of 380°C).

10.1.2.12.1. If the detectors are particularly contaminated, they must be sent to Agilent Technologies in Avondale, Pennsylvania for reconditioning. This should occur if the detector baseline is greater than 100 Hz. Detector reconditioning should be required at a maximum of biannually.

10.1.2.13. Chemstation: HP Chemstation software is used for automation of runs and data acquisition. The system is dedicated to a single GC and does not multitask. Therefore, data manipulation cannot be done while sample analysis is in progress. The data system acquires and stores all chromatographic data.

10.1.2.14. Eurofins Chrom data processing software is used for the processing of the chromatography data files. Calibrations, verification standards and samples are processed and reviewed using this database. Chrom is integral to Eurofins LIMS (TALS) which is used to generate all reports..

10.2. Analytical Sequence

10.2.1. The analytical sequence for performance checks, initial calibration, calibration verifications and sample analysis is described in the following sections.

10.2.2. Before calibration standards are analyzed the GC Performance Check Standard (see Section 9.2.2.1) must first be analyzed and evaluated to check the performance of the injection port and column with regard to catalytic active sites. The breakdown for both Endrin and DDT in the Performance Check Standard must be less than 15%. If the performance check fails this criteria system maintenance must be performed and the check successfully reanalyzed before proceeding with calibration.

10.2.3. A five point initial calibration (ICAL) is analyzed and evaluated for each of the 21 single response pesticides plus surrogates as described in Section 9.2.4.2. When needed a 5 point ICAL is also analyzed for Mirex.

10.2.4. Calibration for Technical Chlordane and Toxaphene: Chlordane and Toxaphene are multiple response pesticides containing at least 3-8 primary peaks each. A single point calibration check standard at a concentration of

1000ug/l is analyzed for Chlordane and Toxaphene. A full 5 point calibration range is analyzed for these compounds should they be detected in client samples..

10.2.5. A second source initial calibration verification (ICV) is analyzed and evaluated for each of the 21 single response pesticides as described in Section 9.2.4.3. When needed an ICV is also analyzed for Mirex. **NOTE**: The 12 hour time clock for Pesticides commences with the injection of the first Pesticide Calibration Standard or Verification.

10.2.6. GC Performance Check Standard is analyzed and the breakdown of Endrin/DDT is measured again before samples are analyzed and at the beginning of each subsequent 12 hour shift. If the breakdown check fails, then injection port/column maintenance is required

10.2.7. Client samples and QC samples may be analyzed after the analysis of the performance check. Sample analysis may proceed for up to 20 samples or 12 hours prior to analysis of another calibration verification (whichever is more frequent).

10.2.8. A Continuing Calibration Verification (CCV) for the 21 single response pesticides plus surrogates must be analyzed and evaluated every 12 hours or 20 samples (whichever is more frequent) as described in Section 9.2.4.4.. This is accomplished by running the midpoint calibration standard as a CCV (Pest Mix 100 ppb check standard and Mirex 100 ppb check standard; see Section 7.2.2.1). The calculated concentration for each compound in the CCV must be +/- 20 % of the expected concentration. Any samples analyzed after a failing CCV must be reanalyzed under a passing CCV. If, after performing instrument maintenance, the reanalysis of a CCV fails criteria, a new initial calibration range must be analyzed (see Section 9.2.4.2). Any data reported against a failing CCV must have a Non-Conformance Memo detailing the issue.

10.2.9. Analytical Sequence: The automation of GC runs is accomplished via the "SEQUENCE" macro of the Chemstation. The sequence file contains the name of Method file corresponding to the type of analysis to be performed, the range of samples to be run, and the number of injections per bottle. It is common practice to run the check standards, evaluate the instrument status, and then complete the Sample Table and Sequence File. If everything else is complete, the run is initiated using the START SEQUENCE soft-key of the SEQUENCE macro.

Example Analytical Sequence	
1. Hexane	12. Mirex 1 (10 ppb) (if needed)
2. Instrument Blank	13. Mirex 2 (20 ppb) (if needed)
3. Endrin/DDT Breakdown	14. Mirex 3 (100 ppb) (if needed)
4. Pesticide Mix 1 (2.5 ppb)	15. Mirex 4 (250 ppb) (if needed)
5. Pesticide Mix 2 (50 ppb)	16. Mirex 5 (500 ppb) (if needed)

Example Analytical Sequence	
6. Pesticide Mix 3 (100 ppb) 7. Pesticide Mix 4 (250 ppb) 8. Pesticide Mix 5 (500 ppb) 9. Chlordane (1000 ppb) 10. Toxaphene (1000 ppb) 11. Pesticide ICV (100 ppb)	17. Mirex ICV (100 ppb) (if needed) 18. Instrument Blank 19. Endrin/DDT Breakdown 20. Pest Mix 3 (100 ppb)…CCV 21. Mirex 3 (100 ppb) …CCV (if needed) 22. 20 or fewer samples or 12 hours 23. Instrument Blank 24. Endrin/DDT Breakdown 25. Pest Mix 3 (100 ppb)…CCV 26. Mirex 3 (100 ppb) …CCV (if needed) 27.20 or fewer samples or 12 hours

10.3. Dual Column Approach

10.3.1. The laboratory designates the rear column as the primary column and the front column as the secondary column. If the difference between the dual columns results in ≤40% RPD report the higher concentration.

10.3.2. The values are calculated from the chromatographic peaks that fall within the daily retention time windows. Retention times are updated using the retention times found in opening CCV for the most recent 12 hour period.

10.3.3. If the calculated values are greater than 40% RPD of each other, report the lower concentration regardless of whether that result is from the primary or secondary column. Report the result with a flag of P*. **Exception:** NJDEP DKQP protocols require reporting the higher concentration in all instances.

10.3.4. If the surrogates on one column are >40% RPD compared to the other column, this may be indicative of a bad injection or columnar blockage. The sample should be reanalyzed. If similar results are obtained following reanalysis, report the lower of the two numbers and describe the circumstances in the job summary and report case narrative.

10.3.5. If one of the columns fails CCV criteria (but the CCV is between 15%-40% greater than expected value), the sample results shall be reported from the compliant column. If the falls outside of acceptance criteria on the low side, reanalysis shall be performed.

10.3.6. If the CCV on one of the columns is more than 40% different from the correct value, it can be assumed that there has been significant drift on that column. The sample shall be reanalyzed against an acceptable calibration.

10.3.6.1. An exception to this requirement would be if the CCV recovery on one column fails on the high side and >40% RPD but the associated samples were non-detect for all target analytes on

both columns. In this case the non-detect results may be reported from the compliant column.

10.3.7. In some cases where the sample chromatography is complex and has largely varying peaks concentrations, the chromatographic separation may not be sufficient on the 0.53mm ID columns. In this case a confirmatory analysis on an instrument with 0.32 ID columns may be required. The supplemental data produced using analysis on the 0.32mm ID 'microbore' column may minimize overlapping and baseline interference difficulties, and better resolves potential positive identifications. Use of this alternative chromatographic technique shall be noted in the job summary and report case narrative.

10.3.8. In summary, the flow chart in Attachment 1 presents a recommended rational approach to selecting the better number to report for dual column data. It shall be noted that these recommendations may be overridden by project specific requirements and that they cannot cover all eventualities. The complexity of some data set will require the final decision to be made utilizing the judgment of experienced analysts. In some cases further cleanup steps to remove interferences may be appropriate.

10.4. Extract Cleanups

10.4.1. Cleanup methods are dictated by the original sample matrix and the parameters being determined.

10.4.2. Cleanup of all water samples, if needed, is performed using Florisil (Eurofins Edison SOP No. ED-ORP-020*: Florisil Cleanup for Pesticide/PCB Sample Extracts*, SW846 Method 3620B, most current revision) and TBA sulfite (Eurofins Edison SOP No. ED-ORP-021: *The Removal of Elemental Sulfur from Pesticide/PCB Sample Extracts, SW846 Method 3660B,* most current revision). Blanks must also undergo cleanup following the same procedures as samples.

10.4.3. Cleanup of all soil samples is conducted using Florisil (Eurofins Edison SOP No. ED-ORP-020*: Florisil Cleanup for Pesticide/PCB Sample Extracts*, SW846 Method 3620B, most current revision) and, if needed, TBA sulfite (Eurofins Edison SOP No. ED-ORP-021: *The Removal of Elemental Sulfur from Pesticide/PCB Sample Extracts, SW846 Method 3660B,* most current revision). Blanks must also undergo cleanup following the same procedures as samples.

10.4.4. Check Florisil prior to use to assure quantitative recovery of target analytes. (see Section 4.3.2 above and Eurofins Edison SOP No. ED-ORP-020*: Florisil Cleanup for Pesticide/PCB Sample Extracts*, SW846 Method 3620B,

10.5. Data Processing and Documentation

10.5.1. Before the analysis sequence is initiated the GC Maintenance logbook must be filled out. It should contain the following information: date, injector temp,

oven temp, detector temp, injector flow, signal A, signal B, analysts initials, and notes for any necessary repairs.

10.5.2. After samples have been run, each standard and sample must be entered into the Instrument Run Log. The Instrument Run Log should contain the following information: run date, data file name, vial position, sample number, initial volume/weight, final volume, dilution factor, method, job number, QA number, extraction date, lab prep batch, Chrom batch signature of analyst at the bottom of each page, lot numbers for standards used, and result of run (O.K., dilution, non-inject, etc.).

10.5.3. The reporting limit is based on the concentration of the lowest standard in the initial calibration, adjusted for the sample wt/vol, final volume, dilution factor and %moisture (No unqualified analytical results or non detects may be reported which correspond to an extract concentration less than the lowest standard in the calibration range).

10.5.4. Any compound concentration that exceeds the concentration of the calibration range must be diluted and re-analyzed.

11.0. Calculations / Data Reduction

11.1. Accuracy:

$$\text{ICV / CCV, LCS \% Recovery} = \frac{\text{observed concentration}}{\text{known concentration}} \times 100$$

$$\text{MS \% Recovery} = \frac{\text{(spiked sample) - (unspiked sample)}}{\text{spiked concentration}} \times 100$$

11.2. Precision (RPD):

$$\text{Matrix Duplicate (MD)} = \frac{|\text{orig. sample value - dup. sample value}|}{[(\text{orig. sample value + dup. sample value})/2]} \times 100$$

11.3. Calculation of Sample Amounts (Internal Standard Procedure)

11.3.1.1 Aqueous Samples

$$\text{Concentration } (\mu g/L) = \frac{(As)(Cis)(D)}{(Ais)(RF)(Vs)\ (Vi)\ (1000)}$$

Where:

As	=	Area of the target analyte peak in the sample
Cis	=	Concentration of the internal standard (ug/L)
D	=	Dilution factor, if the sample or extract was diluted prior to analysis. If no dilution is performed, D = 1.
Vi	=	Volume of the extract injected (ul)
Ais	=	Area of the internal standard peak
RF	=	Average response factor from the initial calibration.
Vs	=	Volume of sample extracted (ml)

The 1000 in the denominator represents the number of ul in 1 ml.

11.3.1.2 Solid Samples

$$\text{Concentration (µg/kg)} = \frac{(As)(Cis)(D)(Vt)}{(Ais)(RF)(Ws)\,(Vi)\,(1000)}$$

Where:

As	=	Area of the target analyte peak in the sample
Cis	=	Concentration of the internal standard (ug/L)
D	=	Dilution factor, if the sample or extract was diluted prior to analysis. If no dilution is performed, D = 1.
Vi	=	Volume of the extract injected (ul)
Ais	=	Area of the internal standard peak
RF	=	Average response factor from the initial calibration.
Vt	=	Volume of concentrated extract (ul)
Ws	=	Weight of sample (g)

The 1000 in the denominator represents the number of ul in 1 ml.

11.4. Relative Response Factors

$$RRF = \frac{A_x}{A_{is}} \times \frac{C_{is}}{C_x}$$

Where:

A_x = Area of target analyte peak
Ais = Area of internal standard peak
Cis = Concentration of internal standard
Cx = Concentration of compound in standard

11.5. Percent Relative Standard Deviation (% RSD):

$$\%\ RSD = \frac{\text{Standard Deviation of RRFs}}{\text{Mean RRF}}$$

11.6. Relative Standard Error (RSE)

$$\%\,RSE = 100\% \times \sqrt{\frac{\sum_{i=1}^{N}\left[\frac{C_i - PC_i}{C_i}\right]^2}{(N-P)}}$$

Where:

N = Number of points in the curve
P = Number of parameters in the curve (= 1 for average response factor, 2 for linear, 3 for quadratic)
CI = True concentration for level i
PCi = Predicted concentration for level i

11.7. Percent Difference (% D):

$$\%\ D = \frac{RRF_c - \overline{RRF_i}}{\overline{RRF_i}} \times 100$$

Where: RRFc = RRF from continuing calibration

$\overline{RRF_i}$ = Mean RRF from current initial calibration

11.8. Percent Recovery (% R): Surrogates and Spikes

$$\text{Recovery (\%)} = \frac{\text{Concentration (or amount) found}}{\text{Concentration (or amount) added}} \times 100$$

11.9. Dry Weight Correction: All solid samples must be corrected for dry weight using the following formula for dry weight determination.

$$DW = \frac{Gd}{Gw} \times 100$$

Where:

DW	=	Percent % Dry Weight
Gd	=	Dry weight of selected sample aliquot
Gw	=	Wet weight of selected sample aliquot

Multiply the DW value times the wet weight of the sample extracted.

NOTE: All dry weight corrections are made in TALS at the time the final report is prepared.

12.0. Method Performance

12.1. Method Detection Limit Study (MDL)

The method detection limit (MDL) is the lowest concentration that can be detected for a given analytical method and sample matrix with 99% confidence that the analyte is present. The MDL is determined according to the laboratory's MDL procedure in Section 20 (Test Methods and Method Validation) of Eurofins Edison's Quality Assurance Manual (ED-QA-LQM). MDLs reflect a calculated (statistical) value determined under ideal laboratory conditions in a clean matrix, and may not be achievable in all environmental matrices. The laboratory maintains MDL studies for analyses performed; these are verified at least annually unless method requirements require a greater frequency.

12.2. Lower Limit of Quantitation (LLOQ) (aka Reporting Limit) Verification:

The LLOQ is the lowest concentration at which the laboratory has demonstrated target analytes can be reliably measured and reported with a certain degree of confidence, which must be ≥ the lowest point in the calibration curve. The lab verifies the LLOQ annually to demonstrate the capability of quantiation at lower analyte concentrations. The verification is performed by the extraction and analysis of an MDL spike at a concentration of 0.5-2 times the established LLOQ. Recovery of target analytes in the LLOQ verification should be within established in-house limits or within other such project-specific acceptance limits to demonstrate acceptable method performance at the LLOQ. Until the laboratory has sufficient data to determine acceptance limits, the LCS criteria ± 20% (i.e., lower limit minus 20% and upper limit plus 20%) will be used for the LLOQ acceptance criteria. The annual LLOQ verification is completed and documented with the required annual MDL evaluation.

12.3. Demonstration of Capabilities

For DOC procedure refer to Section 19 in the most current revision of Eurofins Edison's Quality Assurance Manual (ED-QA-LQM).

12.4. Training Requirements

Refer to Eurofins SOP No. ED-GEN-022, *Training*, for the laboratory's training program.

13.0. Pollution Control

13.1. Pollution prevention encompasses any technique that reduces or eliminates the quantity or toxicity of waste at the point of generation. Numerous opportunities for pollution prevention exist in laboratory operation. The USEPA has established a prevention hierarchy of environmental management techniques that places pollution

prevention as the management option of first choice. Whenever feasible, laboratory personnel should use pollution prevention techniques to address their waste generation. When wastes cannot be feasibly reduced at the source, the agency recommends recycling as the next best option.

13.2. The quantity of chemical purchased should be based on expected usage during its shelf life and disposal cost of unused material. Actual reagent preparation volumes should reflect anticipated usage.

14.0. Waste Management

14.1. The U.S. Environmental Protection Agency requires that laboratory waste management practices conducted be consistent with all applicable rules and regulations. All waste will be disposed of in accordance with Federal, State and Local regulations. Where reasonably feasible, technological changes have been implemented to minimize the potential for pollution of the environment. Employees will abide by this method and the policies in section 13 of the Corporate Safety Manual for "Waste Management and Pollution Prevention."

14.2. The following waste streams are generated as a result of this analysis:

- Expired Standards – The vials are collected in a 1 gallon polyethylene bucket. These vials are then transferred to an open top 55 gallon steel or polyethylene waste drum. These drums are transported to a waste facility for proper disposal.

- Auto sampler vials and expired standards: These vials are collected in satellite accumulation within the instrument laboratory. The vials are then placed into a 55 steel open top drum in the waste room. When the drums are full, the drum will be collected by the waste vendor for disposal. This waste is treated for incineration.

  Teris Profile Number: 50016652
  Onyx Profile WIP Number: 282493

- Mixed Solvent Waste: Mixed solvent waste is collected in a small beaker inside the bench top hood. This waste is then transferred into the satellite accumulation container in the Organic Prep. Lab. on a daily basis. This material is transferred into 5 gallon solvent cans as satellite accumulation. These cans are emptied every 24 hours into a steel drum in the waste room. This drum is kept in the walk in hood until it is full. The full drum is then removed from the hood and placed on secondary containment in the waste room.

  Teris Profile Number: 50016624
  Onyx Profile WIP Number: 545240

- Soil Retain Samples - These samples if not flagged in the system for any hazardous constituents are transferred to poly-lined cubic yard boxes. These boxes when full are sent to stabilization or incineration. These materials are sent out as hazardous for lead and chromium

  Teris Profile Number (incineration): 50016710

Onyx Profile Number: (stabilization) 402535

15.0. References / Cross-References

15.1. United States Environmental Protection Agency, “Method SW8000C: Determinative Chromatographic Separations,”, Test Methods for Evaluating Solid Wastes, SW846, Revision 3, March 2003.

15.2. United States Environmental Protection Agency, “Method 8081B, Organochlorine Pesticide by Gas Chromatography”, Test Methods for Evaluating Solid Wastes, SW846 Third Edition, Volume 1B: Laboratory Manual, Physical/Chemical Methods, Revision 2, February 2007.

15.3. Eurofins Edison Document No. ED-QA-LQM, *Laboratory Quality Manual*, most current revision.

15.4. Eurofins Edison SOP No. ED-ORP-014: *Extraction of Pesticides and PCBs in Water by Separatory Funnel, SW846 Method 3510C,* most current revision.

15.5. Eurofins Edison SOP No. ED-ORP-018*: Extraction of Pesticides/PCBs in Soil Using Low-Level Extraction, SW846 Method 3550B,* most current revision.

15.6. Eurofins Edison SOP ED-ORP-044, *Procedure for the Microwave Extraction of Solids, SW846 3546*

15.7. Eurofins Edison SOP No. ED-ORP-019*: Waste Dilution for Pesticides and PCBs, SW846 Method 3580A,* most current revision.

15.8. Eurofins Edison SOP No. ED-ORP-020*: Florisil Cleanup for Pesticide/PCB Sample Extracts*, SW846 Method 3620B, most current revision.

15.9. Eurofins Edison SOP No. ED-ORP-021: *The Removal of Elemental Sulfur from Pesticide/PCB Sample Extracts, SW846 Method 3660B,* most current revision.

15.10. Eurofins Edison SOP No. ED-GEN-022, *Training,* most current revision.

15.11. Eurofins Corporate Quality SOP No. CA-Q-S-001 (*Solvent & Acid Lot Testing & Approval*), most current revision.

15.12. Eurofins Edison SOP No. ED-GEN-023 (*Bulk Solvent Testing and Approval*), most current revision.

15.13. Eurofins Document CA-Q-W-008, ‘Technical Guidance on the Use and Evaluation of Replicate Continuing Calibration Verification (CCV), most current version.

15.14. Eurofins Corporate Quality SOP No. CA-Q-P-004, “Reporting Results for Methods that Require Second-Column Confirmation”, most current revision.

15.15. Eurofins Corporate Work Instruction No. CA-T-WI-008, “Minimum Requirements for Pesticide Analysis by SW846 Method 8081B”, most current revision.

15.16. Eurofins Corporate Policy CA-T-P-004, "Policy for Determining RT Windows for GC/ECD Tests", most current revision.

15.17. Eurofins Corporate Technical Services Work Instruction No. CA-T-WI-010, "Further Guidance on the RT Window Policy No. CA-T-P-005", most current revision.

16.0. Method Modifications:

None

17.0. Attachments

Attachment 1: Dual Column Reporting Flowchart

18.0. Revision History

Revision 7, dated 10/172022:

- Updated to Eurofins branding throughout document.
- Revised Section 8 to include requirement to treat samples from chlorinated sources with sodium thiosulfate
- Section 9.2.4.3: Included details of evaluation of initial calibration for Relative Standard Error (RSE).
- Section 11.6: added formula for calculation of RSE.
- Section 12.2 added: details the annual LLOQ verification requirement.

Revision 6, dated 03/25/2021:

- Added Section 10.5.3 which details how the reporting limit is established.
- Added Section 10.5.4 which discusses requirement to dilute analytes into range of calibration.

Revision 5, dated 10/12/2020:

- Edited throughout to reflect Eurofins branding and policy.
- Updated Lab Quality Manual section references as needed.

Revision 4, dated 09/13/2016:

- Section 9.2.4.1: completely rewrote this section to reflect requirements of Eurofins Corporate Technical Services Policy No. CA-T-P-005, "Policy for Determining RT Windows for GC/ECD Tests" and TestAmerica Corporate Technical Services Work Instruction No. CA-T-WI-010
- Section 15 (References): added references to TestAmerica Corporate Technical Services Policy No. CA-T-P-005, "Policy for Determining RT Windows for GC/ECD

Tests" and TestAmerica Corporate Technical Services Work Instruction No. CA-T-WI-010, "Further Guidance on the RT Window Policy No. CA-T-P-005".
- Attachment 1: RT Windows for Single Analytes/Surrogates: DELETED and renamed subsequent attachments.

Revision 3, dated 01/05/2016:

- Section 2.2: expanded to include summary of internal standard calibration.
- Section 7.2.2: added 1-Bromo-2-nitrobenzene (internal standard) to the list of standards (Restek 32279). Components and concentration added to table.
- Added Section 7.2.2.4 which describes the preparation of the internal standard solution. All subsequent sections renumbered accordingly.
- Section 7.2.2.1.1: added footnote to ICAL standard prep table detailing requirement to spike each standard with internal standard solution.
- Section 7.2.2.4: describe internal standard spiking protocol (20 ul of 5.0 ug/ml solution into all standards, QC extracts and field sample extracts.
- Section 9.1: added internal standard to 'Sample QC' table.
- Section 9.1.4: added following minimum requirements for surrogate evaluation and reporting: a) Both surrogates must have reportable results that meet the acceptance criteria; b)reported surrogates must be from a column with a passing CCV; c)At least one surrogate must pass on any column from which target analytes are identified and reported. (per corp "Minimum Requirements" document).
- Section 9.1.5: added this section which describes acceptance criteria for internal standards (retention time and response).
- Section 9.2.3: Added the following sentence: "'Samples' here are defined as field samples and batch QC (MB, LCS, MS, MSD) and do not include CCVs, PEMs, instrument blanks, etc… ". Deleted the phrase 'and at the end of every sequence'. (both changes reflect 'Minimum Requirements for Pesticide Analysis' document).
- Added Section 9.2.4.1.3: "Updating absolute retention times: Update retention times with the retention times found in the opening CCV for that 12 hour period."
- Section 9.2.4.2: replaced 'external standard' with 'internal standard'. Added formula for calculation of response factors by internal standard method. Deleted text regarding calibration acceptance criteria from this section as it is duplicated in subsequent sections.
- Section 9.2.4.2.2: added phrase 'for any given compound.'
- Added section 9.2.4.2.5: describes resolution check.
- Sections 9.2.4.3 and 9.2.4.4: added statement concerning 80% resolution requirement.
- Section 9.2.4.4: changed 'ICV' to 'CCV' in last sentence.
- Section 10.1.2.13: Revised first sentence to read: ": HP Chemstation software is used for automation of runs and data acquisition."
- Section 10.2.2: corrected reference to Section 9.2.2.1 (was incorrectly listed as 7.2.2.1).
- Section 10.2.8: revised to clarify that CCV bracketing is not required and that only samples analyzed after a failing CCV must be reanalyzed (i.e., samples analyzed immediately prior to a failing CCV need not be reanalyzed).
- Section 10.3.2: added the following regarding updating retention times: "Retention times are updated using the retention times found in opening CCV for the most recent 12 hour period."

- Section 11: added formulas for calculation of sample concentrations as well as %RSD, %D, % Recovery, RRF and dry weight correction.
- Section 15: removed reference to SOP No. ED-ORP-016*: Automated Soxhlet Extraction of Solid Samples – Pesticides/PCBs, SW846 Method 3541* as it is no longer is use.
- Section 15: added reference to TestAmerica Corporate Quality SOP No. CA-Q-P-004, "Reporting Results for Methods that Require Second-Column Confirmation"
- Section 15: added reference to TestAmerica Corporate Work Instruction No. CA-T-WI-008, "Minimum Requirements for Pesticide Analysis by SW846 Method 8081B".
- Section 15: added reference to TestAmerica Corporate Policy Memorandum No. CA-Q-QM-006, "Technical Guidelines for Analysis of Complex GC/ECD Chromatograms".

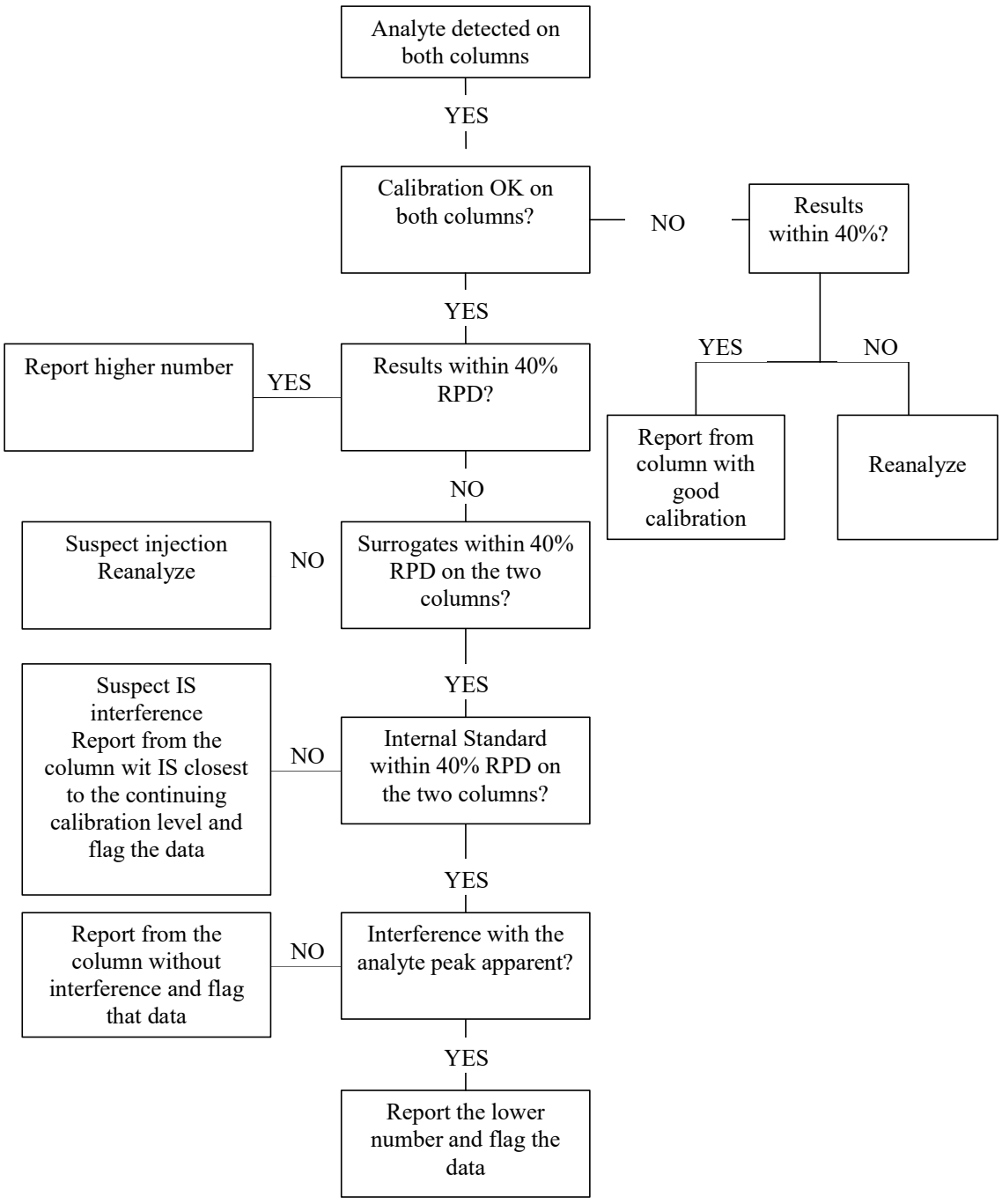
- Revision 2, dated 13 May 2015:
  - Section 1.1: updated RLs to reflect current lab practice.
  - Section 2.1 and throughout: revised initial aqueous/TCLP sample volumes to reflect current lab practice
  - Section 2.1.1: removed note describing option for Reduced Volume Extraction (RVE) as this is now standard lab practice.
  - Section 2.1.2 and throughout document: removed reference to prep by SW3541 as this method is no longer in use at Edison lab.
  - Section 4.3.2 and throughout: updated source of standards used for Florisil check.
  - Section 6.1.2.3.1: updated name of analytical GC columns currently in use.
  - Section 6.1.2.4.1: updated make-up gas to P-5 & Nitrogen.
  - Section 7.1.1: updated carrier gas to Hydrogen and make-up gas to P-5&Nitrogen.
  - Section 7.2.2 and throughout document: updated source and catalog numbers for analytical standards (Restek is now primary source of standards).
  - Section 7.2.2.1: updated standards prep instructions. Removed outdated references to standards and spiking mixes. Removed notes pertaining to RVE since this procedure is now standard and incorporated into instructions.
  - Section 7.2.2.10: added instructions for preparation of ICVs.
  - Section 8.1: adjusted water sample container from 1000ml to 250ml.
  - Section 9.2.2: removed references and instructions pertaining to the Pesticide Resolution Check (a CLP requirement we no longer perform).
  - Section 9.2.3.1: Added extensive discussion regarding the analysis of replicated CCVs.
  - Section 9.2.4.4: updated table to include new concentration for low standard (2.5 ppb).
  - Section 10.1.2.14 and throughout document: replaced references to Target with TestAmerica Chrom.
  - Section 10.2: made extensive revisions to discussion of the analytical sequence (removing duplicate entries and adjusting to reflect current procedures).
  - Section 10.3: added note concerning NJ DKQP requirements for reporting highest concentration of dual columns.
  - Section 15 (References): updated to include "Technical Guidance on the Use and Evaluation of Replicate Continuing Calibration Verification (CCV), most current version."
  - Added Sections 7.2.2.11 and 7.2.2.12 reflecting the addition and preparation of Internal standard

- Revision 1, dated 09 October 2012
  - Throughout: Revised LQM section number references to reflect the most current LQM revision.
  - Section 2.1.1: added description of reduced initial volume (125ml)/final volume (1ml) option.
  - Section 2.1.2: Added reference to TestAmerica Edison SOP ED-ORP-044, *Procedure for the Microwave Extraction of Solids, SW846 3546.*
  - Section 7.2.2.1.1 added preparation of 5ppb standard for the reduced volume method.
  - Section 9.2.2.2 Added 5ppb standard for the reduced volume method
  - Section 9.2.4.4 Added 5 ppb standard to the initial calibration for the reduced volume method
  - Section 10.2.6 Added 5 ppb to the Analytical sequence for the low level method Section 15.0: Removed reference to TestAmerica Edison SOP EDS-GEN-019, *Organic Calculations,* most current revision.
  - Section 15.0: Added reference to TestAmerica Edison SOP ED-ORP-044, *Procedure for the Microwave Extraction of Solids, SW846 3546*

- Revision 0, dated 02/17/2011: New

ATTACHMENT 1

Dual Column Reporting Flowchart
(Note: NJDEP DKQP requires reporting the higher concentration in all instances)



Title: Method SW846 3060A, The Alkaline Digestion of Soil and Wipe Samples for the Analysis of Hexavalent Chromium

Once printed, this is considered an uncontrolled document

Approvals (Signature/Date):

Jasmine Parillo 01/07/21	Dan Helfrich 01/07/21
Jasmine Parillo Date	Dan Helfrich Date
Department Manager	Health & Safety Manager /Coordinator
Carl Armbruster 01/07/21	01/07/21
Carl Armbruster Date	Mark Acierno Date
Quality Assurance Manager	Laboratory Director

1.0 Scope and Application

1.1 Analytes, Matrix(s), and Reporting Limits

1.1.1 SW846 Method 3060A is an alkaline digestion procedure for extracting Hexavalent Chromium [Cr(VI)] from soluble, adsorbed, and precipitated forms of chromium compounds in soils, sludges, sediments, and similar waste materials. Materials applicable to this method include soil, sediment, debris, brick, concrete, sludge, and precipitate. TestAmerica Edison has modified the method to include digestion of surface wipe samples. To quantify total Cr(VI) in a solid matrix, three criteria must be satisfied: 1) the extracting solution must solubilize all forms of Cr(VI), 2) the conditions of the extraction must not induce reduction of native Cr(VI) to Cr(III), and 3) the method must not cause oxidation of native Cr(III) contained in the sample to Cr(VI). Method 3060A meets these criteria for a wide spectrum of solid matrices.

1.1.2 Method 3060A digestates can be analyzed for hexavalent chromium using Method 7196 (colorimetrically by UV-VIS spectrophotometry). Reference the applicable analytical method SOP for details on reporting limits.

1.1.3 On occasion clients may request modifications to this SOP. These modifications are handled following the procedures outlined in Sections 7 (Review of Work Request) and 19 (Test Methods and Method Validation) of TestAmerica Edison's Quality Assurance Manual (ED-QA-LQM).

2.0 Summary of Method

2.1. This method uses an alkaline digestion to solubilize both water-insoluble (with some exceptions) and water soluble Cr(VI) compounds in solid waste samples. The pH of the digestate must be carefully adjusted during the digestion procedure. The sample is digested using 0.28M Na_2CO_3/0.5M NaOH solution and heating at 90-95ºC for 60 minutes to dissolve the Cr(VI) and stabilize it against reduction to Cr(III).

2.2. Under the alkaline conditions of the extraction, minimal reduction of Cr (VI) or oxidation of native Cr(III) occurs. The addition of Mg^{+2} in a phosphate buffer to the alkaline solution has been shown to suppress oxidation, if observed. The accuracy of the extraction procedure is assessed using spike recovery data for soluble and insoluble forms of Cr(VI) (e.g. K_2Cr2O_7 and $PbCrO_4$), coupled with measurement of ancillary soil properties, indicative of the potential for the soil to maintain a Cr(VI) spike during digestion, such as oxidation reduction potential (ORP), pH, organic matter content, ferrous iron, and sulfides. Recovery of an insoluble Cr(VI) spike can be used to assess the first two criteria, and method-induced oxidation is usually not observed except in soils high in Mn and amended with soluble Cr(III) salts or freshly precipitated $Cr(OH)_3$.

3.0 Definitions

For a complete list of definitions refer to Appendix 5 in the most current revision of TestAmerica Edison's Quality Assurance Manual (ED-QA-LQM).

4.0 Interferences

4.1 When analyzing a sample digestate for total Cr (VI), the reducing/oxidizing tendency of each sample matrix can be determined at the client's request. Establishing the tendency of Cr(VI) to exist or not exist in the unspiked sample(s), assists in the interpretation of QC data for matrix spike recoveries outside conventionally accepted criteria for total metals.

4.1.1 This can be accomplished by characterization of each sample for additional analytical parameters such as pH, ferrous iron, sulfides, and Oxidation Reduction Potential. Other indirect indicators of reducing/oxidizing tendency include Total Organic Carbon (TOC), Chemical Oxygen Demand (COD), and Biological Oxygen Demand (BOD).

4.2 Certain substances, not typically found in the alkaline digests of soils, may interfere in the analytical methods for Cr(VI) following alkaline extraction if the concentrations of these interfering substances are high and the Cr(VI) concentration is low. Refer to the SOP for Method 7196A for the specific agents that may interfere with Cr(VI) quantification.

4.3 For waste materials or soils containing soluble Cr(III) concentrations greater than four times the laboratory Cr(VI) reporting limit, Cr(VI) results obtained using this method may be biased high due to method-induced oxidation. The addition of Mg^{2+} in a phosphate buffer to the alkaline extraction solution has been shown to suppress this oxidation

4.4 A sample containing a high buffering capacity may need to be digested using additional digestion solution.

4.5 It is very important to monitor the temperature of the digestion solution during the digestion to maintain a temperature of 90-95^{o}C during the entire time period.

4.6 Do not use Ghost wipe, Whatman, mixed cellulose ester (MCE) or glass fiber filters for sampling wipes because they convert Cr(VI) to Cr(III). Use either PVC filters or binderless quartz fiber filters.

5.0 Safety

Employees must abide by the policies and procedures in the Corporate Environmental Health and Safety Manual (CW-E-M-001) and this document. This procedure may involve hazardous material, operations and equipment. This SOP does not purport to address all of the safety problems associated with its use. It is the responsibility of the user of the method to follow appropriate safety, waste disposal and health practices under the assumption that all samples and reagents are potentially hazardous. Safety glasses, gloves, lab coats and closed-toe, nonabsorbent shoes are a minimum.

5.1. Specific Safety Concerns or Requirements

Samples that contain high concentrations of carbonates or organic material or samples that are at elevated pH can react violently when acids are added.

5.2. Primary Materials Used

The following is a list of the materials used in this method, which have a serious or significant hazard rating. **Note: This list does not include all materials used in the method. The table contains a summary of the primary hazards listed in the MSDS for each of the materials listed in the table.** A complete list of materials used in the method can be found in the reagents and materials Section. Employees must review the information in the MSDS for each material before using it for the first time or when there are major changes to the MSDS.

Material (1)	Hazards	Exposure Limit (2)	Signs and symptoms of exposure
Nitric Acid	Corrosive Oxidizer Poison	2 ppm-TWA 4 ppm-STEL	Nitric acid is extremely hazardous; it is corrosive, reactive, an oxidizer, and a poison. Inhalation of vapors can cause breathing difficulties and lead to pneumonia and pulmonary edema, which may be fatal. Other symptoms may include coughing, choking, and irritation of the nose, throat, and respiratory tract. Can cause redness, pain, and severe skin burns. Concentrated solutions cause deep ulcers and stain skin a yellow or yellow-brown color. Vapors are irritating and may cause damage to the eyes. Contact may cause severe burns and permanent eye damage.
Potassium Dichromate	Oxidizer Corrosive Carcinogen	0.1 Mg/M3 TWA as CrO3	Extremely destructive to tissues of the mucous membranes and upper respiratory tract. May cause ulceration and perforation of the nasal septum. Symptoms of redness, pain, and severe burn can occur. Dusts and strong solutions may cause severe irritation. Contact can cause blurred vision, redness, pain and severe tissue burns. May cause corneal injury or blindness.

6.0 Equipment and Supplies

6.1. Equipment

- Digestion vessel: water bath equipped with shaker (Precision, Model 50)
- pH meter: Orion benchtop or equivalent
- Vacuum filtration apparatus
- Analytical balance capable of accurate weighings to 0.01 grams

6.2. Supplies

- Class A volumetric flasks with stoppers
- Filter paper 0.45 and 0.1 um size.
- Nalgene 115-ml filter disposable unit with 0.45um filter
- Binderless quartz fiber filter, 37 or 47 mm diameter, 0.45 mm thick (for wipe analysis)
- Digestion container: 125ml Nalgene small-mouth polypropylene bottles
- Certified thermometer (NIST traceable)-capable of reading 100ºC
- Finnpipet (1-5 ml) with tips
- Graduated cylinder (class A)
- Specimen cups
- Transfer pipettes
- Wood spatulas

7. Reagents and Standards

7.1. Reagents

7.1.1. The following Reagent grade chemicals are used for all solutions required in this test. For stability information, refer to manufacturer's instructions or 2 years from date opened if no instructions are available. All reagents are stored at room temperature:

7.1.1.1. Concentrated Nitric acid
7.1.1.2. Anhydrous Sodium Carbonate
7.1.1.3. Sodium Hydroxide
7.1.1.4. Potassium Dichromate
7.1.1.5. Magnesium Chloride-$MgCl_2$ (anhydrous)
7.1.1.6. Potassium Phosphate, Dibasic
7.1.1.7. Potassium Phosphate, Monobasic

7.1.2. Phosphate Buffer- 0.5M K_2HPO_4/0.5M KH_2PO_4 buffer at pH 7: Dissolve 87.09g K_2HPO_4 and 68.04g KH_2PO_4 into 700ml reagent water. Bring up to a final volume of 1 liter. Store at room temperature. Stable for 6 months.

7.1.3. Digestion Solution: Weigh 20g of NaOH and 30g of Na_2CO_3 and dissolve in a 1 liter volumetric flask using Type I water. Dilute to the mark and then transfer the solution to a tightly capped polyethylene bottle. Store at room temperature. Prepare fresh monthly. **Note**: Check the pH of the digestion solution to be certain it has a pH of greater than 11.5 before using it. If the pH drops below 11.5, a new solution must be prepared.

7.1.4. 1:1 HNO3: Add 500ml DI to a 1 Liter volumetric flask and then slowly add 500ml of concentrated HNO3. Solution is stable for 6 months. Store at room temperature.

7.2. Standards

7.2.1. Potassium Dichromate Primary Standard Solution (1000 mg/L Cr+6):

Weigh 0.2829 g of Potassium Dichromate ACS grade, J.T. Baker, cat# 3093-01 (dried for 1 hour at 105ºC) and dissolve in deionized water in a 100 ml flask. Dilute to the mark. Store at room temperature. Solution is stable for 6 months.

7.2.2. Potassium Dichromate Secondary Standard Solution (1000 mg/L Cr+6): Weigh 0.2829 g of Potassium Dichromate ACS grade, J.T. Baker, cat# 3090-04 (dried for 1 hour at 105ºC) and dissolve in deionized water in a 100 ml flask. Dilute to the mark. **Note**: Prepare the Secondary standard solution from a different source than what was used to prepare the primary standard solution. Stable for 6 months, store at room temperature.

7.2.3. Hexavalent Chromium Primary Spiking Solution (100 mg/L Cr+6) Prepare by adding 10 ml of 1000 mg/L Cr+6 primary standard (Section 7.2.1) to a 100 ml volumetric flask and dilute to volume with deionized water. Stable for 6 months, store at room temperature.

7.2.4. Hexavalent Chromium Secondary Spiking Solution (100 mg/L Cr+6) Prepare by adding 10 ml of 1000 mg/L Cr+6 Secondary standard (Section 7.2.2) to a 100 ml volumetric flask and dilute to volume with deionized water. Stable for 6 months, store at room temperature.

7.2.5. Lead Chromate-AR Grade; for storage and stability information, refer to manufacturer's instructions

8. Sample Collection, Preservation, Shipment and Storage

Sample container, preservation techniques and holding times may vary and are dependent on sample matrix, method of choice, regulatory compliance, and/or specific contract or client requests. Listed below are the holding times and the references that include preservation requirements.

Matrix	Sample Container[1]	Min. Sample Size	Preservation	Holding Time[3]	Reference
Soils	Glass or plastic	2.50g[2]	Cool 4 ±2ºC	30 Days	SW 846 Method 3060A
Wipes[4]	Glass or plastic	1 wipe	Cool 4 ±2ºC	30 Days	SW 846 Method 3060A

[1] Containers should not contain stainless steel
[2]Additional volume may be required if the sample is chosen for QC or if a re-prep is required.
[3]The alkaline digestate must be analyzed within 168 hours after extraction from the soil or wipe.
[4]In chrome plating environments, wipe samples taken on a PVC filter or an uncoated binderless quartz fiber filter, should be placed in a vial containing 5 mL of an aqueous solution containing 10% Na_2CO_3 with 2% $NaHCO_3$ immediately after sampling to eliminate the interference from the acid used in the chrome plating process. An alternate medium

which does not require extraction in the field is a binderless quartz fiber filter coated with 1% NaOH.

9. Quality Control

9.1. Sample QC - The following quality control samples are prepared daily with each batch of 20 samples or less, whichever is more frequent. Soil and wipe samples must be divided into separate QC batches.

9.1.1. Method Blank:

- *Soil Samples:* One method blank must be digested and analyzed per soil batch. A method blank consists of 50 ml digestion solution and is carried through the entire analytical procedure. The concentration of the method blank must be less than the reporting limit or the entire batch must be re-digested and re-analyzed.

- *Wipe Samples:* One method blank must be digested and analyzed per wipe batch. The method blank will consist of a blank wipe and 50 ml digestion solution and will be carried through the entire analytical procedure. The concentration of the method blank must be less than the reporting limit or the entire batch must be re-digested and re-analyzed.

9.1.2. Laboratory Control Sample (Soils and wipes): Laboratory control samples from Secondary sources, shall be digested and analyzed per batch as follows:

9.1.2.1 Soil LCS:

- **LCS Soluble (LCSSRM):** Obtained from an independent source (ERA). Prepare the LCS soluble solution by diluting the concentrated LCS solution as indicated in the manufacturer's instructions. If necessary, prepare a different dilution so that the concentration after analysis falls within the calibration curve. Add 5ml of the diluted LCS soluble solution into 50ml digestion solution. The results must be within vendor specified QC limits or the entire batch must be re-digested and re-analyzed. Document the preparation of the LCS soluble solution in the TALS reagent module.

- **LCS Insoluble (LCSI):** Prepared by adding 0.011g of PbCrO4 into 50ml digestion solution. The results must be within 80-120% of the true value or the entire batch must be re-digested and re-analyzed.

- **LCS matrix matched (LCSSRM):** Obtained from an independent source (ERA). Add 2.5g of the soil LCS into 50ml digestion solution. The results must be within vendor specified QC limits or the entire batch must be re-digested and re-analyzed. This LCSSRM is included in DKQP guidelines however it is not reported to clients.

9.1.2.2 Wipe LCS/LCSD:

For wipe sample batches, a Laboratory Control Sample (LCS)/Laboratory Control Sample Duplicate must be prepared using the LCS soluble solution. Add a blank wipe to the digestion bottles for each of the LCS and LCSD and follow the prep for LCS Soluble detailed in Section 9.1.2.1 above.

9.1.3. **Predigestion Matrix Spikes (Soils only):** Both the soluble and insoluble pre-digestion matrix spikes must be analyzed per batch of ≤ 20 soil samples.

9.1.3.1. **Soluble Matrix Spike (MSS):** spike the sample with 1.0 ml of the 100 ppm Cr (VI) primary spiking solution prepared in Section 7.2.3 (equivalent to 40mg/kg Cr (VI)).

9.1.3.2. **Insoluble Matrix Spike (MSI):** to a separate sample aliquot add 0.011g of $PbCrO_4$. It is used to evaluate the dissolution during the digestion process.

9.1.3.3. Both matrix spikes are then carried through the digestion process. The acceptance range for the matrix spike recoveries is 75-125%. If the matrix spike recoveries fall outside these recovery limits, the entire batch must be rehomogenized, redigested, and reanalyzed.

9.1.3.4. If either matrix spike recovery is outside of the acceptable range of 75-125% but, the LCS is within the criteria specified in Section 9.1.2, additional laboratory characterization of each sample in the batch for ORP and pH can be performed at the client's request to determine if the sample exhibits reducing conditions. Further characterization for total sulfides, total organic carbon, chemical oxygen demand, or biological oxygen demand may be required by the client.

9.1.4. **Duplicate (DU) (Soils only):** One duplicate laboratory sample must be analyzed per soil batch. The sample used for the predigestion spike should be used for this purpose. Duplicate samples must have a Relative Percent Difference (RPD) of ≤20%, if both the original and the duplicate are ≥ four times the laboratory reporting limit. A control limit of ± 2.0mg/kg (laboratory reporting limit) is used when either the original or the duplicate sample is < four times the laboratory reporting limit.

9.1.5. **Post Digestion Spike (PDS) (Soils only):** Following the analysis (colorimetric determination), a post-digestion spike must be analyzed per soil batch. The spike concentration must be equivalent to 40mg/kg or twice the sample concentration, whichever is greater. The post digestion spike must be performed on a field sample, not on a field blank or preparation blank. It would be helpful to perform this analysis on the sample used for the matrix spike. Recovery limits for the post-digestion spike are 85-115%. If the post digestion spike fails to meet the recovery limits, a new aliquot of the sample

must be re-spiked and re-analyzed as described in SOP ED-WET-011 (The Analysis of Digestates for Hexavalent Chromium by SW846 7196A).

9.2. Instrument QC

See applicable analytical SOP, ED-WET-011 (The Analysis of Digestates for Hexavalent Chromium by SW846 7196A).

10. Procedure

10.1. Sample Preparation

10.1.1. Homogenize soil samples following TestAmerica Edison SOP ED-GEN-007 (Subsampling).

10.1.2. Weigh 2.5g ± 0.10g of sample into a clean labeled 125ml nalgene bottle. Record weight up to two decimal places. For wipe samples, add 1 sample wipe into a clean labeled 125 ml nalgene bottle.

10.1.3. Add 50 +/- 1.0ml of digestion solution to each sample. Then add 400mg of MgCl2 and 0.5ml of 1.0M Phosphate Buffer.

10.1.4. Matrix spike preparation (soils only): Spike the samples designated MS soluble and MS insoluble as detailed in Section 9.1.3. Add 50 +/- 1.0ml of digestion solution to each sample followed by 400mg of MgCl2 and 0.5ml of 1.0M Phosphate Buffer.

10.1.5. LCS preparation: For soil samples, prepare as detailed in Section 9.1.2.1. For wipe samples, prepare as detailed in Section 9.1.2.2. Add 400mg of MgCl2 and 0.5ml of 1.0M Phosphate Buffer.

10.1.6. Place all samples in hot water bath at a temperature of 90-95ºC and begin shaking at 175rpm for 5 minutes. Then release pressure by venting the caps. After the caps are resealed tightly, begin heating and shaking for 60 minutes. Start timing after the water bath has returned to 90-95ºC and maintain this temperature throughout the 60 minute digestion.

10.1.7. Measure and record the digestion start time and temperature, time and temperature after 30 minutes, and the digestion end time and temperature. Use a calibrated thermometer to measure the temperature of one of the samples in the water bath.

10.1.8. Following the heated digestion, gradually cool the solution to room temperature and quantitatively transfer the contents of the bottle to the vacuum filtration apparatus with Type I water rinses. Filter the digestate through a 0.45um filter (commercially purchased filter). Transfer the rinsates and filtrate to a clean labeled 100ml plastic cup. (**Note**: Use a polyethylene bottle if the colorimetric analysis is not following immediately).

10.1.9. Determine the concentration of hexavalent chromium in the sample extract using Method 7196A USEPA SW846 3rd Edition, (TestAmerica Edison SOP ED-WET-011, The Analysis of Digestates for Hexavalent Chromium by SW846 7196A).

10.2. Calibration:

Refer to the applicable analytical SOPs: TestAmerica Edison SOP ED-WET-011, The Analysis of Digestates for Hexavalent Chromium by SW846 7196A.

10.3. Sample Analysis

Refer to the applicable analytical SOPs: TestAmerica Edison SOP ED-WET-011, The Analysis of Digestates for Hexavalent Chromium by SW846 7196A.

11.0. Calculations / Data Reduction

11.1. Calculations:

Refer to the applicable analytical SOP (ED-WET-011) for calculations.

11.2. Data Reduction:

11.2.1. All sample preparation information is recorded directly in the logbook and in TALS at the time of sample preparation.

11.2.2. Record reagent information in the prep batch information (this can be viewed in the “batch information” page).

11.2.3. Record special comments and observations in the ‘worksheet’ tab.

11.2.4. The logbook pages for the prep are attached to the analytical batch as pdf files.

11.2.5. Complete the Data Review Checker (DRC) in TALS: Prior to data submission the analyst must execute the DRC (Data Review Checker) program in the applicable analytical batch (TALS Analyst Desktop II module).

11.2.5.1. Open the analytical batch and click on the Edit tab above to enter the Edit Mode.

11.2.5.2. Press F8 command key on the computer keyboard or right click anywhere on the batch worksheet then click on ‘Run checklist.’

11.2.5.3. Acknowledge by filling in responses to all unacknowledged findings. Highlight the checklist with findings, then highlight the associated list of findings; right click and choose ‘Acknowledge Item.’

11.2.5.4. Fill in appropriate comments in the response box, then hit 'OK.' 'The column labeled 'unacknowledged findings' should show '0' for all questions once completed; then batch is ready for 2nd level review.

12.0. Method Performance

12.1. Method Detection Limit Study (MDL)

The method detection limit (MDL) is the lowest concentration that can be detected for a given analytical method and sample matrix with 99% confidence that the analyte is present. The MDL is determined according to the laboratory's MDL procedure in Section 19 (Test Methods and Method Validation) of TestAmerica Edison's Quality Assurance Manual (ED-QA-LQM). MDLs reflect a calculated (statistical) value determined under ideal laboratory conditions in a clean matrix, and may not be achievable in all environmental matrices. The laboratory maintains MDL studies for analyses performed; these are verified at least annually unless method requirements require a greater frequency.

12.2. Demonstration of Capabilities

For DOC procedure refer to Section 19 in the most current revision of TestAmerica Edison's Quality Assurance Manual (ED-QA-LQM).

12.3. Training Requirements

Refer to TestAmerica SOP No. ED-GEN-022, Training, for the laboratory's training program.

13.0. Pollution Control

It is TestAmerica's policy to evaluate each method and look for opportunities to minimize waste generated (i.e., examine recycling options, ordering chemicals based on quantity needed, preparation of reagents based on anticipated usage and reagent stability). Employees must abide by the policies in Section 13 of the Corporate Environmental Health and Safety Manual (CW-E-M-001) for "Waste Management and Pollution Prevention."

14.0. Waste Management

14.1. Waste management practices are conducted consistent with all applicable rules and regulations. Excess reagents, samples and method process wastes are disposed of in an accepted manner. Waste description rules and land disposal restrictions are followed. Waste disposal procedures are incorporated by reference to TestAmerica SOP No. ED-SPM-008 (Laboratory Waste Disposal Procedures).

14.2. The following waste streams are produced when this method is carried out:

- Soil Retain Samples - These samples if not flagged in the system for any hazardous constituents are transferred to poly-lined cubic yard boxes. These

boxes when full are sent to stabilization or incineration. These materials are sent out as hazardous for lead and chromium.

Teris Profile Number (incineration): 50016710
Onyx Profile Number: (stabilization) 402535

- Sample waste-After analysis, the samples and standards are collected in a polyethylene container labeled 'Cr (VI) waste.' Once the container is full, it is brought to the waste room for disposal.

15.0. References / Cross-References

15.1. Test Methods for Evaluating Solid Waste, Physical/Chemical Methods, 3rd ed., U.S. Environmental Protection Agency, Office of Solid Waste and Emergency Response. U.S. Government Printing Office: Washington, DC, 1995; SW-846, Method 3060A.

15.2. Occupational Health and Safety Administration (OSHA) Method W4001, Hexavalent Chromium, Methods Development Team, OSHA Salt Lake Technical Center, Salt Lake City, UT, April 2001.

15.3. TestAmerica Edison Document No. ED-QA-LQM, *Laboratory Quality Manual*, most current revision.

15.4. TestAmerica Edison SOP ED-GEN-022, *Training*, most current revision.

15.5. TestAmerica Edison SOP No. ED-SPM-008, *Laboratory Waste Disposal Procedures*, current revision.

15.6. Test America Edison SOP ED-WET-011, The Analysis of Digestates for Hexavalent Chromium by SW846 7196A, most current revision.

16.0. Method Modifications:

The method has been modified to include procedures specific to the digestion of wipe samples.

17.0. Attachments

N/A

18.0. Revision History

- **Revision 13.1, dated 08 January 2021**
  - Updated SOP header with Eurofins emblem; no procedural change.
  - Sec 3: Revised the Definitions location as per the most current LQM.

- **Revision 13, dated 14 February 2019**

  - Sec. 9.1.2: Added matrix matched LCS applicable to DKQP projects.
  - Sec. 11.2.5: Revised the use of data review paper checklist to TALS DRC review, instructions added.
  - Deleted reference WI# EDS-WI-008 in Sec 15.7, not applicable.
  - Updated approval signatures to current.

- **Revision 12, dated 20 November 2014**

  - Removed references to SW846 7199 throughout the SOP; laboratory no longer perform this method.
  - Sec. 9.1.2: Revised the LCSS limits from +/-15% to vendor's certified limits to reflect actual laboratory practices.
  - Sec. 9.1.3.3: Removed notation that redigestion/reanalysis of batch is only for NJ sites or with PM/client's advice; Re-digestion and reanalysis is performed for all failed QC batch and is a method requirement.
  - Sec. 10.1.7: Revised procedures for checking sample temperature during sample digestion; temperature of the sample will be checked at start, middle and end of digestion.

- **Revision 11, dated 11 December 2012**

  - Sec 1 and 12: Updated LQM section references to reflect the most current LQM revision.
  - Sec 9.1.2 (LCSI) and 9.1.3.2 (MSI): Deleted 0.012g to reflect actual laboratory practices.

- **Revision 10, dated 18 November 2010**

  - Section 1.1: included statement regarding the modification of the method for preparation of wipe samples.
  - Section 1.1.2: removed soil RL details and included statement referring reader to the applicable method SOP for RL details.
  - Section 4.1: Oxidizing/reducing parameters will be analyzed if requested by the client.
  - Section 4.6: Added interferences for wipe samples.
  - Section 6.2: Removed bottle top dispenser from list of supplies. Added graduated cylinder, specimen cups, transfer pipettes, and binderless quartz fiber filters to the list of supplies.
  - Section 7.1: Reformatted Reagents Section. Added Potassium Phosphate, Dibasic and Potassium Phosphate, Monobasic to list of reagents.
  - Section 8.0: Added wipes to the sample collection, preservation, shipment, and storage Section.

- Section 9.1: Modified Sample QC Section to specify wipe criteria. Added Sections 9.1.2.1 and 9.1.2.2 to differentiate the LCS procedures for soils and wipes.
- Section 9.1.3.4: Section was revised to specify the additional laboratory characterization only when required by the client.
- Sections 9.1.3, 9.1.4 and 9.1.5: added language to clarify that these QC types apply only to soils (and not to wipes).
- Section 10.1.2: added procedures for wipe sample preparation.
- Section 10.1.5 and 10.1.6: added to detail the QC sample (LCS and matrix spike) prep procedures for soils and wipes.
- Section 11.0: Replaced all calculations with a reference to the applicable analytical method SOP. Add
- Section 15.0: Added reference for OSHA Method W4001, Hexavalent Chromium (wipe sample methodology).
- Section 16.0: added method modification language for addition of wipe samples.

- **Revision 9, dated 03 February 2010**
  - Section 9.1.3.1: Changed MS to MSS to be consistent with TALS naming convention.
  - Section 10.1.3: Added Section (previously part of Section 10.1.4) to clarify that spike should be added directly to the sample aliquot prior to the addition of the digestion solution, magnesium chloride, and phosphate buffer. Subsequent Sections adjusted.
  - Section 10.1.4: Section added to include LCS soluble and LCS insoluble. Subsequent Sections adjusted.

- **Revision 8, dated 25 August 2009**
  - Revised SOP format in accordance with TestAmerica Corporate Quality SOP No. CW-QS-002 (Rev 0) Writing a Standard Operating Procedure (SOP).
  - Section 1.1.2: Added reporting limit.
  - Section 7.1.8: Added prep information.
  - Removed midpoint calibration checks standard (CCS) and Calibration blank in the QC Section, both QC samples are not applicable to this SOP.
  - Deleted pH adjustment procedure, previously named Section 9.6; this procedure will be included in the analytical SOP.
  - Section 15: Added applicable references.

- **Revision 7b, dated 30 April 2009**
  - Section 5.6: Added stability and storage information.
  - Section 5.7: Added stability information
  - Section 5.8: Added stability information

- Section 10.8: Added information about the pH adjusted PS to conform to method requirements.
- Section 15: Added applicable references.

- **Revision 7a, dated 10 February 2009**

  - Section 4.4: Add filter size to Nalgene 115-ml disposable filter.
  - Section 4.10: Add Bottle top dispenser use for adding the digestion solution.
  - Section 4.11: Add Wood spatulas; Use wooden spatulas instead of the stainless steel spatulas.
  - Section 6.1: Delete pre-made standard purchased from Inorganic Ventures. Cr6+ Primary Standard will be prepared in the lab.
  - Section 6.2: Specified source of the 1000 ppm Cr6+
  - Section 6.3 and 6.6: Delete 10mg/l Cr6+ standards; Working standard 10mg/L Cr+6 is not utilize for soil analysis.
  - Section 10.3: expanded LCS information to include preparation information for both LCSs.
  - Section 10.4.2: Specified the exact amount of PbCrO4 added to the insoluble matrix spike.
  - Section 6.5: Added storage information.
  - Section 6.4: Added text: source must be different from the primary standard.
  - Section 9.3: Expanded Section to include the spiking of LCS and MS.
  - Section 9.4: Expanded Section to include recording specific information for the water bath.

- **Revision 7, dated March 2007**

  - Section 10.3: Revised to specify the types of LCS used including their source and acceptance limits.
  - Section 10.6: Deleted the requirement that ‘CCS is taken through the entire digestion process to reflect actual laboratory practices.

- **Revision 6, dated 30 October 2006**

  - Section 9.6.3: Corrected referenced SOP number from EDS-WET-010 to EDS-WET-011.
  - Section 10.5:Revised first sentence to read “One duplicate laboratory sample per batch *must* be analyzed.” (‘Must’ previously read ‘should’).
  - Section 10.4.3: The text in italics was added to the following sentence in Section 10.4.3:
    “NOTE: If the spiked sample concentration is greater than 4x the predigestion spike concentration, no redigestion/reanalysis is required unless the samples are from a NJ site in which case the lab project manager should be contacted for further guidance.”
  - Section 10.8: Added the following text:
    “If the Post Digestion Spike fails to meet recovery limits, a new aliquot of the sample must be re-spiked and re-analyzed.

Title: The Analysis of Digestates for Hexavalent Chromium by EPA SW846 7196A

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Approvals (Signature/Date):

Jasmine Parillo 06/16/20	Dan Helfrich 06/17/20
Jasmine Parillo Date	Dan Helfrich Date
Department Manager	Health & Safety Manager /Coordinator
Carl Armbruster 06/16/20	Mark Acierno 06/17/20
Carl Armbruster Date	Mark Acierno Date
Quality Assurance Manager	Laboratory Director

Facility Distribution No. Electronic **Distributed To:** Facility Intranet

1.0 Scope and Application

1.1 Analytes, Matrix(s), and Reporting Limits

1.1.1 This SOP is applicable to the determination of hexavalent chromium digestates of solid samples using SW846 Method 7196A. Solid samples must be first digested according to Method 3060A USEPA SW846 3rd Edition, TestAmerica Edison SOP ED-WET-010 (The Alkaline Digestion of Soil Samples for the analysis of hexavalent chromium).

1.1.2 The laboratory's reporting limit for hexavalent chromium in soil is 2.0 mg/kg.

1.1.3 On occasion clients may request modifications to this SOP. These modifications are handled following the procedures outlined in Sections 7 (Review of Work Request) and 19 (Test Methods and Method Validation) of TestAmerica Edison's Quality Assurance Manual (ED-QA-LQM).

2.0 Summary of Method

Solid samples are digested utilizing SOP ED-WET-010, alkaline digestion of soil samples via Method 3060A. Following the sample preparation and digestion procedure, dissolved hexavalent chromium is determined spectrophotometrically at 540 nm by reaction with diphenylcarbazide in acid solution.

3.0 Definitions

For a complete list of definitions refer to Appendix 2 in the most current revision of TestAmerica Edison's Quality Assurance Manual (ED-QA-LQM).

4.0 Interferences

4.1 Molybdenum and Mercury react to form color with diphenylcarbazide, however the intensity of color is much lower than those for chromium at the specified pH. Concentrations up to 200 mg/L can be accepted.

4.2 Vanadium interferes strongly, but can be tolerated up to 10 times the concentration of Cr (VI) present.

4.3 Iron may cause a yellow color but should not affect the colorimetric measurement at 540 nm.

4.4 Reducing matter may reduce hexavalent Cr to trivalent Cr in varying amounts. No preventative measure is available at this time; however, interference is checked by post digestion spike samples.

4.4.1 The reducing/oxidizing tendency of each matrix may be determined by characterization of samples for additional analytical parameters, such as pH, ferrous iron, sulfides, and Oxidation Reduction Potential. Other indirect indicators of reducing/oxidizing tendency include Total Organic Carbon

(TOC), Chemical Oxygen Demand (COD), and Biological Oxygen Demand (BOD).

4.5 For waste materials or soils containing soluble Cr (Ill) concentrations greater than four times the laboratory Cr (VI) reporting limit, Cr (VI) results obtained using this method may be biased high due to method-induced oxidation.

5.0 Safety

Employees must abide by the policies and procedures in the Corporate Environmental Health and Safety Manual (CW-E-M-001) and this document. This procedure may involve hazardous material, operations and equipment. This SOP does not purport to address all of the safety problems associated with its use. It is the responsibility of the user of the method to follow appropriate safety, waste disposal and health practices under the assumption that all samples and reagents are potentially hazardous. Safety glasses, gloves, lab coats and closed-toe, nonabsorbent shoes are a minimum.

5.1. Specific Safety Concerns or Requirements

Samples that contain high concentrations of carbonates or organic material or samples that are at an elevated pH can react violently when acids are added.

5.2. Primary Materials Used

The following is a list of the materials used in this method, which have a serious or significant hazard rating. **Note: This list does not include all materials used in the method. The table contains a summary of the primary hazards listed in the MSDS for each of the materials listed in the table.** A complete list of materials used in the method can be found in the reagents and materials section. Employees must review the information in the MSDS for each material before using it for the first time or when there are major changes to the MSDS.

Material (1)	Hazards	Exposure Limit (2)	Signs and symptoms of exposure
Nitric Acid	Corrosive Oxidizer Poison	2 ppm-TWA 4 ppm-STEL	Nitric acid is extremely hazardous; it is corrosive, reactive, an oxidizer, and a poison. Inhalation of vapors can cause breathing difficulties and lead to pneumonia and pulmonary edema, which may be fatal. Other symptoms may include coughing, choking, and irritation of the nose, throat, and respiratory tract. Can cause redness, pain, and severe skin burns. Concentrated solutions cause deep ulcers and stain skin a yellow or yellow-brown color. Vapors are irritating and may cause damage to the eyes. Contact may cause severe burns and permanent eye damage.
Potassium Dichromate	Oxidizer Corrosive Carcinogen	0.1 Mg/M3 TWA as CrO3	Extremely destructive to tissues of the mucous membranes and upper respiratory tract. May cause ulceration and perforation of the nasal septum. Symptoms of redness, pain, and severe burn can occur. Dusts and strong solutions may cause severe irritation. Contact can cause blurred vision, redness, pain and severe tissue burns. May cause corneal injury or blindness.
Sulfuric Acid	Corrosive Oxidizer Dehydrator Poison Carcinogen	1 Mg/M3-TWA	Inhalation produces damaging effects on the mucous membranes and upper respiratory tract. Symptoms may include irritation of the nose and throat, and labored breathing. Symptoms of redness, pain, and severe burn can occur. Contact can cause blurred vision, redness, pain and severe tissue burns. Can cause blindness.

6.0 Equipment and Supplies

6.1. Instrument

- Spectrophotometer for use at 540 nm, providing a light path of 1cm or longer
- pH meter, Orion benchtop or equivalent

6.2. Supplies

- Nalgene 115-ml disposable filter unit with 0.45um filter
- Membrane filters, 0.45um and 0.1 um
- Specimen cups (plastic)
- Eppendorf or Finnpipettes, varying volumes
- Class A volumetric flasks
- Class A graduated cylinder
- Vacuum pump
- Magnetic stirrer (Teflon coated)
- Transfer pipettes

7. Reagents and Standards

7.1. Reagents

7.1.1. Sulfuric acid (10% v/v): Measure 100 ml of concentrated reagent grade H_2SO_4 into a 1000 ml volumetric flask (1/2 filled w/D.I. water) and dilute to mark with deionized water. Stable for six months, store at room temperature.

7.1.2. Indicator solution (DPC): Place 5.0 g 1,5-Diphenylcarbazide (AR Grade) in an amber 1000 ml volumetric flask and dilute to mark with acetone. Prepare monthly or when solution becomes cloudy. Store at room temperature.

7.1.3. Acetone: reagent grade; for stability and storage information refer to manufacturer's instructions.

7.1.4. 1:1 HNO3: Add 500ml DI to a 1 Liter volumetric flask and then slowly add 500ml of concentrated reagent grade HNO3. Solution is stable for 6 months. Store at room temperature.

7.1.5. 1N NaOH: Place 4.0g reagent grade NaOH in a 100ml volumetric flask and dilute to the mark with deionized water. Stable for six months, store at room temperature.

7.2 Standards

7.2.1 Potassium Dichromate Primary Standard Solution (1000 mg/L Cr+6): Weigh 0.2829 g of Potassium Dichromate ACS grade, J.T. Baker, cat# 3093-01 (dried for 1 hour at 105ºC) and dissolve in deionized water in a 100 ml flask. Dilute to the mark. Stable for 6 months, store at room temperature.

7.2.2 Potassium Dichromate Secondary Standard Solution (1000 mg/L Cr+6): Weigh 0.2829 g of Potassium Dichromate ACS grade, J.T. Baker, cat# 3090-04 (dried for 1 hour at 105ºC) and dissolve in deionized water in a 100 ml flask. Dilute to the mark. **Note**: Prepare the secondary standard from a source or lot different from the primary standard solution. Stable for 6 months, store at room temperature.

7.2.3 Hexavalent Chromium Primary Spiking Solution (100 mg/L Cr+6): Prepare by adding 10 ml of 1000 mg/L Cr+6 primary standard (Sec 7.2.1) to a 100 ml volumetric flask and dilute to volume with deionized water. Stable for 6 months, store at room temperature.

7.2.4 Hexavalent Chromium Secondary Spiking Solution (100 mg/L Cr+6) Prepare by adding 10 ml of 1000 mg/L Cr+6 secondary standard (Sec 7.2.2) to a 100 ml volumetric flask and dilute to volume with deionized water. Stable for 6 months, store at room temperature.

7.2.5 Lead Chromate: AR Grade; for stability and storage information refer to manufacturer's instructions.

8. Sample Collection, Preservation, Shipment and Storage

Sample container, preservation techniques and holding times may vary and are dependent on sample matrix, method of choice, regulatory compliance, and/or specific contract or client requests. Listed below are the holding times and the references that include preservation requirements.

Matrix	Sample Container [1]	Min. Sample Size[2]	Preservation	Holding Time [3]	Reference
Soils	Glass, plastic	2.5 grams	Cool 4 ± 2°C	30 Days - *from sampling to extraction*	SW846 Method 3060A
				168 Hours – *from extraction to determinative analysis*	SW846 Method 7196A

[1] Containers should not contain stainless steel
[2] Additional volume may be required if the sample is chosen for QA or if a re-prep is required.
[3]The alkaline digestate must be analyzed within 168 hours after extraction from the soil.

9. Quality Control

9.1. **Sample QC** - The following quality control samples are prepared with each batch of 20 samples or less.

9.1.1. Method Blank: One method blank must be digested and analyzed per batch. A method blank consists of 50 ml digestion solution and is carried through the entire digestion and analytical procedure. The concentration of the method blank must be less than the reporting limit or the entire batch must be re-digested and re-analyzed.

9.1.2. Laboratory Control Sample: Two secondary source (LCS) shall be digested and analyzed per batch.

- **LCS soluble (LCSSRM)** is obtained from an independent source (Phenova). Prepare the LCS soluble solution by diluting the concentrated LCS solution as indicated in the manufacturer's instructions. If necessary, prepare a different dilution so that the concentration after analysis falls within the calibration curve. Add 5ml of the diluted LCS soluble solution into 50ml digestion solution. The results must be within vendor specified QC limits or the entire batch must be redigested and re-analyzed. Document the preparation of the LCS soluble solution in the Reagent module in TALS.

- **LCS insoluble (LCSI)** is prepared by adding 0.010g-0.020g of PbCrO4 into 50ml digestion solution. The results must be within 80-120% of the true value or the entire batch must be redigested and re-analyzed. (Typical amount of $PbCrO_4$ added to LCSI is 0.011g, this is equivalent to 708 mg Cr (VI)/Kg).

9.1.3. Predigestion Matrix Spikes: Both a soluble and insoluble pre-digestion matrix spikes must be analyzed per batch of ≤ 20 field samples.

9.1.3.1 Soluble matrix spike (MSS): spike the sample with 1.0 ml of the 100 ppm Cr (VI) primary spiking solution prepared in Sec 7.2.3 (equivalent to 40mg/kg Cr(VI)).

9.1.3.2 Insoluble matrix spike (MSI): to a separate sample aliquot add 0.010g- 0.020g of $PbCrO_4$. It is used to evaluate the dissolution during the digestion process. Typical amount of $PbCrO_4$ added to LCSI is 0.011g, this is equivalent to 708 mg Cr (VI)/Kg.

9.1.3.3 Both matrix spikes are then carried through the digestion process. The acceptance range for the matrix spike recoveries is 75-125%. If the matrix spike recoveries fall outside these recovery limits, the entire batch must be rehomogenized, redigested, and reanalyzed.

- Note: If redigesting/reanalyzing the batch, the Soluble matrix spike (MSS) is spiked at twice the sample concentration or 40mg/kg Cr(VI) whichever is greater.

9.1.3.4 If upon reanalysis, the matrix spike is not within the recovery limits of 75%-125%, but the LCS is within the criteria specified in Section 9.1.2, additional laboratory characterization of each sample in the batch for ORP (see TestAmerica Edison SOP ED-WET-066, Redox by SM2580) and pH (see TestAmerica Edison SOP ED-WET-061, pH Soil by SW846 9045C) may be required by the client to determine if the sample exhibits reducing conditions. Further characterization for total sulfides, Total Organic Carbon (TOC), Chemical Oxygen Demand (COD), or Biochemical Oxygen Demand (BOD) may also be required by the client.

9.1.4. Duplicate (DU): One duplicate laboratory sample must be analyzed per batch. The sample used for the predigestion spike should be used for this purpose. Duplicate samples must have a Relative Percent Difference (RPD) of ≤20%, if both the original and the duplicate are ≥ four times the laboratory reporting limit. A control limit of ± 2.0mg/kg (laboratory reporting limit) is used when either the original or the duplicate sample is < four times the laboratory reporting limit.

9.1.5. Post Digestion Spike (PDS): Following the analysis (colorimetric determination), a post-digestion spike must be analyzed per batch. The spike concentration must be equivalent to 40mg/kg or twice the sample concentration, whichever is greater. To spike the extract with 40mg/Kg Cr

(VI), add 0.50 ml of 100 ppm Cr (VI) primary standard to a 50.0 ml alkaline extract, pour out 45.0 ml of the spiked sample and add color indicator and adjust pH (see Sec 10.3.4 & 10.3.5); bring the final volume to 50ml with deionized water. The post digestion spike must be performed on a field sample, not on a field blank or preparation blank. If possible perform the post digestion spike analysis on the sample used for the matrix spike. Recovery limits for the post-digestion spike are 85-115% of the true value. If the post digestion spike fails to meet the recovery limits, a post spike must be re-analyzed using the alkaline extract. Adjust the pH of the sample Duplicate (alkaline extract) back up to 8.0-8.5 using 1N NaOH; then respike and reanalyze following Sec. 10.3.4-10.3.5.

9.2. Instrument QC

9.2.1. Initial Calibration Verification (ICV): The ICV (0.50 mg/L Cr (VI)) is analyzed immediately after an acceptable initial calibration. The ICV standard must be from a source separate from the calibration standards (i.e., different manufacturer or separate lot) and its recovery must be within ±10% of the expected value. The ICV is prepared and analyzed similar to the calibration standards; prepare by adding 0.5 ml of 100 ppm Cr (VI) Secondary Spiking Solution (Sec. 7.2.4) into a specimen cup containing 50 ml of digestion fluid. If the measured concentration exceeds the ±10% limit, a second analysis should be performed. If the result still exceeded the ±10% limit, the analysis should be terminated until the source of the problem is identified and corrected.

9.2.2. Continuing Calibration Verification (CCV): A CCV is a mid-point calibration Cr (VI) standard (0.5ppm) from a different source than the calibration standards. Add 0.5 ml of 100 ppm Cr (VI) Secondary Spiking Solution (Sec. 7.2.4) into a specimen cup containing 50 ml of digestion fluid. The CCV is analyzed after every 10 samples (20 readings including backgrounds), and after reading the last sample. Acceptance criteria for the CCV are 90-110% of the true value. If the result is not within the acceptance limits, all samples following the last acceptable CCV must be re-analyzed.

9.2.3. Initial Calibration Blank and Calibration Check Blank (ICB,CCB): An initial calibration blank (ICB) must be analyzed immediately following the calibration curve and a continuing calibration blank (CCB) is analyzed after each CCV. The sample consists of 50 ml digestion solution and is carried through the entire analytical procedure. The concentration of the CCB must be below the reporting limit if not, all samples following the last acceptable CCB must be re-analyzed.

10. Procedure

10.1. Sample Preparation:

10.1.1. Just prior to analysis, SLOWLY adjust the pH of the sample extract to a pH of 7.0-8.0 with constant stirring using 1:1 nitric acid. Record pH and time. If a flocculent precipitate forms after the addition of 1:1 HNO3, filter the sample through a 0.45 um membrane filter, a larger size filter may be used to pre-filter the sample. Monitor the pH with a calibrated pH meter. If the pH drops below 7.0, discard the sample extract and re-digest the sample.

10.1.1.1. Remove and rinse the stir bar. Quantitatively transfer the sample extract to a 100 ml volumetric flask and dilute to the mark with deionized water.

10.1.1.2. If necessary, the solution can be diluted to eliminate the effects of color on the analytical determination. Use the smallest dilution necessary and record results.

10.2. Calibration

10.2.1. Prepare fresh calibration standards everyday or before each analysis.

10.2.2. Prepare a set of 6 calibration standards. Prepare 0.0, 0.05, 0.1, 0.5, 0.75, and 1.25 mg/l $K_2Cr_2O_7$ standards by aliquoting 0, 0.05, 0.1, 0.5, 0.75 and 1.25 mls of 100ppm Cr (VI) Primary Spiking Solution (Sec 7.2.3) respectively into the specimen cups containing 50mls of digestion fluid. At the same time prepare the mid-point Initial Calibration Verification and Initial Calibration Blank, see Sec 9.2.

10.2.3. Adjust pH to 7.5±0.5 with 1:1 HNO_3 and dilute to 100 ml with DI water. Record pH reading and analysis time in the logbook.

10.2.4. Develop the color for the standards as explained in Section 10.3 of this SOP.

10.2.5. Following the preparation of the calibration standards, “zero” the spectrophotometer with the 0 ppm standard. Analyze the standards.

10.2.6. The correlation coefficient must be 0.995 or better.

10.2.7. Immediately following calibration of the spectrophotometer and before reading samples, verify the calibration of the spectrophotometer by analyzing an ICV and ICB.

10.3. Sample Analysis

10.3.1. Warm up spectrophotometer and set to 540nm.

10.3.2. Prepare two rows of plastic cups and label them accordingly (first row with sample no. and the 2nd row as background).

10.3.3. Pour out 45.0 ml of the sample extracts, which have already been pH adjusted to between 7.0 and 8.0, into the appropriately labeled cups. Do the same for the background cups using 10 ml of the sample extract.

10.3.4. Add 1.0 ml indicator solution and swirl. (Do not add reagent to background sample).

10.3.5. Slowly add 10% H_2SO_4 (Sec 7.1.1) dropwise while swirling and monitoring pH until pH is 2 ±0.5. Record reading in the appropriate logbook.

10.3.6. Transfer sample to 50 ml volumetric flask and bring to volume with deionized water. Empty contents into labeled specimen cup to mix.

10.3.7. Let stand 5-10 minutes for full color development.

10.3.8. Read calibration standards, QC samples, and samples in a 1 cm cell on a spectrophotometer at 540 nm. (Note: Calibration standards and samples must be read within the same timeframe following full color development).

10.3.9. Continue analyzing samples and quality control samples by first reading the sample followed by its background sample.

10.3.10. Dilute samples that exceed calibration curve using deioinized water and analyze following Sec. 10.3.3-10.3.7. Add dilution factor to TALS under "dilution" column. Note: The dilution should be made from the sample extract that has not had the indicator added.

10.3.11. After every twenty readings (10 samples) and after reading the last sample in the batch, verify the spectrophotometer calibration by reading a calibration check standard (CCV) and calibration check blank (CCB).

11.0. Calculations / Data Reduction

11.1. Accuracy:

$$\text{ICV / CCV, LCS \% Recovery} = \frac{\text{observed concentration}}{\text{known concentration}} \times 100$$

$$\text{MS \% Recovery} = \frac{\text{(spiked sample) - (unspiked sample)}}{\text{spiked concentration}} \times 100$$

11.2. Precision (RPD):

$$\text{Matrix Duplicate (MD)} = \frac{|\text{orig. sample value - dup. sample value}|}{[(\text{orig. sample value + dup. sample value})/2]} \times 100$$

11.3. Concentration = Hexavalent Chromium in mg/Kg = $\frac{A \times B \times E * 100}{C \times D}$

Where:

A = Concentration from the calibration curve in mg/L
B = Final digested volume in Liters
C = Wet sample weight in kg
D = percent solids
E = Dilution (if necessary)

NOTE: All dry weight corrections are made in TALS at the time the final report is prepared.

11.4. Trivalent Chromium Calculation: When required, the calculation for trivalent chromium is completed using TALS Method '7196a_CR3.' Samples must be logged in under this method as well as the methods for Total chromium (metals) and Hexavalent chromium. Upon completion of the total and hexavalent chromium analyses the analyst may process the trivalent chromium results as follows:

- In order for TALS to perform an automated trivalent chromium calculation enter '1' (for 'yes') in trivalent chromium batch information page in response to the prompt 'automatically perform calculation.' As long as both methods are at 2nd level review status in TALS the program will automatically pull in the metals results for chromium and the hexavalent chromium results to perform the automated calculation. TALS will display an 'ok' message on the worksheet tab if the Cr6 result is less than Total Cr result.
- In cases where Total Cr results are not linked to TALS method 7196a_CR3, trivalent chromium is calculated manually. Enter '0' (for 'no') in batch information page 'Perform calculation.' Results will be entered manually.

11.5. Data reduction:

11.5.1. All data is recorded directly in TALS and recorded in the logbook at the time the analysis is performed.

11.5.2. Attach the prep and analytical logbook pages and calibration curve to the batch as a pdf file.

11.5.3. On the worksheet tab, enter the sample absorbance under 'Uncorrected Abs' and background absorbance under 'Color Blank Abs.' Also, enter the pH between 7-8 as 'initial pH',' pH between 1.5-2.5 as 'Final pH' and the background pH under 'Notes.'

11.5.4. Record special notes and observations in the "worksheet" tab and record reagent information in the prep batch information page (see "view batch information" page of ADII).

11.5.5. Complete the Data Review Checker (DRC) in TALS: Prior to data submission the analyst must execute the DRC (Data Review Checker) program in the applicable analytical batch (TALS Analyst Desktop II module).

11.5.5.1. Open the analytical batch and click on the Edit tab above to enter the Edit Mode.

11.5.5.2. Press F8 command key on the computer keyboard or right click anywhere on the batch worksheet then click on 'Run checklist.'

11.5.5.3. Acknowledge by filling in responses to all unacknowledged findings. Highlight the checklist with findings, then highlight the associated list of findings; right click and choose 'Acknowledge Item.'

11.5.5.4. Fill in appropriate comments in the response box, then hit 'OK.' 'The column labeled 'unacknowledged findings' should show '0' for all questions once completed; then batch is ready for 2nd level review.

11.5.6. Analyst must fill out the Wet Chem Data Review checklist (WI# EDS-WI-008) during the first level review. After the batch is second level reviewed, the checklist is filed in wetchem department and scanned in the network drive.

12.0. Method Performance

12.1. Method Detection Limit Study (MDL)

The method detection limit (MDL) is the lowest concentration that can be detected for a given analytical method and sample matrix with 99% confidence that the analyte is present. The MDL is determined according to the laboratory's MDL procedure in Section 19 (Test Methods and Method Validation) of TestAmerica Edison's Quality Assurance Manual (ED-QA-LQM). MDLs reflect a calculated (statistical) value determined under ideal laboratory conditions in a clean matrix, and may not be achievable in all environmental matrices. The laboratory maintains MDL studies for analyses performed; these are verified at least annually unless method requirements require a greater frequency.

12.2. Demonstration of Capabilities

For DOC procedure refer to Section 19 in the most current revision of TestAmerica Edison's Quality Assurance Manual (ED-QA-LQM).

12.3. Training Requirements

Refer to TestAmerica SOP No. ED-GEN-022, Training, for the laboratory's training program.

13.0. Pollution Control

It is TestAmerica's policy to evaluate each method and look for opportunities to minimize waste generated (i.e., examine recycling options, ordering chemicals based on quantity needed, preparation of reagents based on anticipated usage and reagent stability). Employees must abide by the policies in Section 13 of the Corporate Environmental Health and Safety Manual (CW-E-M-001) for "Waste Management and Pollution Prevention."

14.0. Waste Management

14.1. Waste management practices are conducted consistent with all applicable rules and regulations. Excess reagents, samples and method process wastes are

disposed of in an accepted manner. Waste description rules and land disposal restrictions are followed. Waste disposal procedures are incorporated by reference to TestAmerica SOP No. ED-SPM-008 (Laboratory Waste Disposal Procedures).

14.2. The following waste streams are produced when this method is carried out:

- Expired Standards – The vials are collected in a 1 gallon polyethylene bucket. These vials are then transferred to an open top 55 gallon steel or polyethylene waste drum. These drums are transported to a waste facility for proper disposal.
- Sample waste-After analysis, the samples and standards are collected in a polyethylene container labeled 'Cr6+ waste.' Once the container is full, it is brought to the waste room for disposal.

15.0. References / Cross-References

15.1. Test Methods for Evaluating Solid Waste, Physical/Chemical Methods, 3rd ed., U.S. Environmental Protection Agency, Office of Solid Waste and Emergency Response. U.S. Government Printing Office: Washington, DC, 1995; SW-846, Method 7196A.

15.2. Test America Edison SOP ED-WET-010, The Alkaline Digestion of Soil Samples for the Analysis of Hexavalent Chromium, most current revision.

15.3. TestAmerica Edison Document No. ED-QA-LQM, *Laboratory Quality Manual*, most current revision.

15.4. TestAmerica Edison SOP ED-GEN-022, *Training*, most current revision.

15.5. TestAmerica Edison SOP No. ED-SPM-008, *Laboratory Waste Disposal Procedures*, current revision.

15.6. TestAmerica Edison SOP ED-WET-066, Redox, Analysis of Oxidation-Reduction Potential, most current revision.

15.7. TestAmerica Edison SOP ED-WET-061, Analysis of pH for soil and organic samples electrochemically, Method 9045C, most current revision.

15.8. TestAmerica Corporate Environmental Health and Safety Manual, Document No. CW-E-M-001, most current revision.

16. Method Modifications:

N/A

17. Attachments

N/A

18. Revision History

- **Revision 12, dated 17 June 2020**
  - Updated SOP header with Eurofins emblem.
  - Sec. 9.1.2: Changed LCS source vendor from ERA to Phenova.
  - Sec. 11.5.5: Revised data review procedure to include TALS DRC procedures.
  - Sec 15.8: Deleted Wetchem Data Review Checklist Work Instruction # EDS-WI-008, not applicable.

- **Revision 11, dated 05 March 2018**
  - Sec 11.4: Added procedure for Trivalent chromium calculation using TALS method 7196a_CR3; subsequent section adjusted accordingly.

- **Revision 10, dated 23 February 2016**
  - Sec 9.1.2 and 9.1.3.2: Revised the allowable amount of PbCrO4 to add to the QC sample LCSI to reflect the range referenced in the Method. Also included the typical amount of $PbCrO_4$ that is added to the QC sample (LCSI and MSI).
  - Sec 9.1.3.3: Added spiking instructions for MSS when redigesting a batch; MSS should be spiked at 2x sample concentration or 40mg/kg, whichever is greater.

- **Revision 9, dated 25 September 2013**
  - Sec 1 & 12: Updated LQM section references to reflect the most current LQM revision.
  - Sec. 9.1.2: Changed nomenclature to LCSSRM and revised the acceptance limits from 85-115% to vendor specified QC limits.
  - Sec. 9.1.3.3: Deleted text: '*Note: If the spiked sample concentration is greater than 4X the predigestion spike concentration, no redigestion/reanalysis is required unless the samples are from a NJ site in which case the project manager should be contacted for further guidance.'*

    Per method 3060a, all batches must be rehomogenized, redigested and reanalyzed if MSS or MSI fail regardless of location.
  - Sec. 10.3.5: Changed pH range from 1.6-2.2 to pH 2.0 +/-0.5 as per the method.
  - Sec. 11.4.3: Changed pH range from 1.6-2.2 to pH 1.5-2.5 as per the method.

- **Revision 8, dated 06 September 2011**
  - Sec 3: Revised the LQM reference for the list of definitions.
  - Sec 9.1.3.4: Clarified lab procedures for failing matrix spikes.
  - Sec. 9.2.2: Added procedure if the CCV is not within acceptance limits.
  - Sec. 9.2.3: Added procedure if CCB is not below reporting limit.
  - Sec. 11.4: Revised data reduction section to reflect actual laboratory practices.
  - Sec 15.9: Reference added.

- **Revision 7, dated 25 August 2009**

  - Revised SOP format in accordance with TestAmerica Corporate Quality SOP No. CW-QS-002 (Rev 0) Writing a Standard Operating Procedure (SOP).
  - Sec 1.1: added laboratory's reporting limit; change the range of analysis to 0.05-1.25mg/L to reflect actual laboratory practices.
  - Sec 9.1.5: Added preparation instruction for Post digestion spike.
  - Sec. 9.2: added ICV in the Instrument QC.
  - Sec 10.1: Added procedure for adjusting pH of sample extract; procedure formerly described in the Digestion SOP (ED-WET-010).
  - Sec 15: Added applicable references.

- **Revision 6a, dated 21 April 2009**

  - Sec.4.3: Added membrane filters, 0.45um and 0.1um.
  - Sec 4.4: Delete Erlenmeyer flasks and beakers
  - Sec 4.8:Add: Eppendorf or Finnipettes; varying volume
  - Sec 5.1, 5.2 & 5.4: Add stability and storage information.
  - Sec 5.5: Added reagent used for the pH adjusted PS: 1N NaOH: Place 4.0g NaOH in a 100 volumetric flask and dilute to the mark with deionized water. Solution is stable for 6 months. Store at room temperature.
  - Sec. 6.1: Potassium Dichromate Primary Standard Solution (1000mg/L Cr+6): Weight 0.2829g of dried (105ºC) potassium dichromate and dissolve in deionized water in a 100ml flask. Dilute to the mark. Store at room temperature. Solution is stable for 6 months.
  - Sec 6.1: Deleted pre-made standard purchased from Inorganic Ventures. Cr6+ Primary Standard will be prepared in the lab.
  - Sec 6.2: Specified source of the 1000 ppm Cr(VI).
  - Sec 6.3 and 6.6: Deleted the 10mg/L Cr (VI) working standard for soil analysis.
  - Sec 6.5: Add storage information.
  - Sec 6.4: Add text: Source must be different from the primary standard.
  - Sec 9.2.2: Replace 100 ppm stock standard with 100 ppm Cr6+ Primary spiking solution.
  - Sec. 9.2.3: Add text: Record pH reading in the logbook.
  - Sec. 9.3.8: Add text: Make the dilution from the sample that has not had the indicator added.
  - Sec 9.3.6: Add text: Before reading samples, verify the calibration of the spectrophotometer by analyzing a CCV and CCB.
  - Sec. 10.7: Added procedure for reanalyzing Post Spike sample to conform to method requirements: *If the post digestion spike fails to meet the recovery limits, a post spike must be reanalyzed using the alkaline extract. Adjust the pH of the sample Duplicate (alkaline extract) back up to pH 8.0-8.5 using 1N NaOH; then*

respike and reanalyze.

- **Revision 6, dated March 2007**

  - Section 7.2: Section deleted (start color development for digested samples within one hour after adjusting pH between 7.5±0.5); section not applicable.
  - Section 9.3.7: The text in italics was inserted to clarify the time period for measuring sample absorbance.

    "Note: Calibration standards and samples must be read within the same timeframe following full color development."
  - Section 10.2: Deleted the requirement that 'CCS is taken through the entire digestion process' to reflect actual laboratory practices.
  - Section 10.4. Revised to specify the types of LCS used including their source and acceptance limits.

- **Revision 5, dated 30 October 2006**

  - Section 9.2.2: Revised the high point of the calibration curve from 1.0 ppm to 1.25 ppm. Standards preparation instructions revised accordingly.
  - Section 9.2.2: Revised to include instructions for preparation of a second source mid-point Calibration Check Standard.
  - Section 10: Inserted a new Section 10.1 explaining the QA batching system and inserted a new section 10.4 detailing the LCS requirements. All existing sections renumbered accordingly.
  - Section 10.6 (previously Section 10.5): The italicized text was added to the following sentence:

    "NOTE: If the spiked sample concentration is greater than 4x the predigestion spike concentration, no redigestion/reanalysis is required *unless the samples are from a NJ site in which case the lab project manager should be contacted for further guidance."*
  - Section 10.6 (previously Section 10.5): Added STL Edison SOP and method references for ORP and pH.
  - Section 10.7 (previously Section 10.6): Added the following text: "If the post digestion spike fails to meet the recovery limits, a new aliquot of the sample must be re-spiked and re-analyzed."
  - Section 10.8 (previously Section 10.7): Revised first sentence to read "One duplicate laboratory sample per batch *must* be analyzed." ('Must' previously read 'should').

Title: The Analysis of Waters for Hexavalent Chromium by EPA SW846 Method 7196A

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Approvals (Signature/Date):

Jasmine Parillo 09/30/21	Dan Helfrich 10/01/21
Jasmine Parillo Date	Dan Helfrich Date
Department Manager	Health & Safety Manager /Coordinator
Carl Armbruster 09/30/21	Mark Acierno 09/30/21
Carl Armbruster Date	Mark Acierno Date
Quality Assurance Manager	Laboratory Director

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Facility Distribution No. Electronic **Distributed To:** Facility Intranet

1.0 Scope and Application

1.1. SW846 Method 7196A may be used to determine the hexavalent chromium content of groundwater samples or digestates of solid samples. This SOP is applicable for the determination of hexavalent chromium in groundwater samples. Groundwater samples associated with regulated drinking water samples (i.e. potable water wells) are not applicable to this Method.

1.2. The range of analysis by this method is 10-200 ug/l hexavalent chromium.

1.3. On occasion clients may request modifications to this SOP. These modifications are handled following the procedures outlined in Sections 7 (Review of Work Request) and 19 (Test Methods and Method Validation) of TestAmerica Edison's Quality Assurance Manual (ED-QA-LQM).

2.0 Summary of Method

Hexavalent chromium is determined spectrophotometrically at 540nm by reaction with diphenylcarbazide in acid solution. For each batch of samples a method blank, a laboratory control sample, matrix duplicate, and a post verification spike are analyzed and compared to method limits and laboratory control limits.

3.0 Definitions

For a complete list of definitions refer to Appendix 2 in the most current revision of TestAmerica Edison's Quality Assurance Manual (ED-QA-LQM).

4.0 Interferences

4.1. Molybdenum and Mercury react to form color with diphenylcarbazide, however the intensity of color is much lower than those for chromium at the specified pH. Concentrations up to 200 mg/L can be accepted.

4.2. Vanadium interferes strongly, but can be tolerated up to 10 times the concentration of hexavalent chromium present.

4.3. Iron may cause a yellow color but should not affect the colorimetric measurement at 540 nm.

4.4. Reducing matter may reduce hexavalent Cr to trivalent Cr in varying amounts. No preventative measure is available at this time; however, interference is checked by a post verification spike samples.

4.4.1. The reducing/oxidizing tendency of each matrix may be determined by characterization of samples for additional analytical parameters, such as pH, ferrous iron, sulfides, and Oxidation Reduction Potential. Other indirect indicators of reducing/oxidizing tendency include Total Organic Carbon (TOC), Chemical Oxygen Demand (COD), and Biological Oxygen Demand (BOD).

4.5. For waste materials containing soluble trivalent chromium concentrations greater than four times the laboratory hexavalent chromium reporting limit, the hexavalent chromium results obtained using this method may be biased high due to method-induced oxidation.

5.0. Safety

Employees must abide by the policies and procedures in the Corporate Environmental Health and Safety Manual (CW-E-M-001) and this document. This procedure may involve hazardous material, operations and equipment. This SOP does not purport to address all of the safety problems associated with its use. It is the responsibility of the user of the method to follow appropriate safety, waste disposal and health practices under the assumption that all samples and reagents are potentially hazardous. Safety glasses, gloves, lab coats and closed-toe, nonabsorbent shoes are a minimum.

5.1. Specific Safety Concerns or Requirements

Samples that contain high concentrations of carbonates or organic material or samples that are at elevated pH can react violently when acids are added.

5.2. Primary Materials Used

The following is a list of the materials used in this method, which have a serious or significant hazard rating. **Note: This list does not include all materials used in the method. The table contains a summary of the primary hazards listed in the MSDS for each of the materials listed in the table.** A complete list of materials used in the method can be found in the reagents and materials section. Employees must review the information in the MSDS for each material before using it for the first time or when there are major changes to the MSDS.

Material (1)	Hazards	Exposure Limit (2)	Signs and symptoms of exposure

Nitric Acid	Corrosive Oxidizer Poison	2 ppm-TWA 4 ppm-STEL	Nitric acid is extremely hazardous; it is corrosive, reactive, an oxidizer, and a poison. Inhalation of vapors can cause breathing difficulties and lead to pneumonia and pulmonary edema, which may be fatal. Other symptoms may include coughing, choking, and irritation of the nose, throat, and respiratory tract. Can cause redness, pain, and severe skin burns. Concentrated solutions cause deep ulcers and stain skin a yellow or yellow-brown color. Vapors are irritating and may cause damage to the eyes. Contact may cause severe burns and permanent eye damage.
Potassium Dichromate	Oxidizer Corrosive Carcinogen	0.1 Mg/M3 TWA as CrO3	Extremely destructive to tissues of the mucous membranes and upper respiratory tract. May cause ulceration and perforation of the nasal septum. Symptoms of redness, pain, and severe burn can occur. Dusts and strong solutions may cause severe irritation. Contact can cause blurred vision, redness, pain and severe tissue burns. May cause corneal injury or blindness.
Sulfuric Acid	Corrosive Oxidizer Dehydrator Poison Carcinogen	1 Mg/M3-TWA	Inhalation produces damaging effects on the mucous membranes and upper respiratory tract. Symptoms may include irritation of the nose and throat, and labored breathing. Symptoms of redness, pain, and severe burn can occur. Contact can cause blurred vision, redness, pain and severe tissue burns. Can cause blindness.

6.0. Equipment and Supplies

6.1. Instrumentation

- pH meter, Orion benchtop or equivalent
- Spectrophotometer for use at 540 nm with a light path of 1 cm (Hach DR2800 or equivalent).

6.2. Supplies

- Membrane filters, 0.45 um and 0.1 um (NALGENE disposable)
- Glasswares: volumetric pipets (class A) and volumetric Flasks (class A)
- Graduated Cylinder (class A)
- Transfer pipettes
- Calibrated pipettes: Eppendorf or equivalent
- Vacuum pump
- Specimen Cups (plastic)
- Magnetic stirrer (teflon-coated)

7.0. Reagents and Standards

7.1. Reagents

7.1.1. Sulfuric acid (10% v/v) - Measure 100 ml of concentrated reagent grade H_2SO_4 into a 1000 ml volumetric flask (1/2 filled w/D.I. water) and dilute to mark with deionized water. Reagent is stable for 6 months. Store at room temperature.

7.1.2. Indicator solution - Place 5.0 g 1, 5-Diphenylcarbazide (AR Grade) in an amber 1000ml volumetric flask and dilute to mark with acetone. Prepare monthly or when solution becomes cloudy. Store at room temperature.

7.1.3. Acetone (reagent grade) - Store at room temperature. For stability information, refer to manufacturer's instructions or 2 years from date opened if no instructions are available.

7.2. Standards

7.2.1. Primary Potassium Dichromate ($K_2Cr_2O_7$) Stock Standard (1000 mg/L Cr+6) - Weigh 0.2829 g of dried (105°C) analytical reagent grade (or better) potassium dichromate ($K_2Cr_2O_7$) crystals (J.T.Baker catalog nos. 3090, 3093 or equivalent). Dissolve in deionized water in a 100ml flask. Dilute to the mark. Standard solution is stable for 6 months. Store at room temperature.

7.2.2. Hexavalent Chromium Primary Spiking Solution (100 mg/L Cr+6) - Prepare by adding 10ml of the 1000 mg/L Primary Potassium Dichromate Standard (see Section 7.2.1) to a 100ml volumetric flask and dilute to volume with deionized water. Standard solution is stable for 6 months. Store at room temperature.

7.2.3. Hexavalent Chromium Primary Spiking Solution (10 mg/L Cr+6) - Prepare by adding 1.0ml of 1000 mg/L Cr+6 Standard (see Section 7.2.1) to a 100ml volumetric flask and diluting to volume with deionized water. Standard solution is stable for 6 months. Store at room temperature.

7.2.4. Secondary Potassium Dichromate ($K_2Cr_2O_7$) Stock Standard (1000 mg/L Cr+6) - Weigh 0.2829 g of dried (105°C) analytical reagent grade (or better) potassium dichromate ($K_2Cr_2O_7$) crystals (J.T.Baker catalog nos. 3090, 3093 or equivalent). *The source of this standard must be from a different lot or manufacturer than the Primary Potassium Dichromate Stock Standard detailed in Section 7.2*.1. Dissolve in deionized water in a 100ml flask. Dilute to the mark. Standard solution is stable for 6 months. Store at room temperature.

7.2.5. Hexavalent Chromium Secondary Spiking Solution (100 mg/L Cr+6) - Prepare by adding 10ml of 1000 mg/L Cr+6 Secondary Standard (see Section 7.2.4) to a 100ml volumetric flask and diluting to volume with deionized water. Standard solution is stable for 6 months. Store at room temperature.

7.2.6. Hexavalent Chromium Secondary Spiking Solution (10 mg/L Cr+6) - Prepare by adding 1.0ml of 1000 mg/L Cr+6 Secondary Standard (see Section 7.2.4) to a 100ml volumetric flask and diluting to volume with deionized water. Standard solution is stable for 6 months. Store at room temperature.

8.0. Sample Collection, Preservation, Shipment and Storage

Sample container, preservation techniques and holding times may vary and are dependent on sample matrix, method of choice, regulatory compliance, and/or specific contract or client requests. Listed below are the holding times and the references that include preservation requirements.

Matrix	Sample Container	Min. Sample Size	Preservation	Holding Time [1]	Reference
Waters	HDPE[2]	250 mL	Cool 4 ±2°C	24 Hours	SW 7196A

[1] Samples must be analyzed within 24 hours from time of collection. Store samples at 4°C before analysis.
[2] High-density polypropylene container

9.0. Quality Control

9.1. **Sample QC** - - The following MB, LCS, and PVS are analyzed with each batch of 20 samples or each time samples are setup, whichever is more frequent. The DU is analyzed at a frequency of 1 per 10 samples or each time samples are set up, whichever is more frequent.

9.1.1. Method Blank (MB): Use deionized water for the method blank. Analyze using all reagents in the same volumes as used in the analysis of samples. The blank value should fall below the reporting limit if not, all samples associated with the method blank must be re-analyzed. Do not subtract the blank value from sample values.

9.1.2. Post Verification Spike (PS): The PVS concentration must be two times the concentration of Cr(VI) found in the sample or 30.0 ug/L (whichever is greater). If necessary, dilute the PS to fall within the range of the calibration curve. If the PS recovery is not within 85-115%, dilute the sample and re-analyze.

Note: The post spike is identified as “MS” in TALS QC.

9.1.3. Matrix Duplicate (DU): A duplicate is prepared and analyzed in exactly the same manner as the original sample. The relative percent difference (RPD) between the sample and duplicate should be less than or equal to 20%.

9.1.4. Laboratory Control Sample (LCS): The hexavalent chromium LCS is a certified QC standard purchased from Phenova (Catalog WP

Hexavalent Chromium). The LCS is used to monitor the accuracy of the entire analytical process. The LCS recovery must be within vendor specified QC limits or all samples associated with the LCS must be reanalyzed.

9.2. Instrument QC

9.2.1. Initial Calibration Verification (ICV): The ICV (75.0 ug/L Cr (VI)) is analyzed immediately after an acceptable initial calibration. The ICV standard must be from a source separate from the calibration standards (i.e., different manufacturer or separate lot) and its recovery must be within ±10% of the expected value. The ICV is prepared by adding 0.375ml of a 10 mg/L Cr6+ Hexavalent Chromium Secondary Spiking Solution (Section 7.2.6) into a 50 ml volumetric flask and diluting to 50ml with deionized water. If the measured concentration exceeds the ±10% limit, a second analysis should be performed. If the result still exceeded the ±10% limit, the analysis should be terminated until the source of the problem is identified and corrected.

9.2.2. Calibration Check Verification (CCV): A midpoint Calibration Check Verification spiked at 75.0 ug/L is prepared in the same way as the ICV (See Section 9.2.1). The CCV is analyzed after every 10 samples (20 readings including backgrounds) and after reading the last sample. The result must be within ± 10% of the true value. If not, all samples following the last acceptable CCV must be re-analyzed.

9.2.3. Initial and Continuing Calibration Blank (ICB/CCB): Initial Continuing Calibration Blank (ICB) must be analyzed after the ICV. Continuing Calibration Blank (CCB) must be analyzed after every 10 samples or after each Calibration Check Verification (CCV). Use deionized water for the ICB/CCB. The value of the ICB/CCB should be less than the RL. If not, all samples following the last acceptable ICB/CCB must be re-analyzed.

9.2.4. Minimum Reporting Level (MRL), 10ppb: The MRL must be analyzed daily before samples are analyzed. Prepare the MRL by adding 0.05ml of a 10 mg/L Hexavalent Chromium Primary Spiking Solution (Section 7.2.3) into a 50 ml volumetric flask and diluting to 50ml with deionized water. The results must fall within 50-150% of the true value.

10.0 Procedure

10.1. Calibration

10.1.1. Prepare fresh calibration standards everyday before analysis.

10.1.2. Initial Calibration Standards: (6 points). Place about 25 mL of deionized (DI) water in six 50 ml flasks. Prepare the six points as follows:

Cr(6+) Standard conc. (ug/L)	Volume (ml) of 10 mg/L Cr+6 (Section 7.2.3) to add	Final volume (ml) using Deionized water

0.0	0.0	50
10.0	0.05	50
50.0	0.25	50
75.0	0.375	50
100	0.50	50
200	1.0	50

10.1.3. Analyze the calibration standards in the manner detailed in Sections 10.2.1 thru 10.2.7. Zero the spectrometer with the 0 ug/L standard.

10.1.4. Perform linear regression on data resulting from analysis of the initial calibration. The correlation coefficient must be 0.995.

10.1.5. Analyze the Initial Calibration Verification (ICV) standard in the same manner as the initial calibration standards detailed above. If the ICV meets the criteria listed in Section 9.2.1, the analyst may continue with sample analysis (Section 10.2).

10.2. Sample Analysis

10.2.1. Warm up spectrophotometer and set to 540 nm.

10.2.2. Place 45.0 mL of sample into a 100 ml specimen cup. Also, place 45 mL of sample in a separate cup for background.

10.2.3. Add 1.0 ml indicator solution (see Section 7.1.2) and swirl. (Do not add indicator to background sample).

10.2.4. Slowly add 10% H_2SO_4 (See Section 7.1.1) dropwise to sample and background while measuring pH until pH is 1.5-2.5. Record reading.

10.2.5. Place sample in a 50 ml volumetric flask and bring to volume with deionized water. Pour back into original cup and swirl.

10.2.6. Let stand 5-10 minutes for full color development.

10.2.7. Read calibration standards, QC samples, and samples on spectrophotometer at 540 nm. Analyze the calibration check standard and calibration check blank immediately following the calibration standards.

10.2.8. Dilute samples that exceed calibration curve using deionized water. Recolorize and read again on spectrophotometer noting dilution factor.

10.2.9. Continue analyzing samples and quality control samples by first reading the sample followed by its background sample.

10.2.10. After every 10 samples and after reading the last sample in the batch, verify the calibration by reading Calibration Check Verification (CCV) and Continuing Calibration Blank (CCB).

11. Calculations / Data Reduction

11.1. Accuracy:

$$\text{ICV / CCV, LCS \% Recovery} = \frac{\text{observed concentration}}{\text{known concentration}} \times 100$$

11.2. Precision (RPD):

$$\text{Matrix Duplicate (MD)} = \frac{|\text{orig. sample value - dup. sample value}|}{[(\text{orig. sample value + dup. sample value})/2]} \times 100$$

11.3. Calculate the hexavalent chromium result for each aqueous sample as follows:

Hexavalent Chromium in ug/L = A x E

A = Concentration from the calibration curve in ug/L
E = Dilution (if necessary)

11.4. Trivalent Chromium Calculation: When required, the calculation for trivalent chromium is completed using the TALS method '7196a_CR3' which must be logged for the samples being processed along with methods for total chromium (metals) and hexavalent chromium. Upon completion of the total and hexavalent chromium analyses the analyst may process the trivalent chromium results as follows:

- To have TALs perform an automated trivalent chromium calculation: on the trivalent chromium batch information page enter '1' (for 'yes') in response to the prompt to automatically perform the calculation. As long as both methods are at 2nd level review status in TALS the program will automatically pull in the metals results for chromium and the hexavalent chromium results to perform the automated calculation. TALS will display an 'ok' message on the worksheet tab if the Cr6 result is less than Total Cr result.

- If the analyst intends to manually enter trivalent chromium results an entry of '0' (for 'no') should be made the and the results entered manually.

11.5. Data Reduction

11.5.1. Record analytical data directly into TALS and in the 'Hexachrome Run Logbook.'

11.5.2. Record Reagent information on the "batch information" page.

11.5.3. Special comments or observations should be recorded on the "worksheet" tab. Record Sample absorbances and pH readings on the "worksheet" tab.

11.5.4. Attach the calibration curve plot and the logbook pages for the analysis to the analytical batch as pdf files.

11.5.5. Complete the Data Review Checker (DRC) in TALS: Prior to data submission the analyst must execute the DRC (Data Review Checker) program in the applicable analytical batch (TALS Analyst Desktop II module).

11.5.5.1 Open the analytical batch and click on the Edit tab above to enter the Edit Mode.

11.5.5.2 Press F8 command key on the computer keyboard or right click anywhere on the batch worksheet then click on 'Run checklist.'

11.5.5.3 Acknowledge by filling in responses to all unacknowledged findings. Highlight the checklist with findings, then highlight the associated list of findings; right click and choose 'Acknowledge Item.'

11.5.5.4 Fill in appropriate comments in the response box, then hit 'OK.' 'The column labeled 'unacknowledged findings' should show '0' for all questions once completed; then batch is ready for 2nd level review.

12. Method Performance

12.1. Method Detection Limit Study (MDL)

The method detection limit (MDL) is the lowest concentration that can be detected for a given analytical method and sample matrix with 99% confidence that the analyte is present. The MDL is determined according to the laboratory's MDL procedure in Section 19 (Test Methods and Method Validation) of TestAmerica Edison's Quality Assurance Manual (ED-QA-LQM). MDLs reflect a calculated (statistical) value determined under ideal laboratory conditions in a clean matrix, and may not be achievable in all environmental matrices. The laboratory maintains MDL studies for analyses performed; these are verified at least annually unless method requirements require a greater frequency.

12.2. Demonstration of Capabilities

For DOC procedure refer to Section 19 in the most current revision of TestAmerica Edison's Quality Assurance Manual (ED-QA-LQM).

12.3. Training Requirements

Refer to TestAmerica SOP No. ED-GEN-022, Training, for the laboratory's training program.

13.0 Pollution Control

It is TestAmerica's policy to evaluate each method and look for opportunities to minimize waste generated (i.e., examine recycling options, ordering chemicals based on quantity needed, preparation of reagents based on anticipated usage and reagent stability). Employees must abide by the policies in Section 13 of the Corporate Environmental Health and Safety Manual (CW-E-M-001) for "Waste Management and Pollution Prevention."

14.0. Waste Management

14.1. Waste management practices are conducted consistent with all applicable rules and regulations. Excess reagents, samples and method process wastes are disposed of in an accepted manner. Waste description rules and land disposal restrictions are followed. Waste disposal procedures are incorporated by reference to TestAmerica SOP No. ED-SPM-008 (Laboratory Waste Disposal Procedures).

14.2. The following waste streams are generated as a result of this analysis:

- Water Retain Samples -These materials are originally received aqueous samples. These solutions are then transferred into the first drum of the neutralization system and neutralized to a pH of 6 – 9 with sodium bicarbonate (Siedler Chemical SC-0219-25). This solution is discharged into the municipal sewer system.

15.0. References / Cross-References

15.1. Test Methods for Evaluating Solid Waste, Physical/Chemical Methods, 3rd ed., U.S. Environmental Protection Agency, Office of Solid Waste and Emergency Response. U.S. Government Printing Office: Washington, DC, 1995; SW-846, Method 7196A.

15.2. TestAmerica Edison Document No. ED-QA-LQM, *Laboratory Quality Manual*, most current revision.

15.3. TestAmerica Edison SOP ED-GEN-022, *Training*, most current revision.

15.4. TestAmerica Edison SOP No. ED-SPM-008, *Laboratory Waste Disposal Procedures*, current revision.

15.5. TestAmerica Corporate Environmental Health and Safety Manual, Document No. CW-E-M-001, most current revision.

16.0. Method Modifications:

N/A

17.0. Attachments

N/A

18.0. Revision History

- Revision 11, 10/01/2021
  - Sec. 9.2.4: Added criteria to analyze a Minimum Reporting Level (MRL) daily.
- Revision 10, 14 May 2020
  - Updated SOP header with Eurofins logo.
  - Sec 1.1: Clarified matrix applicability for this SOP.
  - Sec. 9.1: Revised the frequency of Matrix Duplicate from 1/20 samples to 1/10 samples.
- Revision 9, 12 February 2019
  - Sec. 9.1.4: Changed LCS source vendor from ERA to Phenova.
  - Sec. 11.5.5: Replaced Wetchemistry Data review checklist (EDS-WI-008) to TALS Data Review Checker (DRC); instructions added.
  - Deleted Sec 15.6, WI EDS-WI-008, not applicable; subsequent sections adjusted accordingly.
- Revision 8, 30 Nov 2016
  - Sec. 11.4: Added Cr3 calc procedure
- Revision 7, 26 November 2012
  - Section 9.1.2: Revised the minimum concentration for PS from 15 ug/L to 30 ug/L to comply with Method 7196A.
- Revision 6, 04 October 2012
  - Section 1&12: Updated LQM reference to reflect the most current LQM revision.
  - Sec 5: Updated to comply with TestAmerica Corporate Quality SOP No. CW-QS-002 (Rev 0) Writing a Standard Operating Procedure (SOP)
  - Sec. 9.1.1: Revised to include corrective action procedure when MB is greater than the RL.
  - Sec. 9.1.4: Changed limits from 85-115% to vendor specified QC limits.
  - Sec. 9.2.2 & 9.2.3: Revised to include corrective action procedure when CCV and CCB are outside the acceptance limits.
- Revision 5, 7 September 2010
  - Section 2.0: Removed text 'Dissolved;' this SOP is only applicable to aqueous samples for method SW7196A which are not filtered.
  - Sec 3: Revised to update LQM reference for the list of definitions.
  - Section 6.2: Added graduated cylinders (class A) and transfer pipettes to the list of supplies.
  - Removed Nitric acid on list of reagents, not applicable for the analysis of Cr6+ waters.

- Section 9.1.1: Revised method blank criteria from <MDL to <RL.
- Section 9.1.4: Replaced LCS vendor specified limits to 85-115%.
- Section 9.2.2: Removed text: “immediately after analyzing the calibration standards.” Renamed CCS to CCV.
- Section 9.2.3: Revised CCB criteria from <MDL to <RL.
- Section 10.1.4: Revised the correlation coefficient criteria from ≥0.997 to ≥0.995.
- Section 13 & 14: Revised to comply with the current Method SOP format (CW-QS-002, Writing a Standard Operating Procedure (SOP)).
- Section 11.4: Revised Data Reduction section in accordance with TALS.
- Section 15: Added applicable references.

- Revision 4, January 22, 2008

  - Updated to new format as per TestAmerica’s SOP format.
  - Added the shelf-life and storage requirements to reagents in section 7, Reagents and standards.
  - Added J.T.Baker (Catalog Nos. 3090, 3093 or equivalent) as source of primary and secondary standards. Deleted reference to Inorganic Ventures as a source of the primary standard.
  - Replaced the source standard used for the preparation of 10 mg/L Cr+6 (primary and secondary spiking solution) from 100 mg/L Cr+6 to 1000 mg/L Cr+6.
  - Added the control limits for Post Verification Spike (PVS).
  - Added ICV as Instrument QC.
  - Added the preparation procedure and control limits for the Calibration Check Standard (CCS).
  - Deleted Matrix spike (matrix spike low and matrix spike high) as sample QC requirement.
  - Section 10.3.3: Revised pH range from 1.6-2.2 to 1.5-2.5 to reflect method requirements.
  - Standardized all analytical units to ug/L and mg/L.
  - Added the following to the References section: TestAmerica Edison SOP ED-GEN-022 (Training) and TestAmerica Laboratory Quality Manual (ED-QA-LQM).

Title: Percent Moisture Determination

Once printed, this is considered an uncontrolled document

Approvals (Signature/Date):

5/8/19	5/8/19
Carl Armbruster Quality Assurance Manager	Date	Mark Acierno Laboratory Director	Date
5/8/19	5/8/19
Dan Helfrich EH&S Manager	Date	Kenneth Rivera Sample Control Manager	Date

1.0 Scope and Application

1.1 Analytes, Matrix(s), and Reporting Limits

This procedure is used to determine the moisture content of soil, solids and sludge samples. The percent solids content can also be determined as 100% - percent moisture result. The moisture content data is subsequently used to adjust and report analytical results on a dry weight basis. The reporting limit is approximately 1% for both percent moisture and percent solids.

1.2 This SOP is based on the procedures USEPA Contract Laboratory Program Statement of Work for Organics Analysis, Multi-Media, Multi-Concentration, SOM01.1 (see References in Section 15).

1.3 On occasion clients may request modifications to this SOP. These modifications are handled following the procedures outlined in Sections 7 (Review of Work Request) and 19 (Test Methods and Method Validation) of TestAmerica Edison's Quality Assurance Manual (ED-QA-LQM).

2.0 Summary of Method

2.1 Percent moisture/percent solid is determined by drying a sub-sample at 100-110°C. The loss of mass due to drying is considered to be moisture.

3.0 Definitions

For a complete list of definitions refer to Appendix 2 in the most current revision of TestAmerica Edison's Quality Assurance Manual (ED-QA-LQM).

4.0 Interferences

Non-representative particles (large rocks and sticks) should be removed to ensure the accuracy of results. Reference TestAmerica Edison SOP No.ED-GEN-007, *Subsampling*, current revision for the proper sub-sampling technique.

5.0 Safety

Employees must abide by the policies and procedures in the Corporate Environmental Health and Safety Manual (CW-E-M-001) and this document. This procedure may involve hazardous material, operations and equipment. This SOP does not purport to address all of the safety problems associated with its use. It is the responsibility of the user of the method to follow appropriate safety, waste disposal and health practices under the assumption that all samples and reagents are potentially hazardous. Safety glasses, gloves, lab coats and closed-toe, nonabsorbent shoes are a minimum.

5.1. Specific Safety Concerns or Requirements

Be very careful when putting in and getting out samples from the oven. It is very hot, wear heat resistant gloves and use tongs.

5.2. Primary Materials Used

There are no materials used in this method that have a significant or serious hazard rating.

6.0 Equipment and Supplies

6.1. Equipment and Instrumentation

- Drying Oven (100°-110°C) (Fisher Isotemp or equivalent)
- Top loading balance (Denver Instrument or equivalent)
- NIST traceable thermometers

6.2. Supplies

- Aluminum Weighing Dish (Fisher p/n 08732 or equivalent)
- 50 ml Beaker (used as backup to aluminum weighing dishes)
- Wooden Tongue Depressors (Fisher p/n 01346PK or equivalent)

7. Reagents and Standards

7.1. None

8. Sample Collection, Preservation, Shipment and Storage

8.1 The holding time for CLP analysis is 10 days from Validated Time of Sample Receipt.

8.2 Holding time for non-CLP samples is 6 months (this is a lab imposed holding time not a regulatory requirement).

8.3 Refrigerate samples at 4°C (+/-2°C) until time of analysis

9. Quality Control

9.1. **Sample QC** - The following quality control samples are prepared with each batch of samples.

Quality Controls	Frequency	Control Limit
Procedure Blank (PB) aka Method Blank	1 in 20 or fewer samples	< Rpt. Limit
Duplicate (DUP)[1]	1 in 20 or fewer samples	20% RPD

[1] The sample for DUP is randomly selected, unless specifically requested by the analyst.

9.2. Instrument QC

9.2.1. The balance calibration must be verified on each day of use as detailed in TestAmerica Edison SOP No. ED-GEN-010, *Calibration of Analytical Balances*, current revision.

9.2.2. The oven thermometers must be calibrated at the frequency detailed in TestAmerica Edison SOP No. ED-GEN-014, *Thermometer Calibration,* current revision.

10. Procedure

10.1. Confirm that the balance has been had the required daily calibration check performed and documented (see Section 9.2.1).

10.2. Confirm that the temperature of the drying oven(s) is between 100 - 110° C.

10.3. At the beginning of each day, document the temperature of the oven(s) on the appropriate temperature chart (WI# EDS-WI-117, Temperature chart- % moisture oven).

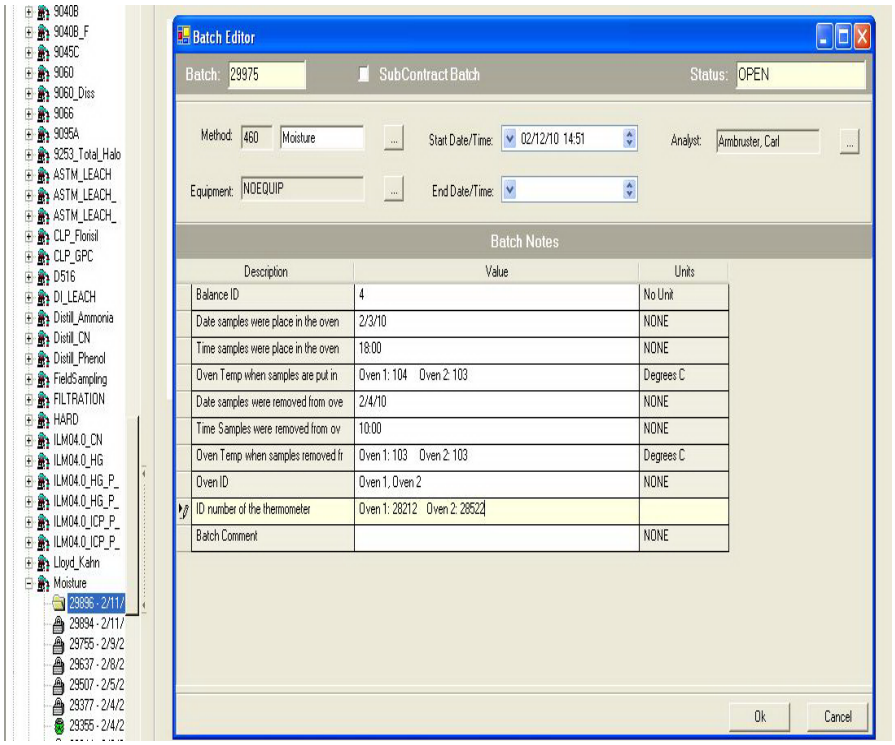
10.4. Document the oven temperature (corrected and uncorrected temperature) on the appropriate line of the batch notes section of the Batch Information page.

10.5. Retrieve and assemble all samples to be analyzed. Homogenize all samples (see Section 4.0).

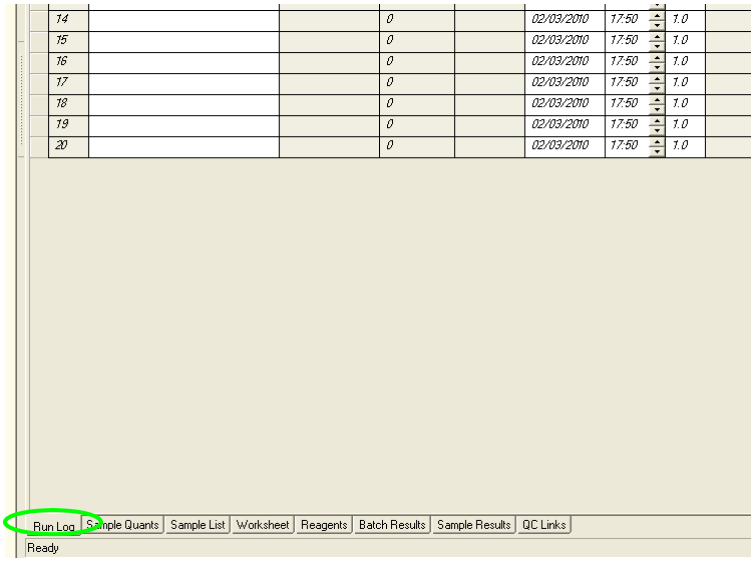
10.6. Open the TALS (LIMS) application and login with your Username and Password.

10.7. Open the Analytical Desktop application by clicking on ***Analyst*** followed by ***Analyst Desktop II.***

10.8. Open the current existing Percent Moisture batch by clicking ***Get Batch by Number*** and entering the batch number as prompted. Alternatively, create a new batch by clicking ***Create Batch from Scratch,*** selecting the applicable method code (“Moisture), and entering the requested information under Batch Notes (oven IDs, thermometer IDs, etc…)



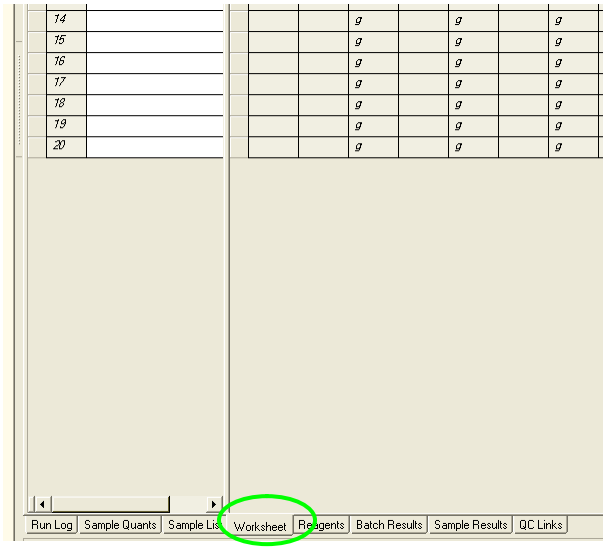
10.9. Begin adding samples ID numbers to the batch on the Run Log tab (the first sample in the batch is always the Procedure Blank which can be entered manually by typing PB in the first LIMS Sample ID field).



10.10. Scan each client sample label into the batch tabbing to the next line number after each label scan.

10.11. The sample scanned into Line 20 must also be scanned into Line 21. Type DU after the scanned sample ID in Line 21 (this is the required sample duplicate). Repeat as required at Lines 40/41, 60/61 and so on.

10.12. Once all samples have been scanned into the Run Log tab in TALS click on the Worksheet Tab.



10.13. Place a new, clean aluminum weighing dish on the balance. Each aluminum dish should be marked with the lab sample number or some other unique identifier (DISH ID). NOTE: aluminum weighing dishes are intended for single use only. Dishes must be discarded after one use.

10.14. Enter the Dish ID number in the TALS provided field.

10.15. Press the button with the printer icon on the Denver Instruments balance. This will automatically send the 'Dish Weight' (in grams) to the 'Dish Weight' field for the selected sample.

10.16. Using the tongue depressor add 5-6 grams of sample to the tared weighing dish (weigh to the nearest 0.01g).

10.17. Once again press the printer icon button on the Denver Instruments balance. This will automatically send the sample weight (in grams) to the "SampleMassWet' field for the selected sample.

10.18. Repeat 10.11 through 10.17 for all samples in the batch (NOTE: no sample is weighed out for the Procedure Blank. An empty weighing dish is carried through the entire procedure for the Procedure Blank).

10.19. When all samples have been processed as described above click ***Save*** in TALS.

10.20. Place all of the weighing dishes into the drying oven. Samples must dry overnight at minimum (approximately 12 hours). *Note: Due to scheduling of technicians and observed holidays by the laboratory, there are occasions when the drying time will be longer than 24 hours.*

10.21. Upon removal from the drying oven the weighing dishes are placed into a desiccator to cool for a minimum of 10 minutes.

10.22. Confirm that the balance has been had the required daily calibration check performed and documented (see Section 9.2.1).

10.23. Confirm that the temperature of the drying oven(s) is between 100 - 110° C. Record the corrected and uncorrected oven temperature in TALS batch information page.

10.24. Open the TALS (LIMS) application and login with your Username and Password.

10.25. Open the Analytical Desktop application by clicking on ***Analyst*** followed by ***Analyst Desktop II.***

10.26. Open the applicable Percent Moisture batch by clicking ***Get Batch by Number*** and entering the batch number as prompted.

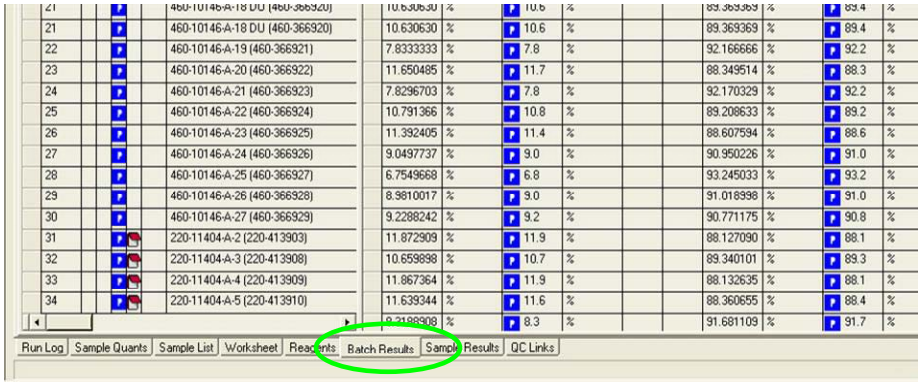
10.27. Click ***Edit.*** Click on the ***Worksheet tab*** along the bottom of the screen.

10.28. Click in the empty ***SampleMassDry Value*** field for the first sample to be weighed.

10.29. Place the sample weighing dish on the balance and press the printer icon button on the balance. This transfers the final weight to the SampleMassDry field.

10.30. Repeat the steps in Sections 10.27 through 10.28 for each sample. When all samples have been weighed in the manner click ***Save.***

10.31. Click on the ***Batch Results*** tab. Review the accuracy of the Percent Moisture and Percent Solids results against the data entered. The formula for calculating these parameters is found in Section 11.0.



11.0. Calculations / Data Reduction

11.1. Precision (RPD):

$$\text{Matrix Duplicate (MD)} = \frac{|\text{orig. sample value - dup. sample value}|}{[(\text{orig. sample value + dup. sample value})/2]} \times 100$$

11.2. Percent Solids

$$\%\ \text{Solids} = \frac{\text{Sample Dry Weight}}{\text{Sample Wet Weight}} \times 100$$

11.3. Percent Moisture:

$$\%\ \text{Moisture} = \frac{\text{grams wet sample – grams dry sample}}{\text{grams of wet sample}} \times 100$$

NOTE: All dry weight corrections are made in LIMS at the time the final report is prepared.

11.4. Data Reduction:

11.4.1. Document in TALS batch worksheet the following information:

- Date and time samples were placed in and out of the oven
- Temperature (corrected and uncorrected temperature) of the oven when samples were put in and taken out of the oven
- Balance ID, Oven ID and Thermometer ID

11.4.2. Analyst performs the first level review and completes the first level review section of the data review checklist. The manager or his/her designee performs the second level review and completes the second level review section of the data review checklist.

11.4.3. Monthly temperature charts are reviewed by the department manager and are bound and stored in the manager's office.

12.0. Method Performance

12.1. Method Detection Limit Study (MDL)

The method detection limit (MDL) is the lowest concentration that can be detected for a given analytical method and sample matrix with 99% confidence that the analyte is present. The MDL is determined according to the laboratory's MDL procedure in Section 19 (Test Methods and Method Validation) of TestAmerica Edison's Quality Assurance Manual (ED-QA-LQM). MDLs reflect a calculated (statistical) value determined under ideal laboratory conditions in a clean matrix, and may not be achievable in all environmental matrices. The laboratory maintains MDL studies for analyses performed; these are verified at least annually unless method requirements require a greater frequency. ***An MDL study is not required for this method.***

12.2. Demonstration of Capabilities

For DOC procedure refer to Section 19 in the most current revision of TestAmerica Edison's Quality Assurance Manual (ED-QA-LQM).

12.3. Training Requirements

Refer to TestAmerica SOP No. ED-GEN-022, Training, for the laboratory's training program.

13.0. Pollution Control

It is TestAmerica's policy to evaluate each method and look for opportunities to minimize waste generated (i.e., examine recycling options, ordering chemicals based on quantity needed, preparation of reagents based on anticipated usage and reagent stability). Employees must abide by the policies in Section 13 of the Corporate Environmental Health and Safety Manual (CW-E-M-001) for "Waste Management and Pollution Prevention."

14.0. Waste Management

14.1. The U.S. Environmental Protection Agency requires that laboratory waste management practices conducted be consistent with all applicable rules and regulations. All waste will be disposed of in accordance with Federal, State and Local regulations. Where reasonably feasible, technological changes have been implemented to minimize the potential for pollution of the environment. Employees will abide by this method and the policies in section 13 of the Corporate Safety Manual for "Waste Management and Pollution Prevention."

14.2. Waste management practices are conducted consistent with all applicable rules and regulations. Excess reagents, samples and method process wastes are disposed of in an accepted manner. Waste description rules and land disposal restrictions are followed. Waste disposal procedures are incorporated by reference to TestAmerica Edison SOP No. ED-SPM-008 (Laboratory Waste Disposal)

14.3. All used sample and aluminum weighing dishes are disposed of as excess environmental soil samples (*Waste Code SS*). This waste should be containerized or wrapped in aluminum foil and placed directly into cubic-yard "Jupiter" boxes. All other labeling and accumulating regulations apply. These containers area shipped directly to the TSDF under Veolia profile number 402535.

15.0. References / Cross-References

15.1. USEPA Contract Laboratory Program Statement of Work for Inorganics Analysis, Multi-Media, Multi-Concentration, ISM01.2 (Exhibit D) January 2010.

15.2. TestAmerica Edison Document No. ED-QA-LQM, *Laboratory Quality Manual*, most current revision.

15.3. TestAmerica Edison SOP No.ED-GEN-007, *Subsampling*, current revision

15.4. TestAmerica Edison SOP No. ED-GEN-010, *Calibration of Analytical Balances*, current revision.

15.5. TestAmerica Edison SOP No. ED-GEN-014, *Thermometer Calibration,* current revision.

15.6. TestAmerica Edison SOP ED-GEN-022, *Training*, most current revision.

15.7. TestAmerica Edison SOP No. ED-SPM-008, *Laboratory Waste Disposal*

15.8. TestAmerica Corporate Environmental Health and Safety Manual, Document No. CW-E-M-001, most current revision.

15.9. TestAmerica Edison Work Instruction EDS-WI-117, Temperature Chart - % moisture oven, most current revision.

16.0. Method Modifications:

Item	Method no.	Modification
1	Exhibit D, CLP ISM01.2	Sec 1.6 of CLP ISM01.2 requires drying time of 12-24 hours. Due to scheduling of technicians and observed holidays, there are occasions when the drying time will be longer than 24 hours.

17.0. Attachments

None

18.0. Revision History

- Revision 8, dated 08 May 2019
  - Section 11.4.2 changed to reflect the requirement to use the available checklists when performing first and second level data review.

- Revision 7, dated 05 January 2015
  - Cover page: Updated to current names.
  - Deleted Sec 10.16 (“Tare the balance with the empty aluminum dish by pressing the ‘Tare’ button.”) to reflect current procedure followed in TALS. Subsequent sections adjusted accordingly.
  - Sec 10.16 (formerly Sec 10.17): Revised to include sample weight range (5-6 grams).
  - Sec 10.20: Revised to include note: *Due to scheduling of technicians and observed holidays by the laboratory, there are occasions when the drying time will be longer than 24 hours.*
  - Deleted Sec 10.21, drying time procedure for CLP samples deleted, laboratory is not CLP certified. Text deleted: “***FOR CLP ISM01.2 SAMPLES****: Dry the samples overnight (12-24 hours), but no longer than 24 hours. If necessary, samples can be dried for less than 12 hours. In which case, it must be documented that a constant weight was attained and record the drying time.*

***Note**: Samples which are dried less than 12 hours should have a minimum of <u>two</u> drying cycle (weigh, dry, dessicate) with a minimum of one hour drying time in each cycle. Constant weight is obtained if the difference between the start weight and the final weight of the last cycle is <0.01g. Record the samples time in and time out of the oven to document the drying time")*."

- Sec 16.1: Added method modifications to include exceptions to drying time procedures.
- Screen shots re-cropped throughout document.

- Revision 6, dated 06 December 2012
  - Sec 1 and 12: Updated LQM references as per the most current LQM revision.
  - Various sections in SOP where applicable: Revised the temperature range of the drying oven from 103-105°C to 100 – 110°C to comply with the method.
  - Section 10.3: Added to include the use of the oven temperature chart (WI# EDS-WI-117); subsequent sections adjusted accordingly.
  - Section 10.4 and 10.25: Added to include the documentation of 'corrected and uncorrected temperature.'
  - Sec 11.4.1: Revised to include 'corrected and uncorrected temperature.'
  - Sec 11.4.3: Added the review and storage information of the oven's monthly temperature charts.
  - Sec 15.9: Added applicable reference.

- Revision 5, dated 24 October 2011
  - Section 10.20: Added to reflect the requirement of the ISM01.2 Exhibit D Section 1.6.3; subsequent sections adjusted accordingly.
  - Sec 11.4: Data reduction added to include data review requirements and to clarify information required in batch worksheet.
  - Sec 15: Added applicable references.

- Revision 4, dated 16 February 2010
  - Revised SOP format in accordance with TestAmerica Corporate Quality SOP No. CW-QS-002 (Rev 0) Writing a Standard Operating Procedure (SOP).
  - Updated to include procedures used in TALS (TestAmerica LIMS)
  - Updated method reference to current USEPA CLP SOW; deleted ASTM D2974-87 as method reference.
  - Section 3: revised to reference new location for definitions.
  - Section 5: Revised to include most up to date corporate health and safety references and information.
  - Section 9.1: Expanded QC sample preparation, analysis, evaluation and corrective action details.
  - Section 14.2: updated waste disposal procedures.
  - Section 15.0 (References): Expanded to include more specific SOP references
  - Section 18: Added this Revision History section.

APPENDIX C

QUALIFICATIONS OF PROPOSED STAFF

DEBORAH G. SHAPIRO, QEP

SENIOR VICE PRESIDENT

Deborah Shapiro, QEP is a Senior Vice President with experience in the assessment and remediation of hazardous waste issues. Ms. Shapiro supervises project teams and manages all aspects of assessment and remediation projects. Ms. Shapiro works with developers, non-profit organizations, architects, local community groups, local businesses, and government agencies. Her projects fall under the regulatory oversight of New York State Department of Environmental Conservation (NYSDEC), New York City Department of Environmental Protection (NYCDEP), and New York City Office of Environmental Remediation (NYCOER) including the New York State Brownfield Cleanup Program (BCP), New York City Voluntary Cleanup Program (VCP), NYSDEC petroleum spills program, Resource Conservation and Recovery Act (RCRA)/Underground Injection Control (UIC) closures, and NYCOER's E-designation program. Ms. Shapiro has also assisted commercial and industrial property owners with maintaining the integrity of their portfolios by providing compliance related cleanup and chemical storage management services.

Ms. Shapiro manages all aspects of redevelopment projects from the initial Phase I ESA, Phase II, and remediation through post-remedial site management. In addition, her experience includes groundwater investigations, monitoring, and sampling programs; Brownfield and hazardous waste site investigations; In-Situ Chemical Oxidation; underground storage tank studies, including soil contamination delineation, classification, removal and disposal; waste characterization sampling; exposure assessments; on-going remedial action (especially air sparging (AS)/soil vapor extraction (SVE)), and permitting.

BACKGROUND

Education

MS, American University, Environmental Science, 2001
BA, American University, Environmental Studies, 1998

Licenses/Certifications

Health and Safety Operations at Hazardous Materials Sites 29 CFR 1910.120
OSHA 10 Hour Construction Safety & Health Course
OSHA 40 Hour HAZWOPER
OSHA 8 Hour Refresher
OSHA 8 Hour Supervisor
Qualified Environmental Professional, Institute of Professional Environmental Practice

Professional Memberships

Past President, New York City Brownfield Partnership,
Board Member, Residents Forward,
Member, Institute of Professional Environmental Practice,

Years of Experience

22 years in the industry
7 years with AKRF

RELEVANT EXPERIENCE

New York City Office of Environmental Remediation, OER On Call Contract, Various Locations, NY

The work has included conducting Phase I environmental site assessments (ESAs) and multi-media sampling of soil, groundwater, and soil vapor for various sites funded by EPA grants. The work plans and investigation reports were completed in accordance with OER and EPA requirements. AKRF also implemented a remedial plan for capping a

park site in Staten Island. In addition, AKRF provided support to OER and an affordable housing developer to expedite an application for entry into the New York State Department of Environmental Conservation (NYSDEC) Brownfield Cleanup Program (BCP), as well as preparation and implementation of the remedial investigation and remedial plan.

As Project Manager, Ms. Shapiro is managing an on-call contract with the OER for brownfields environmental assessment and remediation.

Brook 156 HDFC, Brook 156, Bronx, NY

AKRF was retained to provide environmental consulting services in connection with the purchase and development of the Site. AKRF prepared a Phase I Environmental Site Assessment (ESA) of the NYC-owned former gasoline service station and a former railroad. A Tier 1 Vapor Encroachment Screening was also conducted to satisfy HUD's vapor intrusion requirements. AKRF prepared a Remedial Investigation Work Plan (RIWP) and conducted a Remedial Investigation (RI) at the site, which included the collection and analysis of soil, soil vapor, and groundwater. The results of the RI, which were documented in a Remedial Investigation Report (RIR), were used to prepare a New York City Brownfield Cleanup Program (NYCBCP) application. The site was accepted into the New York State Brownfield Cleanup Program (NYSBCP). AKRF prepared a Citizen Participation Plan (CPP), distributed public notices, and conducted multiple Remedial Investigations to further investigate soil, soil vapor, and groundwater at the site prior to redevelopment. The results of the investigations were used to prepare a Remedial Action Work Plan (RAWP), which is undergoing review and approval by NYSDEC. The proposed remedy includes excavation of soil, design and installation of a soil vapor extraction system and sub-slab depressurization system, contingent groundwater treatment program, and installation of a vapor barrier and composite cover system.

AS Project Manager, Ms. Shapiro is responsible for managing all technical components of the project, communication with NYSDEC and the Client, and managing the budget.

Elton Crossing - Melrose Commons North Site C, Bronx, NY

AKRF provided environmental consulting services in connection with the purchase and redevelopment of the Elton Crossing site at 899 Elton Avenue in the Bronx, NY. The work initially involved the preparation of a Phase II subsurface investigation including soil and soil vapor testing to determine if the site would be eligible for the New York State Brownfield Cleanup Program (NYSBCP). Upon completion of the investigation, AKRF prepared a NYCBCP Application and the site was accepted into the NYSBCP. AKRF managed all aspects of the brownfield cleanup including; development of Investigation Work Plans, performing Remedial Investigations and Reports, preparation of Phase I ESAs, preparation of a Citizen Participation Plan, distribution of public notices, preparation and implementation of a Remedial Action Work Plan (RAWP), design of a sub-slab depressurization system, preparation of the Final Engineering Report and Site Management Plan, and sampling and management of soil disposal. AKRF is in the midst of implementing the Site Management Plan.

As Project Manager, Ms. Shapiro was responsible for managing all technical components of the project, communication with NYSDEC and the Client, and managing the budget.

Bradhurst Cornerstone II Residences, New York, NY

AKRF, Inc. prepared a Part 58 Environmental Assessment (EA) and a NYC CEQR Environmental Assessment Statement for the Bradhurst Cornerstone II Apartments project. This project, which required conveyance of City-owned property to the applicant and HOME funding from the HUD, will result in the construction of 31 units of affordable

housing on four sites in the Harlem neighborhood of Manhattan. The New York City Department of Housing Preservation & Development (HPD) served as lead agency for the review and has issued a Negative Declaration for the project. Issues of concern for the environmental review included the identification of project commitments for certain of the four sites related to historic resources, hazardous materials, air quality, and building attenuation. As part of the mitigation of hazardous materials, AKRF conducted a Phase II investigation and prepared a RAP and CHASP.

AKRF prepared a Construction Protection Plan that was reviewed and approved by the New York City Landmarks Preservation Commission and the New York State Office of Parks, Recreation and Historic Preservation. This plan was implemented during construction to protect the Wadleigh Secondary School for the Performing and Visual Arts, a New York City Landmark that is also eligible for listing on the State and National Registers.

As Project Manager, Ms. Shapiro was responsible for managing all technical components of the hazardous materials portion of the project, communication with the regulatory agency and the Client, and managing the budget.

Lambert Houses Redevelopment, Bronx, NY

AKRF performed an Environmental Impact Statement (EIS) of the Lambert Houses affordable housing complex located in the West Farms section of the Bronx, NY. Lambert Houses consisted of multi-story apartment buildings, parking garage, and a multi-tenant retail/commercial building alongside the elevated NYC subway. AKRF also conducted a Phase I ESA with a vapor intrusion screen of the Property to satisfy U.S. Department of Housing and Urban Development (HUD)'s vapor intrusion requirements. The Phase I and vapor intrusion screens were prepared in accordance with ASTM E1527-05, ASTM E2600, and U.S. Environmental Protection Agency (EPA)'s All Appropriate Inquiry (AAI) rule. After completion of the EIS, an E-designation for hazardous materials was placed on the site. A subsurface investigation was conducted and a Remedial Action Work Plan (RAWP) was prepared under New York City Office of Environmental Remediation (OER) oversight. The site was subsequently entered in the NYC Voluntary Cleanup Program. AKRF is in the midst of implementing the RAWP, which includes remediation of a hydraulic oil spill.

Ms. Shapiro was responsible for managing all technical components of the hazardous materials portion of the project, communication with the regulatory agency and the Client, and managing the budget.

New York City Office of Environmental Remediation, Second Farms, Bronx, NY

AKRF, Inc. was initially contracted by the New York City Office of Environmental Remediation (NYCOER) to conduct a subsurface investigation of a 1.12-acre parcel in the Bronx, New York under the United States Environmental Protection Agency (USEPA) Brownfield Assessment Grant program. The investigation included a geophysical survey and utility mark-outs, and the collection and analysis of soil, groundwater, soil vapor, indoor air and ambient air samples. AKRF continued working on the project for the developer by preparing a Remedial Action Plan and Environmental Assessment Statement. AKRF is in the midst of implementing the remedy.

As Project Manager, Ms. Shapiro was responsible for managing all technical components of the project, communication with OER, NYCDEP, and the Client, and managing the budget.

3301 Atlantic Avenue, Brooklyn, NY

AKRF was retained to provide environmental consulting services in connection with the purchase and redevelopment of former burned manufacturing buildings encompassing an entire city block in Brooklyn, New York. As part of due diligence, AKRF prepared a Phase I Environmental Site Assessment (ESA) Report for the property. After acquisition, the property was divided into three separate sites (3264 Fulton Street, 235 Chestnut Street, and 3301 Atlantic Avenue).

AKRF prepared a Subsurface (Phase II) Investigation Work Plans and conducted Phase IIs at each of the sites, which included the collection and analysis of soil, soil vapor, and groundwater samples. Based on the results of the Phase IIs, which were documented in Subsurface (Phase II) Reports, New York State Brownfield Cleanup Program (NYSBCP) applications were prepared for each of the sites. After acceptance into the NYSBCP, AKRF prepared Citizen Participation Plans (CPPs) and distributed public notices. AKRF prepared Remedial Investigation (RI) Work Plans (RIWPs) and implemented numerous Remediation Investigations for each of the sites to further investigate contaminated media at the site prior to redevelopment, and prepared the RI Reports (RIRs). AKRF is in the midst of preparing Interim Remedial Work Plans for each Site, which include installation of a Soil Vapor Extraction to prevent the off-site migration of contaminants.

As Project Manager, Ms. Shapiro was responsible for managing all technical components of the project, communication with NYSDEC and the Client, and managing the budget.

Atlantic Chestnut Lots 1, 2 & 3, Brooklyn, NY

AKRF was retained to provide environmental consulting services in connection with the purchase and redevelopment of former burned manufacturing buildings encompassing an entire city block in Brooklyn, New York. As part of due diligence, AKRF prepared a Phase I Environmental Site Assessment (ESA) Report for the property. After acquisition, the property was divided into three separate sites (3264 Fulton Street, 235 Chestnut Street, and 3301 Atlantic Avenue). AKRF prepared a Subsurface (Phase II) Investigation Work Plans and conducted Phase IIs at each of the sites, which included the collection and analysis of soil, soil vapor, and groundwater samples. Based on the results of the Phase IIs, which were documented in Subsurface (Phase II) Reports, New York State Brownfield Cleanup Program (NYSBCP) applications were prepared for each of the sites. After acceptance into the NYSBCP, AKRF prepared Citizen Participation Plans (CPPs) and distributed public notices. AKRF prepared Remedial Investigation (RI) Work Plans (RIWPs) for each of the sites to further investigate contaminated media prior to redevelopment, conducted the RIs, and is in the process of preparing the RI Reports (RIRs).

As Project Manager, Ms. Shapiro was responsible for managing all technical components of the project, communication with NYSDEC and the Client, and managing the budget.

MICHELLE LAPIN, P.E.

SENIOR VICE PRESIDENT

Michelle Lapin is a Senior Vice President with more than 30 years of experience in the assessment and remediation of hazardous waste issues. She leads the firm's Hazardous Materials group and offers extensive experience providing strategic planning and management for clients. Ms. Lapin has been responsible for the administration of technical solutions to contaminated soil, groundwater, air and geotechnical problems. Her other duties have included technical and report review, proposal writing, scheduling, budgeting, and acting as liaison between clients and regulatory agencies, and project coordination with federal, state, and local authorities.

Ms. Lapin's hydrogeologic experience includes groundwater investigations, formulation and administration of groundwater monitoring programs and remediation throughout the Northeast. Her experience with groundwater contamination includes Level B hazardous waste site investigations; leaking underground storage tank studies, including hazardous soil removal and disposal and associated soil and water issues; soil gas/vapor intrusion surveys; and wetlands issues. Ms. Lapin is experienced in coordinating and monitoring field programs concerning hazardous waste cell closures. She has directed hundreds of Phase I, Phase II, and Phase III investigations and remediations, many of them in conjunction with developers, law firms, lending institutions, and national retail chains. She is also experienced in the cleanup of contaminated properties under Brownfield Cleanup Program (BCP) and Voluntary Cleanup Program (VCP) regulations.

BACKGROUND

Education

M.S., Civil Engineering, Syracuse University, 1985
B.S., Civil Engineering, Clarkson University, 1983

Professional Licenses/Certifications

New York State P.E.
State of Connecticut P.E.

Professional Memberships

Member, National Society of Professional Engineers (NSPE), National and CT Chapters
Member, American Society of Civil Engineers (ASCE), National and CT Chapters
Member, Connecticut Business & Industry Association (CBIA), CBIA Environmental Policies Council (EPC)
Member, Environmental Professionals' Organization of Connecticut (EPOC)
Board Member, New York City Brownfield Partnership
Member, NAIOP, a Commercial Real Estate Development Association

Years of Experience

Year started in company: 1994
Year started in industry: 1986

RELEVANT EXPERIENCE

11833, 11934, 11935 Manhattan West, Manhattan, NY - NYC OER and USEPA
AKRF is providing environmental consulting services to Brookfield Office Properties in connection with the Manhattan West development site, which encompasses an entire city-block above the Amtrak approach to Penn Station. The four towers that comprise the Manhattan west development site are being remediated as four different

sites under the New York City Mayor's Office of Environmental Remediation (OER), due to an E-Designation for hazardous materials, air quality, and noise attenuation. Ms. Lapin is the Remedial Engineer for the project, and oversees all remedial activities.

20111 85 Jay Street, Brooklyn, NY - NYS Brownfield Redevelopment

AKRF's work includes the preparation and implementation of a NYSDEC-approved Remedial Action Work Plan for this approximately three-acre former industrial site that encompasses an entire city-block. The remediation is being conducted under the NYSDEC Brownfield Cleanup Program, primarily due to high levels of lead associated with former smelting operations. Ms. Lapin is the Remedial Engineer for this project and oversees all remedial activities.

11901 Elton Crossing (Melrose C - Family), Bronx, NY - NYS Brownfield Redevelopment

AKRF's work includes the implementation of the NYSDEC-approved Remedial Action Work Plan for this former industrial property, including: in-situ testing, off-site transport, the closure of two petroleum spills; the registration, removal, and closure of five petroleum storage tanks encountered during excavation; and the delineation of soil contaminants, including hazardous lead, petroleum, and pesticides. Ms. Lapin was the Remedial Engineer for the project, and oversaw all remedial activities.

70004 Yonkers Waterfront Redevelopment Project, Yonkers, NY

For this redevelopment along Yonkers' Hudson River waterfront, Ms. Lapin headed the remedial investigation and remediation work that included Phase I Environmental Site Assessments of 12 parcels, investigations of underground storage tank removals and associated soil remediation, remedial alternatives reports, and remedial work plans for multiple parcels. Several of the city-owned parcels were remediated under a Voluntary Cleanup Agreement; others were administered with state Brownfields grants. Hazardous waste remediation was completed on both brownfield and voluntary clean-up parcels, which enabled construction of mixed-use retail, residential development, and parking.

12492, 12493, 12184 Atlantic Chestnut, Brooklyn, NY

AKRF was retained by Phipps Houses to provide environmental consulting services in connection with the purchase and development of former burned manufacturing buildings encompassing an entire city block in Brooklyn, New York. As part of due diligence, AKRF prepared a Phase I Environmental Site Assessment (ESA) Report for the property. After acquisition, the property was divided into three separate sites (3264 Fulton Street, 235 Chestnut Street, and 3301 Atlantic Avenue). AKRF prepared a Subsurface (Phase II) Investigation Work Plans and conducted Phase IIs at each of the sites, which included the collection and analysis of soil, soil vapor, and groundwater samples. Based on the results of the Phase IIs, documented in Subsurface (Phase II) Reports, New York State Brownfield Cleanup Program (NYSBCP) applications were prepared for each of the sites. After acceptance into the NYSBCP, AKRF prepared Citizen Participation Plans (CPPs) and distributed public notices. AKRF prepared Remedial Investigation (RI) Work Plans (RIWPs) for each of the sites to further investigate contaminated media prior to redevelopment, conducted the RIs, and is in the process of preparing the RI Reports (RIRs). Ms. Lapin is the Remedial Engineer for the project, and oversees all remedial activities.

10321 West 61st Street Rezoning/Residential Development, New York, NY

Ms. Lapin directed the firm's hazardous materials work for this mixed-use development in Manhattan. The Algin Management Company hired AKRF to prepare an environmental impact statement (EIS) for the proposed rezoning of the western portion of the block between West 60th and 61st Streets, between Amsterdam and West End Avenues. The purpose of the proposed action was to facilitate the development of two 30-story residential towers with accessory parking spaces, and landscaped open space. The EIS examined a "worst case" condition for rezoning the block, which allowed Algin to build a residential building of approximately 375,000 square feet at their site. The building now contains 475 apartments, 200 accessory parking spaces, a health club, and community facility space. This site, with the services of AKRF, entered into New York State's Brownfield Cleanup Program (BCP). On-site

issues included underground storage tanks remaining from previous on-site buildings, petroleum contamination from these tanks and possibly from off-site sources, and other soil contaminants (metals, semi-volatile organic compounds, etc.) from fill materials and previous on-site buildings. AKRF oversaw the adherence to the Construction Health and Safety Plan (HASP), which was submitted to and approved by the New York State Department of Environmental Conservation (NYSDEC), and monitored the waste streams, to ensure that the different types of waste were disposed of at the correct receiving facilities. This oversight also included confirmation and characteristic soil sampling for the receiving facilities and NYSDEC. A "Track 1" Clean up of the majority of the property (the portion including the buildings) was completed and the final Engineering Report was approved by the NYSDEC. AKRF has also completed a smaller portion of the property as a "Track 4" cleanup, which includes a tennis court and landscaped areas. Ms. Lapin continues to manage the annual inspections for the property owner in accordance with the Brownfield Cleanup Agreement.

11160 2477 Third Avenue, Bronx, NY

AKRF conducted the investigation and remediation of the former 2477 Third Avenue gasoline station property under the New York State Department of Environmental Conservation's (NYSDEC's) Brownfield Cleanup Program (BCP). The work included shallow and deep aquifer groundwater testing, delineation of known areas of soil contamination, soil vapor analyses, and investigation and delineation of non-aqueous phase liquid (DNAPL) from past industrial activities. Upon NYSDEC approval of the Remedial Action Work Plan (RAWP), AKRF conducted the removal of the nine on-site underground storage tanks (USTs) and 1,100 tons of petroleum-contaminated soil, the application of six in-situ chemical oxidation (ISCO) groundwater treatments, and the implementation of four Enhanced Fluid Recovery (EFR) events to remove desorbed gasoline-related hydrocarbons in the groundwater. The site received a Certificate of Completion (COC) from the BCP in December 2015 and a Notice of Satisfaction (NOS) in October 2016 from the Mayor's Office of Environmental Remediation (OER) in connection with the hazardous materials E-Designation assigned to the property. Ms. Lapin was the professional engineer of record, responsible for the remediation design elements and overall adherence to the NYSDEC and New York City Office of Environmental Remediation (OER) regulations.

11430 164 Kent Avenue, Brooklyn, NY (AKA Northside Piers and 1 North 4th Place)

The project was a multi-phase development consisting of a large waterfront block in the Williamsburg Rezoning Area. The project site was developed with a mixed-use residential-commercial high rise towers with an esplanade and a pier along the East River. AKRF provided acquisition and development support, including performing Phase I and II environmental site assessments, and preparation of Remedial Action Plans (RAPs) and Construction Health and Safety Plan (CHASPs) for approval by New York City Department of Environmental Protection (DEP) and New York City Mayor's Office of Environmental Remediation (OER). AKRF provided assistance with construction oversight during soil handling activities and managing the Community Air Monitoring Plan (CAMP) activities. To date, closure reports have been prepared and occupancy achieved for three of the four buildings. Ms. Lapin is the Professional Engineer (P.E.) of record for the DEP and OER RAPs, CHASPs and Remedial Closure Reports (RCRs).

11646 443 Greenwich Street, Manhattan, NY

This Site was assigned an E-Designation for hazardous materials (and air quality and noise) during the North Tribeca Rezoning in 2010, which requires environmental testing and, if necessary, remediation to the satisfaction of the New York City Mayor's Office of Environmental Remediation (OER). After years of public opposition to the original redevelopment scheme calling for a boutique hotel, this former manufacturing building and its current developer gained acceptance through the Department of City Planning and the Landmarks Preservation Commission to move forward with redevelopment as residential lofts. The redevelopment process began in 2012 and led to initial re-occupancy in 2016 after overcoming several regulatory challenges while seeking LEED® certification.

Once trichloroethene (TCE) was identified on-site, the typically straight forward assignment of delineating contaminant sources for AKRF became much more complex following the identification of an off-site TCE

groundwater plume. Based on the completion of several rounds of additional sampling and investigation activities including a compound specific isotopic analysis (CSIA) of the chlorinated volatile organic compounds (VOCs) detected in the central portion of the Site and the off-site monitor wells south of the Site, the presence of two separate releases (one originating on-site and one originating off-site) of TCE was confirmed. Based on the confirmation that the Site was not the contamination source associated with the off-site plume, the redevelopment of the Site proceeded under the review of the OER, and did not require direct or continued oversight from the New York State Department of Environmental Conservation (NYSDEC). Furthermore, the developer of the Site, who had become the owner, was not deemed responsible to complete additional off-site investigation or remediation associated with the separate, off-site TCE groundwater plume.

For this project, AKRF utilized forensic-based analysis of chlorinated VOC plumes and was one of the first projects that included a groundwater treatment technology managed by the OER in its E-Designation program. The Site also includes an engineered cap to prevent exposure to underlying soil/fill, a vapor barrier/waterproofing system beneath the building slab and along foundation sidewalls, and the operation of an active sub-slab depressurization (SSD) system. The project was awarded the 2017 Environmental Protection award by the New York City Brownfield Partnership. Ms. Lapin was the professional engineer of record, responsible for the remediation design and adherence of the remediation and remediation systems installation and ongoing operation.

12538 Larkin Plaza, Yonkers, NY – Remedial Investigation, Construction Oversight

AKRF assisted RXR Realty with enrolling the 1.1-acre Larkin Plaza site in the New York State Department of Environmental Conservation's (NYSDEC's) Brownfield Cleanup Program (BCP). Since being accepted into the program, AKRF conducted an extensive remedial investigation, prepared the necessary remedial action plans, managed the citizen participation tasks, and is in the process of conducting the remediation in conjunction with NYSDEC oversight. To date, the remedial work has included in-situ chemical oxidation (ISCO) treatments, contaminated soil removal, and petroleum product recovery. AKRF also assisted RXR with various construction-related services, including dewatering discharge permitting, soil disposal characterization testing, and stormwater pollution prevention plan (SWPPP) preparation. AKRF's Cultural Resources department is in the process of preparing a submission to the State Historic Preservation Office (SHPO) on behalf of RXR related to the acquisition of additional public funding sources for the construction project. A Certificate of Completion (COC) from the NYSDEC is anticipated at the end of 2018. Ms. Lapin is the professional engineer of record, responsible for the remediation design elements and adherence to the NYSDEC-approved work plans and remediation design.

REFERENCES

Michael Bogin; Sive, Paget & Riesel, P.C.; 460 Park Avenue, 10th Floor, New York, NY 10022; T: (212) 421-2150; 210; E: mbogin@sprlaw.com

Steve Novenstein; CEO; UOVO; Queens Plaza, 41-54 22nd Street Long Island City, NY 11101; T: (212) 904-0406; E: snovenstein@uovo.org

L. Ryan Kiefer, Sr. Project Manager; Memorial Sloan-Kettering Cancer Center; 307 East 63rd Street, 2nd Floor, New York, NY 10065; T: (646) 888-8449; E: rkiefer@mskcc.org

TIMOTHY MCCLINTOCK

ENVIRONMENTAL SCIENTIST

Timothy McClintock has over 12 years of environmental consulting experience primarily in environmental investigation, remediation oversight and project management throughout the northeast. His experience includes writing proposals; planning, implementing and managing Phase I Environmental Site Assessments, Phase II Environmental Site Investigations and Remedial Investigations; overseeing remedial action programs including soil excavation, groundwater handling, remediation system installation, and operation and maintenance; project management and reporting. Many of his remediation projects have been successfully remediated and obtained closure from the New York State Department of Environmental Conservation (NYSDEC), New Jersey Department of Environmental Protection (NJDEP), Pennsylvania Department of Department of Environmental Protection (PADEP), Connecticut Department of Energy & Environmental Protection (CTDEEP) and Massachusetts Department of Environmental Protection (MassDEP).

BACKGROUND

Education
B.S. Environmental Science/Earth Science, University at Albany, 2008

Licenses & Certifications
OSHA 40-hour Health & Safety Training for Hazardous Waste Operations (February, 2018)
NJDEP Subsurface Evaluator & UST Closure (December, 2019)
NYSDOH Certified Asbestos Inspector & Mold Assessor (November, 2018)
USEPA Lead Paint Inspector (October, 2017)

Years of Experience
Date started at AKRF: August, 2017
Prior industry experience: Dorson Environmental Management, Inc. – August 2008 to August 2017 (9 years)

RELEVANT EXPERIENCE

Former Farm Gasoline Underground Storage Tank Remediation, Somerset County, NJ

Mr. McClintock has been serving as the field team leader and deputy project manager for the LSRP-led remediation of a former gasoline UST located on a farm property in the Watchung Mountain region of New Jersey. Following performance of a site investigation (contamination screening), AKRF is currently conducting an investigation to bioremediate groundwater at the site, which contains primarily residual benzene and MTBE. As the project environmental scientist, Mr. McClintock assisted with the preparation of a Remedial Action Workplan (RAWP), and completed groundwater sampling investigations, a biotreatability study, and an evaluation of data resulting from the site work.

White Plains Mall, 200 Hamilton Avenue, White Plains, New York - Spill Investigation and Brownfield Cleanup Program Enrollment

Mr. McClintock served as the field team leader for the Spill Investigation work associated with historic gasoline stations at the White Plains Mall. As the project environmental scientist, Mr. McClintock assisted senior project staff with the evaluation of historical assessment information, and the development and implementation of a Spill Investigation to delineate the extent of petroleum-contaminated soil and groundwater. Mr. McClintock directed the field effort including the collection of soil and groundwater samples, completed an evaluation of data resulting from the site work, and prepared investigation reports. The project would apply for the NYSDEC Brownfield Cleanup Program.

New York City School Construction Authority, New York, NY

AKRF provides the New York City School Construction Authority (NYCSCA) with hazardous materials consulting services under an on-call contract. Mr. McClintock has served as field team leader and deputy project manager at various NYCSCA project sites related to due diligence and environmental assessments. As the project environmental scientist, Mr. McClintock has completed Phase I Environmental Site Assessments (ESAs), Phase II (Subsurface) Investigations, Indoor Air Quality (IAQ) Assessments, and underground storage tank (UST) and aboveground storage tank (AST) inspections and closures.

Confidential Client: New York City Institutional Site - Soil Classification:

Mr. McClintock served as the field team leader for the soil classification work associated with the proposed development of an addition at a New York City institutional site. As the project environmental scientist, Mr. McClintock assisted senior project staff with the coordination of site work, and directed the field effort including the collection of soil samples to characterize the current subsurface conditions. Mr. McClintock's role also included evaluating the data resulting from the site work and project reporting.

Proposed Public School, Queens, New York - Phase II Investigation

Mr. McClintock served as field team leader for the Phase II Investigation work associated with proposed development of NYCDOE Public School at a vacant lot in Queens, New York. As the project environmental scientist, Mr. McClintock assisted senior project staff with the evaluation of historical assessment information and the development and implementation of the Phase II Investigation to characterize the current subsurface conditions. Mr. McClintock directed the field effort including the collection of soil, soil vapor, and groundwater samples, completed an evaluation of the data resulting from the site work, and prepared investigation reports.

900 King Street Property, 900 King Street, Rye Brook, NY

Mr. McClintock served as field team leader for the Phase II Investigation work associated with proposed development of the 900 King Street property in Rye Brook, NY. As the project environmental scientist, Mr. McClintock assisted senior project staff with the evaluation of historical assessment information and the development and implementation of the Phase II Investigation to characterize the current subsurface conditions. Mr. McClintock directed the field effort including the collection of soil, soil vapor, and groundwater samples, completed an evaluation of the data resulting from the site work, and prepared investigation reports.

1-65 North 12th Street, Brooklyn, New York

The former Bayside Fuel Oil Company operated a commercial petroleum bulk storage facility at the property for several decades. Soil and groundwater contamination resultant of on-site and off-site petroleum releases, off-site manufactured gas plant (MGP) releases, and historic fill have been identified throughout the property. The site is currently owned by the City of New York Department of Parks and Recreation (DPR) and the New York City Economic Development Corporation (EDC) is implementing a demolition project to remove all above grade structures at the property. Mr. McClintock assisted senior AKRF project staff with the evaluation of historical assessment information and the preparation of a Remedial Action Plan (RAP) and Construction Health and Safety Plan (CHASP) for the proposed demolition project.

Petroleum Release/Oil Tank Remediation Projects – New York, New Jersey, Pennsylvania and Connecticut (2008 – 2017)

While at another firm, Mr. McClintock completed the design and implementation of environmental investigations and remediation projects associated with petroleum releases at residential and commercial sites throughout the northeast. Tasks included project design, site investigation, project direction and oversight, soil and groundwater

sampling, data evaluation, client and contractor coordination, regulatory agency interaction and associated reporting and deliverable production.

Phase I Environmental Site Assessment and Phase II Environmental Site Investigation Projects – New York and New Jersey (2008 – 2017)

During his time with Dorson Environmental Management, Inc., Mr. McClintock completed Phase I and Phase II environmental site assessments (ESAs) and investigations (ESIs) at residential and commercial properties associated with real estate transactions. Tasks included site inspections, historic environmental data report and regulatory record evaluations, environmental media sampling, client and contractor coordination, and associated reporting and deliverable production.

Storm Water Investigation Projects – New York (2008 – 2017)

Mr. McClintock assisted senior project staff with the investigation of actual and suspected storm water discharges at various sites throughout New York while at Dorson Environmental Management, Inc. Tasks included investigation into suspected non-permitted storm water discharges for environmental attorneys, preparation of Storm Water Pollution Prevention Plans (SWPPP) to assist property owners with obtaining the NYSDEC General Permit for Storm Water Discharges, storm water sampling, storm water drainage mapping, data evaluation, and associated reporting and deliverable production.

Former Flamingo Cleaners, 149 North Avenue, New Rochelle, New York

Mr. McClintock completed site investigations, developed a Remedial Action Work Plan (RAWP), provided remediation oversight and regulatory agency interaction, conducted environmental media sampling, and prepared report packages associated with comingled petroleum and chlorinated contamination at a former dry cleaning facility. The work was conducted in accordance with the NYSDEC Brownfield Cleanup Program and included site characterization, excavation and disposal of contaminated source material, removal and treatment of contaminated groundwater, in situ chemical oxidation of residual contamination and the implementation of institutional and engineering controls.

Former Gasoline Station, 66 Milton Road, Rye, NY

Mr. McClintock designed and implemented a site investigation and remedial excavation program to address historic contamination at a former gasoline station. The site work included the delineation of the residual soil and groundwater contamination, excavation of contaminated source material, removal of contaminated groundwater, post-remedial soil and groundwater sampling and associated reporting to close the NYSDEC spill number associated with the property. All site work was coordinated through the current building management and tenant association, the NYSDEC and the City of Rye.

Water and Mold Damage Investigation and Remediation Projects – New York, New Jersey, and Connecticut (2008 – 2017)

Mr. McClintock completed the design and implementation of environmental investigations, cause and origin analyses, and remedial projects associated with water and mold damage claims for various insurance carriers during his tenure with Dorson Environmental Management. Tasks included site investigation, cause and origin determination, project direction and oversight, environmental media sampling, data evaluation, client and contractor coordination, and associated reporting and deliverable production.

MICHAEL BATES

GEOLOGIST – SITE ASSESSMENT AND REMEDIATION

Michael Bates is an Geologist/Environmental Professional II in AKRF's Site Assessment and Remediation group, with experience in environmental sampling and monitoring during site remediation, subsurface and vapor intrusion investigations, remediation system operation and maintenance, and technical reporting.

BACKGROUND

Role in Project
Junior Environmental Scientist

EDUCATION

B.A. Geology, SUNY Geneseo, May 2017

CERTIFICATIONS

OSHA 40-hour Hazardous Waste Operations and Emergency Response Training
OSHA 30-hour Construction Safety Training
EPA Lead Risk Assessor
NY Certified Asbestos Inspector

YEARS OF EXPERIENCE

Date started at AKRF: October 2022
Prior industry experience: Wood PLC: May 2020-September 2022 (2 years, 4 months)
Roux Associates: April 2019-November 2019 (9 months)

RELEVANT EXPERIENCE - AKRF

Remedial Investigation – 221 Glenmore Avenue, Brooklyn, New York

AKRF is conducting a Remedial Investigation at a former lighting company facility in support of a NYSDEC Brownfield Cleanup Program application. Mr. Bates performed groundwater sampling of newly installed and existing monitoring wells at the Site in accordance with EPA low-flow sampling protocols.

Construction Oversight and Community Air Monitoring – BESS, Astoria, Queens, New York

AKRF prepared and is implementing a Construction Health and Safety Plan (CHASP, approved by the New York Power Authority) during construction of a stand-along new battery energy storage system at a Con Edison facility in Astoria Queens. Mr. Bates serves as an on-site environmental monitor during construction to ensure compliance with the CHASP. His duties include community and work zone air monitoring during utility clearance and waste characterization sampling.

RELEVANT EXPERIENCE – WOOD PLC, BAYSIDE, NY

As a Staff Geologist at Wood, Mr. Bates conducted subsurface investigations, low-flow groundwater sampling, and soil vapor sampling, and prepared associated technical reports. He also conducted routine O&M and monitoring of large groundwater and soil vapor treatment systems and sub-slab depressurization systems, and oversaw installation/rehabilitation of recovery wells for system upgrades.

RELEVANT EXPERIENCE – ROUX ASSOCIATES, ISLANDIA, NY

As a Staff Geologist at Roux, Mr. Bates performed field work for investigation and remediation of petroleum-contaminated sites in the New York metro areas, including soil and groundwater sampling, air monitoring, and oversight of UST closures/contaminated soil removal. In this role, Mr. Bates also reviewed and evaluated field and laboratory data and prepared technical reports to document the investigation and remediation activities.

L.A.B. Validation Corp., 14 West Point Drive, East Northport, New York 11731

Lori A. Beyer

SUMMARY: General Manager/Laboratory Director with a solid technical background combined with Management experience in environmental testing industry. Outstanding organizational, leadership, communication and technical skills. Customer focused, quality oriented professional with consistently high marks in customer/employee satisfaction.

EXPERIENCE:

1998-Present L.A.B. Validation Corporation, 14 West Point Drive, East Northport, NY
President

- Perform Data Validation activities relating to laboratory generated Organic and Inorganic Environmental Data.

1998-Present American Analytical Laboratories, LLC. 56 Toledo Street, Farmingdale, NY
Laboratory Director/Technical Director

- Plan, direct and control the operation, development and implementation of programs for the entire laboratory in order to meet AAL's financial and operational performance standards.
- Ensures that all operations are in compliance with AAL's QA manual and other appropriate regulatory requirements.
- Actively maintains a safe and healthy working environmental that is demanded by local laws/regulations.
- Monitors and manages group's performance with respect to data quality, on time delivery, safety, analyst development/goal achievement and any other key performance indices.
- Reviews work for accuracy and completeness prior to release of results to customers.

1996-1998 Nytest Environmental, Inc. (NEI) Port Washington, New York
General Manager

- Responsible for controlling the operation of an 18,000 square foot facility to meet NEI's financial and operational performance standards.
- Management of 65 FTEs including Sales and Operations
- Ensure that all operations are in compliance with NEI's QA procedures
- Ensures that productivity indicators, staffing levels and other cost factors are held within established guidelines
- Maintains a quantified model of laboratory's capacity and uses this model as the basis for controlling the flow of work into and through the lab so as to ensure that customer requirements and lab's revenue and contribution targets are achieved.

1994-1996 Nytest Environmental, Inc. (NEI) Port Washington, New York
Technical Project Manager

- Responsible for the coordination and implementation of environmental testing programs requirements between NEI and their customers
- Supervise Customer Service Department
- Assist in the development of major proposals
- Complete management of all Federal and State Contracts and assigned commercial contracts
- Provide technical assistance to the customer, including data validation and interpretation
- Review and implement Project specific QAPP's.

1995-1996 Nytest Environmental, Inc. (NEI) Port Washington, New York
Corporate QA/QC Officer

- Responsible for the implementation of QA practices as required in the NJDEP and EPA Contracts
- Primary contact for NJDEP QA/QC issues including SOP preparation, review and approval
- Responsible for review, verification and adherence to the Contract requirements and NEI QA Plan

1992-1994 Nytest Environmental, Inc. (NEI) Port Washington, New York
Data Review Manager

- Responsible for the accurate compilation, review and delivery of analytical data to the company's customers. Directly and effectively supervised a department of 22 personnel.
- Managed activities of the data processing software including method development, form creation, and production
- Implement new protocol requirements for report and data management formats
- Maintained control of data storage/archival areas as EPA/CLP document control officer

1987-1991 Nytest Environmental, Inc. (NEI) Port Washington, New York
Data Review Specialist

- Responsible for the review of GC, GC/MS, Metals and Wet Chemistry data in accordance with regulatory requirements
- Proficient with USEPA, NYSDEC, NJDEP and NEESA requirements
- Review data generated in accordance with SW846, NYSDEC ASP, EPA/CLP and 40 CFR Methodologies

1986-1987 Nytest Environmental, Inc (NEI) Port Washington, New York
GC/MS VOA Analyst

EDUCATION:

1982-1985 State University of New York at Stony Brook, New York; BS Biology/Biochemistry
1981-1982 University of Delaware; Biology/Chemistry
5/91 Rutgers University; Mass Spectral Data Interpretation Course, GC/MS Training
8/92 Westchester Community College; Organic Data Validation Course
9/93 Westchester Community College; Inorganic Data Validation Course

Westchester Community College

Professional Development Center

Awards this Certificate of Achievement To

LORI BEYER

for Successfully Completing

ORGANIC DATA VALIDATION COURSE (35 HOURS)

Dr. John Samuelian

Date AUGUST 1992

Assistant Dean
Professional Development Center

President

The Professional
Development Center

SUNY
WESTCHESTER COMMUNITY COLLEGE
Valhalla, New York 10595

Westchester Community College

Professional Development Center

Awards this Certificate of Achievement To

LORI BEYER

for Successfully Completing

INORGANIC DATA VALIDATION

Instructor: Dale Boshart

Date MARCH 1993

Assistant Dean
Professional Development Center

President

The Professional
Development Center

SUNY
WESTCHESTER COMMUNITY COLLEGE
Valhalla, New York 10595

New York State Department of Environmental Conservation
50 Wolf Road, Albany, New York 12233

Thomas C. Jorling
Commissioner

July 8, 1992

Ms. Elaine Sall
Program Coordinator
Westchester Community College
Valhalla, NY 10595-1698

Dear Elaine,

Thank you for your letter of June 29, 1992. I have reviewed the course outline for organic data validation, qualifications for teachers and qualifications for students. The course that you propose to offer would be deemed equivalent to that which is offered by EPA. The individuals who successfully complete the course and pass the final written exam would be acceptable to perform the task of organic data validation for the Department of Environmental Conservation, Division of Hazardous Waste Remediation.

As we have discussed in our conversation of July 7, 1992, you will forward to me prior to the August course deadline, the differences between the EPA SOW/90 and the NYSDEC ASP 12/91. You stated these differences will be compiled by Mr. John Samulian.

I strongly encourage you to offer an inorganic data validation course. I anticipate the same list of candidates would be interested in an inorganic validation course as well, since most of the data to be validated consists of both organic and inorganic data.

Thank you for you efforts and please contact me if I can be of any further assistance.

Sincerely,

Maureen P. Serafini

Maureen P. Serafini
Environmental Chemist II
Division of Hazardous Waste
Remediation

The Professional
Development Center
AT
WESTCHESTER COMMUNITY COLLEGE

914 285-6619

October 2, 1992

Ms. Lori Beyer
3 sparkill Drive
East Northport, NY 11731

Dear Ms. Beyer:

Congratulations upon successful completion of the Organic Data Validation course held August 17 - 21, 1992, through Westchester Community College, Professional Development Center. This course has been deemed by New York State Department of Environmental Conservation as equivalent to EPA's Organic Data Validation Course.

Enclosed is your Certificate. Holders of this Certificate are deemed competent to perform organic data validation for the New York State DEC Division of Hazardous Waste Remediation.

The Professional Development Center at Westchester Community College plans to continue to offer courses and seminars which will be valuable to environmental engineers, chemists and related personnel. Current plans include a TCLP seminar on November 17th and a conference on Environmental Monitoring Regulations on November 18th.

We look forward to seeing you again soon at another environmental program or event. Again, congratulations.

Very truly yours,

Passing Grade is 70%
Your Grade is 99%

Elaine Sall
Program Coordinator

ES/bf

SUNY
WESTCHESTER COMMUNITY COLLEGE
Valhalla, New York 10595

The Professional
Development Center
AT
WESTCHESTER COMMUNITY COLLEGE

914 285-6619

June 21, 1993

Dear Ms. Beyer:

Enclosed is your graded final examination in the Inorganic Data Validation course you completed this past March. A score of 70% was required in order to receive a certificate of satisfactory completion. Persons holding this certificate are deemed acceptable to perform Inorganic Data Validation for the New York State Department of Environmental Conservation, Division of Hazardous Waste Remediation.

I am also enclosing a course evaluation for you to complete if you have not already done so. The information you provide will greatly aid us in structuring further courses. We wish to make these course offerings as relevant, targeted and comprehensive as possible. Your evaluation is vital to that end.

Congratulations on your achievement. I look forward to seeing you again at another professional conference or course. We will be co-sponsoring an environmental monitoring conference on October 21, 1993 with the New York Water Pollution Control Association, Lower Hudson Chapter, at IBM's Yorktown Heights, NY site. Information regarding this event will be going out in August.

Very truly yours,

Elaine Sall

Elaine Sall
Program Coordinator

ES/bf

Enclosures

SUNY
WESTCHESTER COMMUNITY COLLEGE
Valhalla, New York 10595

eurofins | Environment Testing TestAmerica

Personnel Resume

Carl Armbruster
Quality Assurance Manager

Qualifications Summary

Mr. Armbruster began his career in the environmental testing business in 1983 His experience in the environmental laboratory and engineering industry includes extensive technical, management/leadership experience in all aspects of the laboratory business. He is an action-oriented manager dedicated to ensuring the laboratory maintains a quality program that values data integrity, continuous improvement and customer satisfaction. His unique experience lends itself to working successfully with employees, managers and clients at all levels.

Professional Experience

Quality Assurance Manager – Eurofins TestAmerica - 2005 to Present

Mr. Armbruster is responsible for establishment and implementation of the quality assurance program at the Edison facility; and for interfacing with the corporate Quality Assurance Director to ensure adherence with the overall Quality Management Plan. He is also responsible for monitoring implementation and compliance with NELAC and the company's QMP, conducting annual management system audits and data audits, as well as providing regulatory updates and technical support to the Laboratory Director, Operations Manager, Client Services and Sales department.

Project Manager/Assistant Technical Director – 2000 to 2005

Laboratory Director − 1998 to 2000

Account Manager − Clean Harbors Environmental Services − 1997 to 1998

Laboratory Manager − Waste Management Inc., and Chemical Waste Management Inc − 1988 to 1997

Environmental Scientist − ICF Technology − 1987 to 1988

Analytical Chemist − IT Corporation – 1985 to 1987

Analytical Chemist − Hess Environmental Laboratories – 1983 to 1985

Education

- MS in Biology – East Stroudsburg University, 1984
- BS in Environmental Studies - East Stroudsburg University, 1980

APPENDIX D

EUROFINS ENVIRONMENT TESTING OF AMERICA QUALITY ASSURANCE MANUAL

Quality Assurance Manual Cover Page

Eurofins Edison
777 New Durham Road
Edison, New Jersey 08817
Phone No. 732-549-3900
Fax No.732-549-3679

https://www.eurofinsus.com/environment-testing

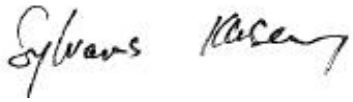
Facility Distribution No. Electronic **Distributed To: Facility Intranet**

1.0 Title Page

Quality Assurance Manual
Approval Signatures

Laboratory Director/Business Unit Manager-Mark Acierno — 09/13/2022 Date

Quality Assurance Manager - Carl Armbruster — 09/13/2022 Date



Technical Manager, Organics – Sylvanus Klusey — 09/13/2022 Date

Technical Manager, Inorganics – Donald Evans — 09/13/2022 Date

2.0 TABLE OF CONTENTS

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3.0 INTRODUCTION, SCOPE AND APPLICABILITY

3.1 Introduction and Compliance References

Eurofins Edison's Quality Assurance (QA) Manual is a document prepared to define the overall policies, organization objectives and functional responsibilities for achieving the laboratory's QA Program. Governing SOPs are in place within the organization to ensure the proper execution of this QA Manual (refer to Appendix 1). This manual and referenced documents are required reading for all personnel.

The laboratory is a team of people who work together to serve the health and environmental needs of society through science and technology. We offer comprehensive expertise in environmental laboratory applications and client relations and maintain a national perspective in terms of quality.

As such, this QA Manual has been prepared to assure compliance with The NELAC Institute (TNI) Standard, dated 2009 and 2016; ISO/IEC Guide 17025:2005 and 2017. Policies and procedures listed in Appendix 1 are compliant with the National Divisional Support Center (NDSC) Quality Management Plan (QMP) for Eurofins Environment Testing America and the various accreditation and certification programs which are held by the laboratory to support environmental work (Appendix 2). The QMP provides a summary of Eurofins Environment Testing America quality and data integrity system. It contains requirements and general guidelines under which all Eurofins Environment Testing America facilities shall conduct their operations.

Refer to Appendix 3 for a list of additional references for which this QA Manual is compliant; and Appendix 4 for the compliance crosswalk of this manual to the TNI & ISO/IEC Guide 17025 requirements.

3.2 Terms and Definitions

A QA Program is a system designed to ensure that data produced by the laboratory conforms to the standards set by state and/or federal regulations. The program functions at the local management level through company goals, from guidance at the executive management level, and at the analytical level through Standard Operating Procedures (SOPs) and quality control. Our program is designed to minimize systematic error, encourage constructive, documented problem solving, and provide a framework for continuous improvement within the organization to better serve our clients.

Refer to Appendix 5 for a list of Terms and Acronyms.

3.3 Scope / Fields of Testing

The laboratory analyzes a broad range of environmental and industrial samples. Sample matrices vary among drinking water, effluent water, groundwater, hazardous waste, sludge and soils. The QA Program contains specific procedures and methods to test samples of differing matrices for chemical and physical parameters. The Program also contains guidelines on maintaining documentation of analytical processes, reviewing results, servicing clients and tracking samples through the laboratory. The technical and service requirements of all analytical requests are thoroughly evaluated before commitments are made to accept the work. Measurements are made using published reference methods or methods developed and validated by the laboratory.

The methods covered by this manual include the most frequently requested methodologies needed to provide analytical services in the United States and its territories. The specific list of test methods used by the laboratory can be found our active LIMS method database. Our areas of expertise include:

Standard Services	Specialty Services
• Volatiles • Semivolatiles • Metals • Pesticides/PCBs/Herbicides • Petroleum Hydrocarbons • Waste Characterization • Non-Potable Water Testing • Drinking Water Testing • Soil and Surface Water Testing	• 1,4-Dioxane • Free Cyanide • PCBs by EPA 680

NOTE: All current certificates and scopes of accreditation are available on the laboratory's website at https://www.eurofinsus.com/environment-testing.

The approach of this manual is to define the minimum level of quality assurance and quality control necessary to meet these requirements. All methods performed by the laboratory shall meet these criteria as appropriate. In some instances, quality assurance project plans (QAPPs), project specific data quality objectives (DQOs) or local regulations may require criteria other than those contained in this manual. In these cases, the laboratory will abide by the requested criteria following review and acceptance of the requirements by the Laboratory President, Laboratory Director/Business Unit Manager and the Quality Assurance (QA) Manager. In some cases, QAPPs and DQOs may specify less stringent requirements. The Laboratory President,, Laboratory Director/Business Unit Manager and the QA Manager must determine if it is in the lab's best interest to follow the less stringent requirements.

3.4 Management of the Manual

3.4.1 Review Process

This manual is reviewed every two years by senior laboratory management to assure that it reflects current practices and meets the requirements of the laboratory's clients and regulators as well as the QMP. Occasionally, the manual may need changes in order to meet new or changing regulations and operations. The QA Manager will review the changes in the normal course of business and incorporate changes into revised sections of the document. All updates will be reviewed by the senior laboratory management staff. The laboratory updates and approves such changes according to our Document Control procedures (refer to SOP No. ED-GEN-002, *Document Control*).

4.0 MANAGEMENT REQUIREMENTS

4.1 Overview

The laboratory is a local operating unit of Eurofins Environment Testing America. The laboratory's operational and support staff have the day-to-day independent operational authority under the direction of the Laboratory President, Laboratory Director/Business Unit Manager and is supported by the NDSC QA team. The laboratory management staff includes directors,

managers and group leaders. The organizational chart of the management staff are presented in Figure 4-1. Individual departmental staff lists are maintained in the laboratory's internal intranet.

4.2 Roles and Responsibilities

In order for the QA Program to function properly, all members of the staff must clearly understand and meet their individual responsibilities as they relate to the quality program. The responsibility for quality resides with every employee of the laboratory. All employees have access to the QA Manual, are trained to this manual, and are responsible for upholding the standards therein. Each person carries out his/her daily tasks impartially and in a manner consistent with the goals and in accordance with the procedures in this manual and the laboratory's SOPs. The following descriptions briefly define each role in its relationship to the Quality Assurance Program.

4.2.1 Vice President of Quality and Environmental Health and Safety (VP-QA/EHS)

The Vice President (VP) of QA/EHS reports directly to Eurofins Environment Testing America Chief Operating Officer (COO). With the aid of the NDSC Quality Team Members, Business Unit Managers and Laboratory Directors, the VP-QA/EHS has the responsibility for the establishment, general overview and maintenance of the Quality Assurance and EH&S Programs within Eurofins Environment Testing America. Additional responsibilities include:

- Review of QA/QC and EHS aspects of NDSC Official Documents, national projects and expansions or changes in services.
- Work with various organizations outside of the laboratory to further the development of quality standards and represent the laboratory at various trade meetings.
- Prepare monthly reports for quality and EH&S metrics across the environmental testing laboratories and a summary of any quality and EH&S related initiatives and issues.
- With the assistance of the Executive Management and the EHS Managers, maintenance and implementation of the Eurofins Environment Testing America Environmental, Health and Safety Program.

4.2.2 Quality Directors

There are four (4) Quality Directors within NDSC that report directly to the VP-QA/EHS. These Quality Directors have oversight of the general overview and maintenance of the QA Program within the Eurofins Environment Testing America laboratories. Supported tasks include:

- Monitors laboratory internal audit findings;
- Identifies common laboratory weaknesses and monitors corrective action closures.
- Develops NDSC quality guidance documents and management tools for ensuring and improving compliance;
- Monitors and communicates DoD/DoE requirements;
- Monitors and communicates regulatory and certification requirements;
- Training and OnBoarding
- Laboratory assessments, mentoring, and interventions
- Track/drive root cause investigations and corrective action plans
- Builds knowledge base for preventive actions

4.2.3 Quality Information Manager

The Quality Information works directly with the NDSC Quality Directors and EHS Managers; and reports directly to the VP-QA/EHS. The Quality Information Manager is responsible for the management of:

- NDSC Official Documents
- TALS/LIMS Certification Module Data
- Company's Intranet website
- Company's Regulatory Limits Database
- Subcontract laboratory and approved vendor information
- Internal and External client support for various company groups (e.g., Client Services, EH&S, Legal, IT, Sales) for both quality and operational functions
- Communicate regulatory information and lists

4.2.4 Environmental Health and Safety (EH&S) Managers

There are 3 EH&S Managers within NDSC that report directly to the VP-QA/EHS. These EH&S Managers have oversight of the general overview and maintenance of the EH&S Program within the Eurofins Environment Testing America laboratories. Supported tasks include:

- Consolidation and tracking all safety and health-related information and reports for the company, and managing compliance activities for Eurofins Environment Testing America locations.
- Coordination/preparation of the Environmental, Health and Safety Manual Template that is used by each laboratory to prepare its own laboratory-specific Safety Manual/ CHP.
- Preparation of information and training materials for laboratory EHS Coordinators.
- Assistance in the coordination of employee exposure and medical monitoring programs to insure compliance with applicable safety and health regulations.
- Serving as Department of Transportation (D.O.T.) focal point and providing technical assistance to location management.
- Serving as Hazardous Waste Management main contact and providing technical assistance to location management.

4.2.5 Ethics and Compliance Officers (ECOs)

The NDSC VP-QA/EHS and Corporate Counsel are designated Ethics and Compliance Officer (ECO). Each ECO acts as a back-up to the other ECO and both are involved when data investigations occur. Each ECO has a direct line of communication to the entire executive management personnel and lab management staff.

The ECOs monitor and audit procedures to determine compliance with policies and to make recommendations for policy enhancements to the President, COO, Laboratory Director or other appropriate individuals within the laboratory. The ECO will assist the laboratory QA Manager in the coordination of internal auditing of ethical policy related activities and processes within the laboratory, in conjunction with the laboratory's regular internal auditing function.

The ECOs will also participate in investigations of alleged violations of policies and work with the appropriate internal departments to investigate misconduct, remedy the situation, and prevent recurrence of any such activity.

4.2.6 Laboratory Director

The Laboratory Director is responsible for the overall quality, safety, financial, technical, human resource and service performance of the whole laboratory and reports to their business unit President. The Laboratory Director is also responsible for any service centers connected with their laboratory that perform analytical tests, such as short holding time analyses for pH. The Laboratory Director provides the resources necessary to implement and maintain an effective and comprehensive Quality Assurance and Data Integrity Program.

Specific responsibilities include, but are not limited to:

- Provides one or more technical managers for the appropriate fields of testing. If the Technical Manager is absent for a period of time exceeding 15 consecutive calendar days, the Laboratory Director must designate another full time staff member meeting the qualifications of the Technical Manager to temporarily perform this function. If the absence exceeds 35 consecutive calendar days, the primary accrediting authority must be notified in writing.
- Ensures that all analysts and supervisors have the appropriate education and training to properly carry out the duties assigned to them and ensures that this training has been documented. Works with Eurofins Environment Testing Human Resources for hiring of new personnel.
- Ensures that personnel are free from any commercial, financial and other undue pressures which might adversely affect the quality of their work.
- Ensures company human resource policies are adhered to and maintained.
- Ensures that sufficient numbers of qualified personnel are employed to supervise and perform the work of the laboratory. Assesses laboratory capacity and workload.
- Ensures that appropriate corrective actions are taken to address analyses identified as requiring such actions by internal and external performance or procedural audits. Procedures that do not meet the standards set forth in the QA Manual or laboratory SOPs may be temporarily suspended by the Laboratory Director.
- Reviews and approves all SOPs prior to their implementation and ensures all approved SOPs are implemented and adhered to.
- Pursues and maintains appropriate laboratory certification and contract approvals. Supports ISO 17025 requirements.
- Ensures client specific reporting and quality control requirements are met.
- Contributes to the continuous improvement of the laboratory operations.
- Maintains an awareness of technical developments and regulatory requirements.
- Captains the management team, consisting of the QA Manager, the Technical Manager(s), and the Operations Manager as direct reports.

4.2.7 Quality Assurance (QA) Manager or Designee

The QA Manager has responsibility and authority to ensure the continuous implementation of the quality system at the laboratory where they work. The QA Manager is also responsible for any service centers connected with their laboratory that perform analytical tests, such as short holding time analyses for pH.

The QA Manager reports directly to the Laboratory Director and their NDSC Quality Director. This position is able to evaluate data objectively and perform assessments without outside (e.g., managerial) influence. The NDSC QA Team may be used as a resource in dealing with regulatory requirements, certifications and other quality assurance related items. The QA Manager directs the activities of the QA office to accomplish specific responsibilities, which include, but are not limited to:

- Serves as the focal point for QA/QC in the laboratory.
- Have functions independent from laboratory operations for which he/she has quality assurance oversight.
- Have documented training and/or experience in QA/QC procedures and the laboratory's Quality System.
- Have a general knowledge of the analytical test methods for which data audit/review is performed (and/or having the means of getting this information when needed).
- Arrange for or conducting internal audits on quality systems and the technical operation
- Notifying laboratory management of deficiencies in the quality system and ensuring corrective action is taken. Procedures that do not meet the standards set forth in the QA Manual or laboratory SOPs shall be investigated following procedures outlined in Section 14 and if deemed necessary may be temporarily suspended during the investigation.
- Maintaining and updating the QA Manual.
- Monitoring and evaluating laboratory certifications; scheduling proficiency testing samples.
- Monitoring and communicating regulatory changes that may affect the laboratory to management.
- Training and advising the laboratory staff on quality assurance/quality control procedures that are pertinent to their daily activities.
- The laboratory QA Manager will maintain records of all ethics-related training, including the type and proof of attendance.
- Maintain, improve, and evaluate the corrective action database and the corrective and preventive action systems.
- Objectively monitor standards of performance in quality control and quality assurance without outside (e.g., managerial) influence.
- Coordinating of document control of SOPs, MDLs, control limits, and miscellaneous forms and information.
- Performing technical data audits and method audits to ensure consistency and compliance with regulatory requirements.
- Review of external audit reports and data validation requests.
- Follow-up with audits to ensure client QAPP requirements are met.
- Establishment of reporting schedule and preparation of various quality reports for the Laboratory Director, clients and/or NDSC QA Team.
- Development of suggestions and recommendations to improve quality systems.
- Research of current state and federal requirements and guidelines.

- Participate in the vendor and supplier approval process, including subcontractors.
- Captains the QA team to enable communication and to distribute duties and responsibilities.
- Communication to the relevant regulatory authorities when there are management or facility changes that impact the laboratory.
- Ensuring communication & monitoring standards of performance to ensure that systems are in place to produce the level of quality as defined in this document.
- Evaluation of the thoroughness and effectiveness of training.
- Compliance with ISO 17025

4.2.8 Technical Manager or Designee

The Technical Manager(s) report(s) directly to the Operations Manager – Organic and Operations Manager – Inorganic. (Note: Technical Managers at the Edison lab serve a dual role as Department Supervisors. See Section 4.2.11). . He/she is accountable for all analyses and analysts under their experienced supervision and for compliance with the ISO 17025:2017 Standard. The scope of responsibility ranges from the new-hire process and existing technology through the ongoing training and development programs for existing analysts and new instrumentation. Specific responsibilities include, but are not limited to:

- Exercises day-to-day supervision of laboratory operations for the appropriate field of accreditation and reporting of results. Coordinating, writing, and reviewing preparation of all test methods, i. e., SOPs, with regard to quality, integrity, regulatory and optimum and efficient production techniques, and subsequent analyst training and interpretation of the SOPs for implementation and unusual project samples. He/she insures that the SOPs are properly managed and adhered to at the bench. He/she develops standard costing of SOPs to include supplies, labor, overhead, and capacity (design vs. demonstrated versus first-run yield) utilization.
- Reviewing and approving, with input from the QA Manager, proposals from marketing, in accordance with an established procedure for the review of requests and contracts. This procedure addresses the adequate definition of methods to be used for analysis and any limitations, the laboratory's capability and resources, the client's expectations. Differences are resolved before the contract is signed and work begins. A system documenting any significant changes is maintained, as well as pertinent discussions with the client regarding their requirements or the results of the analyses during the performance of the contract. All work subcontracted by the laboratory must be approved by the client. Any deviations from the contract must be disclosed to the client. Once the work has begun, any amendments to the contract must be discussed with the client and so documented.
- Monitoring the validity of the analyses performed and data generated in the laboratory. This activity begins with reviewing and supporting all new business contracts, insuring data quality, analyzing internal and external non-conformances to identify root cause issues and implementing the resulting corrective and preventive actions, facilitating the data review process (training, development, and accountability at the bench), and providing technical and troubleshooting expertise on routine and unusual or complex problems.
- Providing training and development programs to applicable laboratory staff as new hires and, subsequently, on a scheduled basis. Training includes instruction on calculations, instrumentation management to include troubleshooting and preventive maintenance.

- Enhancing efficiency and improving quality through technical advances and improved LIMS utilization. Capital forecasting and instrument life cycle planning for second generation methods and instruments as well as asset inventory management.
- Coordinating sample management from “cradle to grave,” insuring that no time is lost in locating samples.
- Scheduling all QA/QC-related requirements for compliance, e.g., MDLs, etc.
- Captains department personnel to communicate quality, technical, personnel, and instrumental issues for a consistent team approach.
- Coordinates audit responses with the QA Manager.
- Compliance with ISO 17025.

4.2.9 Operations Manager

The Operations Manager manages and directs the analytical production sections of the laboratory. The lab employs and Organics Operations Manager and an Inorganic Operations Manager. They report directly to the Laboratory Director and act as co-Technical Managers for their areas of responsibility. He/She assists the Technical Manager in determining the most efficient instrument utilization. More specifically, he/she:

- Evaluates the level of internal/external non-conformances for all departments
- Continuously evaluates production capacity and improves capacity utilization.
- Continuously evaluates turnaround time and addresses any problems that may hinder meeting the required and committed turnaround time from the various departments.
- Develops and improves the training of all analysts in cooperation with the Technical Manager and QA Manager and in compliance with regulatory requirements.
- Works with the Preventive Maintenance Coordinator to ensure that scheduled instrument maintenance is completed.
- Is responsible for efficient utilization of supplies.
- Constantly monitors and modifies the processing of samples through the departments.
- Fully supports the quality system and, if called upon in the absence of the QA Manager, serves as his substitute in the interim.

4.2.10 Project Manager (PM)

Members of the laboratory Client Services/Project Management Group are responsible for organizing and managing client projects. Clients are assigned a project manager who serves as their primary contact at the laboratory. It is the PM’s responsibility to act as the client advocate by communicating client requirements to laboratory personnel and ensuring that clients provide complete information needed by the laboratory to meet those requirements – including all verbal communications.

- Scheduling sample submissions, sample container orders and sample pick-up via the laboratory courier service.
- Confirming certification status

- Coordinating and communicating turnaround time (TAT) requirements for high priority samples/projects.
- Answering common technical questions, facilitating problem resolution and coordinating technical details with the laboratory staff.
- Ensuring that client specifications, when known, are met by communicating project and quality assurance requirements to the laboratory.
- Notifying the supervisors of incoming projects and sample delivery schedules.
- Accountable to clients for communicating sample status reports or results prior to receipt of the final report.
- Monitor the status of all data package projects in-house to ensure timely and accurate delivery of reports.
- Inform clients of data package-related problems and resolve service issues.

4.2.11 Department Supervisors

Department Supervisors (aka Department Managers) report to their respective Operations Managers (Organic or Inorganic). Department Supervisors in conjunction with their Operations Manager serve as Technical Managers (see Section 4.2.8 above) for their department. Each one is responsible to:

- Ensure that analysts in their department adhere to applicable SOPs and the QA Manual. They perform frequent SOP and QA Manual review to determine if analysts are in compliance and if new, modified, and optimized measures are feasible and should be added to these documents.
- With regard to analysts, participates in the selection, training (as documented in Section 5.3), development of performance objectives and standards of performance, appraisal (measurement of objectives), scheduling, counseling, discipline, and motivation of analysts and documents these activities in accordance with systems developed by the QA and Personnel Departments. They evaluate staffing sufficiency and overtime needs. Training consists of familiarization with SOP, QC, Safety, and computer systems.
- Encourage the development of analysts to become cross-trained in various methods and/or operate multiple instruments efficiently while performing maintenance and documentation, self-supervise, and function as a department team.
- Provide guidance to analysts in resolving problems encountered daily during sample prep/analysis in conjunction with the Technical Manager, Operations Manager, and/or QA Manager. Each is responsible for 100% of the data review and documentation, non-conformance issues, the timely and accurate completion of performance evaluation samples and MDLs, for his department.
- Ensure all logbooks are maintained, current, and properly labeled or archived.
- Report all non-conformance conditions to the QA Manager, Technical Manager, Operations Manager, and/or BU / Laboratory Director.
- Ensure that preventive maintenance is performed on instrumentation as detailed in the QA Manual or SOPs. He is responsible for developing and implementing a system for preventive maintenance, troubleshooting, and repairing or arranging for repair of instruments.

- Maintain adequate and valid inventory of reagents, standards, spare parts, and other relevant resources required to perform daily analysis.
- Achieve optimum turnaround time on analyses and compliance with holding times.
- Conduct efficiency and cost control evaluations on an ongoing basis to determine optimization of labor, supplies, overtime, first-run yield, capacity (designed vs. demonstrated), second- and third-generation production techniques/instruments, and long-term needs for budgetary planning.
- Develop, implement, and enhance calibration programs.
- Provide written responses to external and internal audit issues.

4.2.12 Laboratory Analysts

Laboratory analysts are responsible for conducting analysis and performing all tasks assigned to them by the group leader or supervisor. The responsibilities of the analysts are listed below:

- Perform analyses by adhering to analytical and quality control protocols prescribed by current SOPs, this QA Manual, and project-specific plans honestly, accurately, timely, safely, and in the most cost-effective manner.
- Document standard and sample preparation, instrument calibration and maintenance, data calculations, sample matrix effects, and any observed non-conformance on worklists, benchsheets, lab notebooks and/or the Non-Conformance Database.
- Report all non-conformance situations, instrument problems, matrix problems and QC failures, which might affect the reliability of the data, to their supervisor, the Technical Manager, and/or the QA Manager or member of QA staff.
- Perform 100% review of the data generated prior to entering and submitting for secondary level review.
- Suggest method improvements to their supervisor, the Technical Manager, and the QA Manager. These improvements, if approved, will be incorporated. Ideas for the optimum performance of their assigned area, for example, through the proper cleaning and maintenance of the assigned instruments and equipment, are encouraged.
- Work cohesively as a team in their department to achieve the goals of accurate results, optimum turnaround time, cost effectiveness, cleanliness, complete documentation, and personal knowledge of environmental analysis.

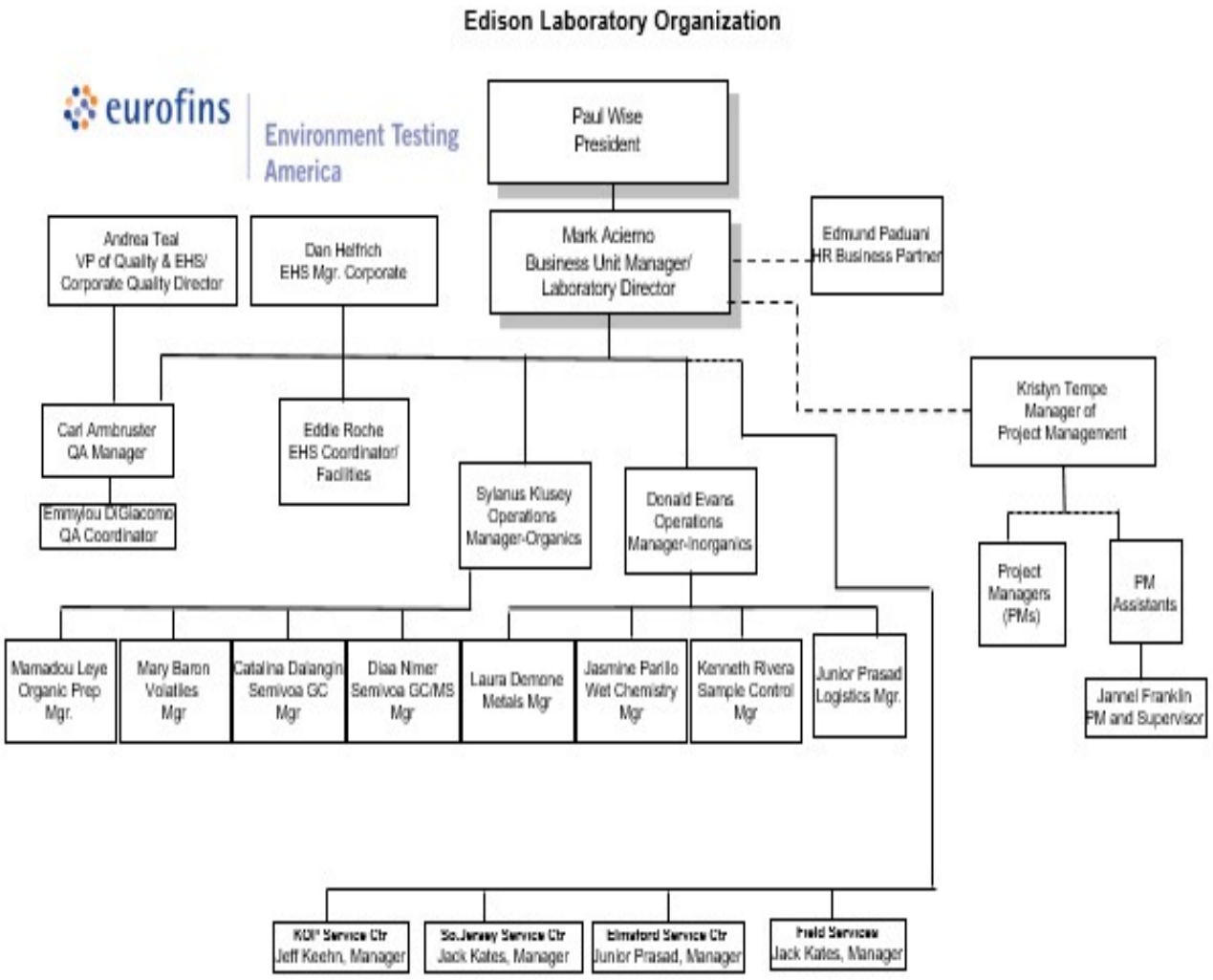
4.3 Business Continuity and Contingency Plans

Various policies and practices are in place to address continuity of business and contingency plans to ensure continued operations or minimal disruption in operations should unplanned events (natural disasters, unexpected management changes, etc.) occur. Deputies are identified for all key management personnel. Deputies would temporarily fill a role if the primary is absent for more than 15 consecutive calendar days. The deputies must meet the same qualifications as the primary person should they be required to take on the responsibilities. The QA Manager communicates to the relevant regulatory authorities when there are management or facility changes that impact the laboratory. Changes in the technical director must be communicated within a period of time and in the manner dictated by each regulatory authority.

The following table defines who assumes the responsibilities of key personnel in their absence:

Key Personnel	Deputy
Mark Acierno Laboratory Director	In the event of absence the Laboratory Director's responsibilities are shared by the Laboratory Operations Manager, the Quality Assurance Manager and the Client Services Manager, as appropriate
Carl Armbruster Quality Manager	Emmylou Digiacomo Quality Assurance Specialist Mark Acierno Laboratory Director
Sylvanus Klusey Organic Operations Manager	Donald Evans Inorganics Operations Manager
Donald Evans Inorganics Operations Manager	Sylvanus Klusey Organic Operations Manager
VOA, SVOA & Organic Prep Department/Technical Managers	Sylvanus Klusey Organic Technical Manager
Wet Chemistry, Metals Department/ Technical Managers	Donald Evans Inorganics Operations Manager
Dan Helfrich EH&S Manager	Edward Roche EH&S Coordinator
Kristyn Tempe Manager of Project Managers	Mark Acierno Laboratory Director
John Kates South Jersey/King of Prussia Service Center Manager	Alonzo Hall, Sample Control Tech. South Jersey Service Center Jeffery Keehn, Sample Control Tech. King of Prussia Service Center
John Kates Fields Services Manager	Stephen Schulze Thomas Lesinski Field Specialists – Edison
Junior Prasad Logistics Manager/Elmsford Service Center Manager	Donald Evans Inorganics Operations Manager

Figure 4-1. Laboratory Organization Chart



5.0 PERSONNEL

5.1 Overview

The laboratory's management believes that its highly qualified and professional staff is the single most important aspect in assuring a high level of data quality and service. The staff consists of professionals and support personnel.

All personnel must demonstrate competence in the areas where they have responsibility. Any staff that is undergoing training shall have appropriate supervision until they have demonstrated their ability to perform their job function on their own. Staff shall be qualified for their tasks based on appropriate education, training, experience and/or demonstrated skills as required.

The laboratory employs sufficient personnel with the necessary education, training, technical knowledge and experience for their assigned responsibilities.

All personnel are responsible for complying with all QA/QC requirements that pertain to the laboratory and their area of responsibility. Each staff member must have a combination of experience and education to adequately demonstrate a specific knowledge of their particular area of responsibility. Technical staff must also have a general knowledge of lab operations, test methods, QA/QC procedures and records management.

Laboratory management is responsible for formulating goals for lab staff with respect to education, training and skills and ensuring that the laboratory has a policy and procedures for identifying training needs and providing training of personnel. The training shall be relevant to the present and anticipated responsibilities of the lab staff.

The laboratory only uses personnel that are employed by or under contract to, the laboratory. Contracted personnel, when used, must meet competency standards of the laboratory and work in accordance to the laboratory's quality system.

5.2 Education and Experience Requirements for Technical Personnel

The laboratory makes every effort to hire analytical staffs that possess a college degree (AA, BA, BS) in an applied science with some chemistry in the curriculum. Exceptions can be made based upon the individual's experience and ability to learn. Selection of qualified candidates for laboratory employment begins with documentation of minimum education, training, and experience prerequisites needed to perform the prescribed task. Minimum education and training requirements for laboratory employees are outlined in job descriptions maintained by Eurofins Environment Testing America Human Resources.

Experience and specialized training are occasionally accepted in lieu of a college degree (basic lab skills such as using a balance, colony counting, aseptic or quantitation techniques, etc., are also considered).

As a general rule for analytical staff:

Specialty	Education	Experience
Extractions, Digestions, some electrode methods (pH, DO, Redox, etc.), or Titrimetric and Gravimetric Analyses	H.S. Diploma	On the job training (OJT)

Specialty	Education	Experience
GFAA, CVAA, FLAA, Single component or short list Chromatography (e.g., Fuels, BTEX-GC, IC	A college degree in an applied science or 2 years of college and at least 1 year of college chemistry	Or 2 years prior analytical experience is required
ICP, ICPMS, Long List or complex chromatography (e.g., Pesticides, PCB, Herbicides, HPLC, etc.), GCMS	A college degree in an applied science or 2 years of college chemistry	or 5 years of prior analytical experience
Spectra Interpretation	A college degree in an applied science or 2 years of college chemistry	And 2 years relevant experience Or 5 years of prior analytical experience
Technical Managers – **General**	Bachelor's Degree in an applied science or engineering with 24 semester hours in chemistry An advanced (MS, PhD.) degree may substitute for one year of experience	And 2 years experience in environmental analysis of representative analytes for which they will oversee
Technical Managers – **Wet Chem** only (no advanced instrumentation)	Associates degree in an applied science or engineering or 2 years of college with 16 semester hours in chemistry	And 2 years relevant experience

When an analyst does not meet these requirements, they can perform a task under the direct supervision of a qualified analyst, peer reviewer or Technical Manager, and are considered an analyst in training. The person supervising an analyst in training is accountable for the quality of the analytical data and must review and approve data and associated corrective actions.

5.3 Training

The laboratory is committed to furthering the professional and technical development of employees at all levels.

Orientation to the laboratory's policies and procedures, in-house method training, and employee attendance at outside training courses and conferences all contribute toward employee proficiency. Below are examples of various areas of required employee training:

Required Training	Time Frame	Employee Type
Environmental Health & Safety	Prior to lab work	All
Ethics Statement – New Hires and Annually	1 week of hire & annually	All

Required Training	Time Frame	Employee Type
Ethics – New Hires and Annually	60 days of hire and annually	All
Data Integrity – New Hires and Annually	60 days of hire and annually	Technical and PMs
Initial Demonstration of Capability (DOC)	Prior to unsupervised method performance	Technical

The laboratory maintains records of relevant authorization/competence, education, professional qualifications, training, skills and experience of technical personnel (including contracted personnel) as well as the date that approval/authorization was given. These records are kept on file at the laboratory. Also refer to "Demonstration of Capability" in Section 19.

The training of technical staff is kept up to date by:

- Each employee must have documentation in their training file that they have read, understood and agreed to follow the most recent version of the laboratory QA Manual and SOPs in their area of responsibility. This documentation is updated as SOPs are updated.
- Documentation from any training courses or workshops on specific equipment, analytical techniques or other relevant topics.
- Documentation of proficiency (refer to Section 19).
- An Ethics Agreement signed by each staff member (renewed each year) and evidence of annual ethics training.
- A Confidentiality Agreement signed by each staff member signed at the time of employment.
- Human Resources maintains documentation and attestation forms on employment status and records; benefit programs; timekeeping/payroll; and employee conduct (e.g., ethics violations). This information is maintained in the employee's secured personnel file.

Evidence of successful training could include such items as:

- Adequate documentation of training within operational areas, including one-on-one technical training for individual technologies, and particularly for people cross-trained.
- Analysts knowledge to refer to QA Manual for quality issues.
- Analysts following SOPs, i.e., practice matches SOPs.
- Analysts regularly communicate to supervisors and QA if SOPs need revision, rather than waiting for auditors to find problems.

Further details of the laboratory's training program are described in the Laboratory Training SOP ED-GEN-022, *Training*.

5.4 Data Integrity and Ethics Training Program

The laboratory's Ethics and Data Integrity Program is discussed in Section 6.2. Employees are trained as to the legal and environmental repercussions that result from data misrepresentation. Key topics covered in the presentation include:

- Organizational mission and its relationship to the critical need for honesty and full disclosure in all analytical reporting.

- Ethics Policy
- How and when to report ethical/data integrity issues. Confidential reporting.
- Record keeping.
- Discussion regarding data integrity procedures.
- Specific examples of breaches of ethical behavior (e.g. peak shaving, altering data or computer clocks, improper macros, etc., accepting/offering kickbacks, illegal accounting practices, unfair competition/collusion)
- Internal monitoring. Investigations and data recalls.
- Consequences for infractions including potential for immediate termination, debarment, or criminal prosecution.
- Importance of proper written narration / data qualification by the analyst and project manager with respect to those cases where the data may still be usable but are in one sense or another partially deficient.

Additionally, a data integrity hotline (855-910-0005) is maintained by the NDSC.

6.0 ACCOMMODATIONS AND ENVIRONMENTAL CONDITIONS

6.1 Overview

The laboratory is a 42,000 ft^2 secure laboratory facility with controlled access and designed to accommodate an efficient workflow and to provide a safe and comfortable work environment for employees. All visitors sign in and are escorted by laboratory personnel. Access is controlled by various measures.

The laboratory is equipped with structural safety features. Each employee is familiar with the location, use, and capabilities of general and specialized safety features associated with their workplace. The laboratory provides and requires the use of protective equipment including safety glasses, protective clothing, gloves, etc., OSHA and other regulatory agency guidelines regarding required amounts of bench and fume hood space, lighting, ventilation (temperature and humidity controlled), access, and safety equipment are met or exceeded.

Traffic flow through sample preparation and analysis areas is minimized to reduce the likelihood of contamination. Adequate floor space and bench top area is provided to allow unencumbered sample preparation and analysis space. Sufficient space is also provided for storage of reagents and media, glassware, and portable equipment. Ample space is also provided for refrigerated sample storage before analysis and archival storage of samples after analysis. Laboratory HVAC and deionized water systems are designed to minimize potential trace contaminants.

The laboratory is separated into specific areas for sample receiving, sample preparation, volatile organic sample analysis, non-volatile organic sample analysis, inorganic sample analysis, and administrative functions.

6.2 Environment

Laboratory accommodation, test areas, energy sources, and lighting are adequate to facilitate proper performance of tests. The facility is equipped with heating, ventilation, and air conditioning (HVAC) systems appropriate to the needs of environmental testing performed at this laboratory.

The environment in which these activities are undertaken does not invalidate the results or adversely affect the required accuracy of any measurements.

The laboratory provides for the effective monitoring, control and recording of environmental conditions that may affect the results of environmental tests as required by the relevant specifications, methods, and procedures.

When any of the method or regulatory required environmental conditions change to a point where they may adversely affect test results, analytical testing will be discontinued until the environmental conditions are returned to the required levels.

Environmental conditions of the facility housing the computer network and LIMS are regulated to protect against raw data loss.

When the laboratory performs laboratory activities at sites or facilities outside its permanent control, it shall ensure that the requirements related to facilities and environmental conditions of this document are met.

Specific requirements for facility and environmental conditions, as well as periodic monitoring of conditions, are given in the Environmental Health & Safety Manual plus each laboratory's Facility Addendum.

6.3 Work Areas

There is effective separation between neighboring areas when the activities therein are incompatible with each other. Examples include:

- Volatile organic chemical handling areas, including sample preparation and waste disposal, and volatile organic chemical analysis areas.

Access to and use of all areas affecting the quality of analytical testing is defined and controlled by secure access to the laboratory building as described below in the Building Security section.

Adequate measures are taken to ensure good housekeeping in the laboratory and to ensure that any contamination does not adversely affect data quality. These measures include regular cleaning to control dirt and dust within the laboratory. Work areas are available to ensure an unencumbered work area. Work areas include:

- Access and entryways to the laboratory.
- Sample receipt areas.
- Sample storage areas.
- Chemical and waste storage areas.
- Data handling and storage areas.
- Sample processing areas.
- Sample analysis areas.

6.4 Responding to Emergencies

Employees must be aware of procedures to respond to all emergencies that might occur in the workplace. Employees must be familiar with the location and proper operation of all emergency equipment, evacuation routes and designated assembly areas for all areas where they work. Refer to the NDSC EH&S Manual Document No. CW-E-M-001. Sec. 7 and the laboratory's local EH&S addendum for complete details. These documents provide direction for situations where

normal operations of the laboratory are not possible (e.g., electrical failures, heating/air conditioning failures, fire/building evacuation, computer failures, hazardous material spills, injury to employees, pandemic flu, disruption of phone service, etc.)

In the event that the building or information technology (IT) systems would be severely challenged, a designated disaster recovery team, which includes Facility Management, Maintenance, Safety, Laboratory/Executive Management, Public Relations, IT, QA and other applicable personnel depending on the scope of the disaster, would assemble at a designated area to assess the situation and formulate a plan.

6.5 Building Security

The laboratory is considered a secure facility. All outside doors (except the main lobby entrance) are locked during normal business hours to prevent unauthorized entry. (An attendant monitors this entrance at all times.) Building keys and alarm codes are distributed to employees as necessary.

All visitors to the laboratory must sign in and out in a visitor's logbook that is located in the lobby. A visitor is defined as any person who visits the laboratory who is not an employee of the laboratory. Both visitors and vendors must review and sign specific EH&S forms; and are escorted by laboratory personnel at all times, or the location of the visitor is noted in the visitor's logbook.

Signs are posted in the laboratory designating employee only areas - "Authorized employees beyond this point".

7.0 QUALITY SYSTEM

7.1 Quality Policy Statement

The Quality Policy statement gives employees clear requirements for the production of analytical data. As an organization, all personnel are committed to high quality professional practice, testing and data, and service to our clients.

We strive to provide the highest quality data achievable by:

- Reading and understanding all of the quality documents applicable to each position and implementing the process in our work.
- Following all recordkeeping requirements; describing clearly and accurately all activities performed; recording "real time" as the task is carried out; understanding that it is never acceptable to "back date" entries and should additional information be required at a later date, the actual date and by whom the notation is made must be documented.
- Ensuring data integrity through the completeness, consistency, impartiality and accuracy of the data generated. Data is attributable, legible, contemporaneously recorded, original or a true copy, and accurate (ALCOA). This applies to manual paper documentation and electronic records.
- Providing accountability and traceability for each sample analyzed through proper sample handling, labeling, preparation, instrument calibration/qualification/validation, analysis, and reporting; establishing an audit trail (the who, what, when, and why) that identifies date, time, analyst, instrument used, instrument conditions, quality control samples (where appropriate and/or required by the method), and associated standard material.

- Emphasizing a total quality management process which provides impartiality, accuracy, and strict compliance with agency regulations and client requirements, giving the highest degree of confidence; understanding that meeting the requirements of the next employee in the work flow process is just as important as meeting the needs of the external client.
- Providing thorough documentation and explanation to qualify reported data that may not meet all requirements and specifications, but is still of use to the client; understanding this occurs only after discussion with the client on the data limitations and acceptability of this approach.
- Responding immediately to indications of questionable data, out-of-specification occurrences, equipment malfunctions, and other types of laboratory problems, with investigation and applicable corrective action; documenting these activities completely, including the reasons for the decisions made.
- Providing a work environment that ensures accessibility to all levels of management and encourages questions and expression of concerns on quality issues to management. Eurofins recognizes that the implementation of a quality assurance program requires management's commitment and support as well as the involvement of the entire staff
- Continually improve systems and manage risk to support quality improvement efforts in laboratory, administrative and managerial activities

7.2 Ethics and Data Integrity

Eurofins Environment Testing America is committed to ensuring the integrity of its data and meeting the quality needs of its clients. The laboratory operates our Ethics and Data Integrity program under the guidance of Eurofin's Key Guidance Document (KGD). The elements of our program include:

- An Ethics Policy (NDSC Document No. CW-L-P-004) and Employee Ethics Statements.
- Ethics and Compliance Officers (ECOs).
- A Training Program.
- Self-governance through disciplinary action for violations.
- A confidential mechanism for anonymously reporting alleged misconduct and a means for conducting internal investigations of all alleged misconduct. (NDSC Document No. CW-L-S-002).
- Procedures and guidance for recalling data if necessary (NDSC Document No. CW-Q-S-005).
- Effective external and internal monitoring system that includes procedures for internal audits (Section 17).
- Produce results, which are accurate and include QA/QC information that meets client pre-defined Data Quality Objectives (DQOs).
- Present services in a confidential, honest and forthright manner.
- Provide employees with guidelines and an understanding of the Ethical and Quality Standards of our Industry.
- Provide procedures and guidance to ensure the impartiality and confidentiality of all data and customer information

- Operate our facilities in a manner that protects the environment and the health and safety of employees and the public.
- Obey all pertinent federal, state and local laws and regulations and encourage other members of our industry to do the same.
- Educate clients as to the extent and kinds of services available.
- Assert competency only for work for which adequate personnel and equipment are available and for which adequate preparation has been made.
- Promote the status of environmental laboratories, their employees, and the value of services rendered by them.

7.3 Quality System Documentation

The laboratory's Quality System is communicated through a variety of documents.

- Quality Assurance (QA) Manual – Each laboratory has a lab-specific QA manual.
- NDSC Official Documents – Each laboratory may use the Guidance (instructional use) documents at their discretion. Template documents are process documents that the laboratory's need to implement locally by using the document as is or as an outline to define their internal practices that meet the minimum requirements of the template. Required documents need to be implemented as is and listed in the laboratory's document control list.
- Key Guidance Documents (KGDs) - Documents compiled at the Group Service Centre (GSC) level by Functional Leaders (document owners) aimed at providing specific Eurofins groups of employees with guidelines necessary for the good conduct of their respective work.
- Laboratory SOPs and Policies – General and Technical
- Work Instructions – A subset of procedural steps, tasks or forms associated with an operation of a management system (e.g., checklists, preformatted bench sheets, forms).

7.3.1 Order of Precedence

In the event of a conflict or discrepancy between policies, the order of precedence is as follows:

- Quality Management Plan (QMP)
- NDSC Guidance Documents
- KGDs
- Laboratory Quality Assurance Manual (QA Manual)
- Laboratory SOPs and Policies
- Other (Work Instructions (WI), memos, flow charts, etc.)

NOTE: The laboratory has the responsibility and authority to operate in compliance with regulatory requirements of the jurisdiction in which the work is performed. Where the QMP conflicts with those regulatory requirements, the regulatory requirements of the jurisdiction shall hold primacy. The laboratory's QA Manual shall take precedence over the QMP in those cases.

7.4 QA/QC Objectives for the Measurement of Data

Quality Assurance (QA) is responsible for developing planned activities whose purpose is to provide assurance to all levels of management that a quality program is in place within the laboratory, and that it is functioning in an effective manner that is consistent with the requirements of NELAP, ISO 17025, and any other regulatory agencies (i.e., states) in which the laboratory maintains accreditation.

Quality Control (QC) is generally understood to be limited to the analyses of samples and to be synonymous with the term *"analytical quality control"*. QC refers to the routine application of statistically based procedures to evaluate and control the accuracy of results from analytical measurements. The QC program includes procedures for estimating and controlling precision and bias and for determining reporting limits.

Request for Proposals (RFPs) and Quality Assurance Project Plans (QAPP) provide a mechanism for the client and the laboratory to discuss the data quality objectives in order to ensure that analytical services closely correspond to client needs. In order to ensure the ability of the laboratory to meet the Data Quality Objectives (DQOs) specified in the QAPP, clients are advised to allow time for the laboratory to review the QAPP before being finalized. The client is responsible for developing the QAPP; however, the laboratory will provide support to the client for developing the sections of the QAPP that concern laboratory activities.

Historically, laboratories have described their QC objectives in terms of precision, accuracy, representativeness, comparability, completeness, selectivity and sensitivity (PARCCSS). Each laboratory SOP defines the required QC indicators.

7.4.1 Precision

The objective is to meet the performance for precision demonstrated for the methods on similar samples and to meet DQOs of the EPA and/or other regulatory programs. Precision is defined as the degree of reproducibility of measurements under a given set of analytical conditions (exclusive of field sampling variability). Precision is documented on the basis of replicate analysis, usually duplicate or matrix spike (MS) duplicate samples.

7.4.2 Accuracy

The objective is to meet the performance for accuracy demonstrated for The objective is to meet the performance for accuracy demonstrated for the methods on similar samples and to meet DQOs of the EPA and/or other regulatory programs. Accuracy is defined as the degree of bias in a measurement system. Accuracy may be documented through the use of laboratory control samples (LCS) and/or MS. A statement of accuracy is expressed as an interval of acceptance recovery about the mean recovery.

7.4.3 Representativeness

The objective is to provide data which is representative of the sampled medium. Representativeness is defined as the degree to which data represent a characteristic of a population or set of samples and is a measurement of both analytical and field sampling precision. The representativeness of the analytical data is a function of the procedures used in procuring and processing the samples. The representativeness can be documented by the relative percent difference between separately procured, but otherwise identical samples or sample aliquots.

The representativeness of the data from the sampling sites depends on both the sampling procedures and the analytical procedures. Refer to laboratory SOPs for subsampling and homogenization techniques appropriate to the analytical method.

7.4.4 Comparability

The objective is to provide analytical data for which the accuracy, precision, representativeness, and reporting limit statistics are similar to these quality indicators generated by other laboratories for similar samples, and data generated by the laboratory over time.

Comparability is documented by inter-laboratory studies carried out by regulatory agencies or carried out for specific projects or contracts, by comparison of periodically generated statements of accuracy, precision, and reporting limits with those of other laboratories.

7.4.5 Completeness

The completeness objective for data is 90% (or as specified by a particular project), expressed as the ratio of the valid data to the total data over the course of the project. Data will be considered valid if they are adequate for their intended use. Data usability will be defined in a QAPP, project scope, or regulatory requirement. Data validation is the process for reviewing data to determine its usability and completeness. If the completeness objective is not met, actions will be taken internally and with the data user to improve performance. This may take the form of an audit to evaluate the methodology and procedures as possible sources for the difficulty or may result in a recommendation to use a different method.

7.4.6 Selectivity

Selectivity is defined as the capability of a test method or instrument to respond to a target substance or constituent in the presence of non-target substances. Target analytes are separated from non-target constituents and subsequently identified/detected through one or more of the following, depending on the analytical method: extractions (separation), digestions (separation), interelement corrections (separation), use of matrix modifiers (separation), specific retention times (separation and identification), confirmations with different columns or detectors (separation and identification), specific wavelengths (identification), specific mass spectra (identification), and specific electrodes (separation and identification).

7.4.7 Sensitivity

Sensitivity refers to the amount of analyte necessary to produce a detector response that can be reliably detected (above the Method Detection Limit) or quantified (above the Reporting Limit).

7.5 Criteria for Quality Indicators

The laboratory maintains tables, housed in LIMS, that summarize the precision and accuracy acceptability limits for performed analyses. This summary includes an effective date, is updated each time new limits are generated, and are managed by the laboratory's QA department. Unless otherwise noted, limits within these tables are laboratory generated. Some acceptability limits are derived from US EPA methods when they are required. Where US EPA method limits are not required, the laboratory has developed limits from evaluation of data from similar matrices. Criteria for development of control limits is contained in lab SOP ED-GEN-026, Evaluation of Analytical Accuracy and Precision Through The Use of Control Charts.

7.6 Statistical Quality Control

Statistically-derived precision and accuracy limits are required by selected methods (such as SW-846) and programs.. The laboratory routinely utilizes statistically-derived limits to evaluate method performance and determine when corrective action is appropriate. The analysts use the current limits entered into LIMS. The QA department maintains an archive of all limits used within the laboratory. If a method defines the QC limits, the method limits are used.

If a method requires the generation of historical limits, the lab develops such limits from recent data in the QC database of the LIMS following the guidelines described in lab SOP ED-GEN-026, Evaluation of Analytical Accuracy and Precision Through The Use of Control Charts and Section 26. All calculations and limits are documented and dated when approved and effective. On occasion, a client requests contract-specified limits for a specific project.

Current QC limits are entered and maintained in the LIMS analyte database. As sample results and the related QC are entered into LIMS, the sample QC values are compared with the limits in LIMS to determine if they are within the acceptable range. The analyst then evaluates if the sample needs to be rerun or re-extracted/rerun or if a comment should be added to the report explaining the reason for the QC outlier.

7.6.1 QC Charts

The QA Manager may evaluate QC control charts as needed to determine if adjustments need to be made or for corrective actions to methods. All findings are documented and kept on file.

7.7 Quality System Metrics

In addition to the QC parameters discussed above, the entire Quality System is evaluated on a monthly basis through the use of specific metrics (refer to Section 18). These metrics are used to drive continuous improvement in the laboratory's Quality System.

8.0 DOCUMENT CONTROL

8.1 Overview

The QA Department is responsible for the control of documents used in the laboratory to ensure that approved, up-to-date documents are in circulation and out-of-date (obsolete) documents are archived or destroyed. The following documents, at a minimum, must be controlled:

- Laboratory Quality Assurance (QA) Manual
- Laboratory Standard Operating Procedures (SOP)
- Laboratory Policies
- Work Instructions and Forms
- NDSC Documents[1]
- KGDs[1]

[1]Includes locally implemented documents that are document controlled within the laboratory's document control system. The NDSC and/or KGD documents are only considered controlled when they are read on the intranet site. Printed copies are considered uncontrolled unless the laboratory physically distributes them as controlled documents. A detailed description of the procedure for issuing, authorizing, controlling, distributing, and archiving NDSC Official

Documents is found in Document No. CW-Q-S-001, NDSC Document Control and Archiving. KGD documents are controlled according to this document. The laboratory's internal document control procedure is defined in SOP No. ED-GEN-002, Document Control.

The laboratory QA Department also maintains access to various references and document sources integral to the operation of the laboratory. This includes reference methods and regulations. Instrument manuals (hard or electronic copies) are also maintained by the laboratory.

The laboratory maintains control of records for raw analytical data and supporting records such as audit reports and responses, logbooks, standard logs, training files, MDL studies, Proficiency Testing (PT) studies, certifications and related correspondence, and corrective action reports. Raw analytical data consists of bound logbooks, instrument printouts, any other notes, magnetic media, electronic data, and final reports.

8.2 Document Approval and Issue

The pertinent elements of a document control system includes a unique document title and number, pagination, the total number of pages of the item or an 'end of document' page, the effective date, revision number, and the laboratory's name. The QA personnel are responsible for the maintenance of this system.

Controlled documents are authorized by the QA Department. In order to develop a new document, a responsible manager submits an electronic draft to the QA Department for suggestions and approval before use. Upon approval, QA personnel add the identifying version information to the document and retains that document as the official document on file. That document is then provided to all applicable operational units. Controlled documents are identified as such and records of their distribution are kept by the QA Department. Document control may be achieved by either electronic or hardcopy distribution.

The QA Department maintains a list of the official versions of controlled documents.

Quality System Policies and Procedures will be reviewed at a minimum of every two years and revised as appropriate. Changes to documents occur when a procedural change warrants.

8.3 Procedures for Document Control Policy

For changes to the QA Manual, SOPS and Work Instructions refer to SOP No. ED-GEN-002, Document Control. Uncontrolled copies must not be used within the laboratory. Previous revisions and back-up data are stored by the QA department. Electronic copies are stored on the Public server in the QA folder for the applicable revision.

8.4 Obsolete Documents

All invalid or obsolete documents are removed, or otherwise prevented from unintended use. The laboratory has specific procedures as described above to accomplish this. In general, obsolete documents are collected from employees according to distribution lists and are marked obsolete on the cover or destroyed. At least one copy of the obsolete document is archived according to SOP No. ED-GEN-002, Document Control.

9.0 SERVICE TO THE CLIENT

9.1 Overview

The laboratory has established procedures for the review of work requests and contracts, oral or written. The procedures include evaluation of the laboratory's capability and resources to meet the contract's requirements within the requested time period. All requirements, including the methods to be used, must be adequately defined, documented and understood. For many environmental sampling and analysis programs, testing design is site or program specific and does not necessarily fit into a standard laboratory service or product. It is the laboratory's intent to provide both standard and customized environmental laboratory services to our clients.

A thorough review of technical and QC requirements contained in contracts is performed to ensure project success. The appropriateness of requested methods, and the lab's capability to perform them must be established. Projects, proposals, and contracts are reviewed for adequately defined requirements and the laboratory's capability to meet those requirements. Alternate test methods that are capable of meeting the clients' requirements may be proposed by the lab. A review of the lab's capability to analyze non-routine analytes is also part of this review process.

All projects, proposals and contracts are reviewed for the client's requirements in terms of compound lists, test methodology requested, sensitivity (detection and reporting levels), accuracy, and precision requirements (% Recovery and RPD). The reviewer ensures that the laboratory's test methods are suitable to achieve these requirements and that the laboratory holds the appropriate certifications and approvals to perform the work. The laboratory and any potential subcontract laboratories must be certified, as required, for all proposed tests.

Electronic or hard copy deliverable requirements are evaluated against the laboratory's capacity for production of the documentation.

If the laboratory cannot provide all services but intends to subcontract such services, whether to another Eurofins facility on the same LIMS or to an outside firm, this will be documented and discussed with the client prior to contract approval. (Refer to Section 10 for Subcontracting Procedures.)

The laboratory informs the client of the results of the review if it indicates any potential conflict, deficiency, lack of accreditation, or inability of the lab to complete the work satisfactorily. Any discrepancy between the client's requirements and the laboratory's capability to meet those requirements is resolved in writing before acceptance of the contract. It is necessary that the contract be acceptable to both the laboratory and the client. Amendments initiated by the client and/or Eurofins Edison are documented in writing.

All contracts, QAPPs, Sampling and Analysis Plans (SAPs), contract amendments, and documented communications become part of the project record.

The same contract review process used for the initial review is repeated when there are amendments to the original contract by the client, and the participating personnel are informed of the changes.

9.2 Review Sequence and Key Personnel

Appropriate personnel will review the work request at each stage of evaluation.

For routine projects and other simple tasks, a review by the Project Manager (PM) is considered adequate. The PM confirms that the laboratory has any required certifications, that it can meet

the clients' data quality and reporting requirements and that the lab has the capacity to meet the clients turn around needs. It is recommended that, where there is a sales person assigned to the account, an attempt should be made to contact that sales person to inform them of the incoming samples.

For new, complex or large projects, the proposed contract is given to the Client Relationship Manager or Proposal Team, who will decide which lab will receive the work based on the scope of work and other requirements, including certification, testing methodology, and available capacity to perform the work. The contract review process is outlined in NDSC Document No. CA-L-P-002**,** Contract Compliance Policy.

This review encompasses all facets of the operation. The scope of work is distributed to the appropriate personnel, as needed based on scope of contract, to evaluate all of the requirements shown above (not necessarily in the order below):

- Contract Administrator
- Laboratory Project Manager
- Laboratory Directors and/or Technical Managers
- Account Executives
- Quality Managers
- Laboratory Environmental Health and Safety Managers/Directors

The Laboratory Director reviews the formal laboratory quote and makes final acceptance for their facility.

The Sales Director, Contract Administrator, Account Executive or Proposal Coordinator then submits the final proposal to the client.

In the event that one of the above personnel is not available to review the contract, his or her back-up will fulfill the review requirements.

9.3 Balancing Laboratory Capacity and Workload

Evaluating laboratory capacity to perform specific projects is the responsibility of the Vice-President, Laboratory Directors and Managers, and the Client Services director and manager. Many analysts are cross-trained to perform a variety of tests, and there is redundant equipment available in case of malfunctions. This minimizes the need to evaluate small and medium size projects against capacity available to complete them. Large and complex projects are reviewed against capacity estimates before bids are submitted to ensure that the client's analysis schedule is met. Regularly scheduled meetings are held between laboratory management, PMs, Client Services and QA personnel to review progress with current projects, as well as special requirements of new work scheduled for the laboratory. Laboratory capacity and backlog is tracked on a continuous basis using information from the Laboratory Sample Information System (LIMS) including turnaround time, and work in-house..

9.4 Documentation

The Contracts Department maintains copies of all signed contracts**.** The Manager of Project Management maintains a copy locally.

Appropriate records are maintained for every contract or work request. All stages of the contract review process are documented and include records of any significant changes. This documentation is maintained by the laboratory PM on the lab's network server.

The contract will be distributed to and maintained by the appropriate sales/marketing personnel and the Account Executive. A copy of the contract and formal quote will be filed with the laboratory PM and the Laboratory Director.

Records are maintained of pertinent discussions with a client relating to the client's requirements or the results of the work during the period of execution of the contract. The PM keeps a phone log of conversations with the client and a copy of all related email correspondence.

9.4.1 Project-Specific Quality Planning

Communication of contract specific technical and QC criteria is an essential activity in ensuring the success of site specific testing programs. To achieve this goal, a PM is assigned to each client. It is the PM's responsibility to ensure that project-specific technical and QC requirements are effectively evaluated and communicated to the laboratory personnel before and during the project. QA department involvement may be needed to assist in the evaluation of custom QC requirements.

PM's are the primary client contact and they ensure resources are available to meet project requirements, They coordinate opportunities and work with laboratory management and supervisory staff to ensure available resources are sufficient to perform work for the client's project.

Prior to work on a new project, the dissemination of project information and/or project opening meetings may occur to discuss schedules and unique aspects of the project. Items to be discussed may include the project technical profile, turnaround times, holding times, methods, analyte lists, reporting limits, deliverables, sample hazards, or other special requirements. The PM introduces new project information to maximize production and client satisfaction, while maintaining quality. Project notes may be associated with each sample batch as a reminder upon sample receipt and analytical processing.

Any change that may occur within an active project is agreed upon between the client/regulatory agency and the PM/laboratory. These changes (e.g., use of a non-standard method or modification of a method) and approvals must be documented prior to implementation. Documentation pertains to any document (e.g., letter, e-mail, variance, contract addendum), which has been signed by both parties.

Such changes are also communicated to the laboratory. The laboratory staff is introduced to the modified requirements via the PM or the individual laboratory Technical Manager. After the modification is implemented into the laboratory process, documentation of the modification is made in the case narrative of the data report(s).

The laboratory strongly encourages client visits to the laboratory and for formal/informal information sharing session with employees in order to effectively communicate ongoing client needs as well as project specific details for customized testing programs.

9.5 Special Services

The laboratory cooperates with clients and their representatives to monitor the laboratory's performance in relation to work performed for the client. It is the laboratory's goal to meet all

client requirements in addition to statutory and regulatory requirements. The laboratory has procedures to ensure confidentiality to clients (Section 17 and 25).

The laboratory's standard procedures for reporting data are described in Section 25. Special services are also available and provided upon request. These services include:

- Reasonable access for our clients or their representatives to the relevant areas of the laboratory for the witnessing of tests performed for the client.
- Assisting client-specified third party data validators as specified in the client's contract.
- Supplemental information pertaining to the analysis of their samples. Note: An additional charge may apply for additional data/information that was not requested prior to the time of sample analysis or previously agreed upon.

When the client requests a statement of conformity to a specification or standard based on the analysis performed by the laboratory (e.g., pass/fail, in-tolerance/out-of-tolerance), the decision rule shall be clearly defined. Unless inherent in the requested specification or standard, the decision rule selected shall be communicated to the client. Associated reporting requirements are addressed in Section 25.2.18.

9.6 Client Communication

PMs are the primary communication link to the clients. They shall inform their clients of any delays in project completion as well as any non-conformances in either sample receipt or sample analysis. Project management will maintain ongoing client communication throughout the entire client project.

Technical Managers are available to discuss any technical questions or concerns that the client may have.

9.7 Reporting

The laboratory works with our clients to produce any special communication reports required by the contract.

9.8 Client Feedback and Surveys

The laboratory assesses both positive and negative client feedback. The results are used to improve overall laboratory quality and client service. Eurofins Sales and Marketing teams periodically develop lab and client specific surveys to assess client satisfaction. Client satisfaction surveys are sent to the client via email with the final report deliverables for the client's job(s).

When a complaint is received, we determine, to the best of our ability, the extent of the issue and what data is in question. The person receiving the complaint documents this information and promptly forwards it to the appropriate management personnel where the work in question was performed. If a data reporting error is discovered, the final report and/or data must be regenerated with the correct value(s).

The PM is responsible for entering client concerns into an NCM in TALS LIMS. In some cases, an ICAT is initiated to address and document the situation. While an individual issue may not warrant a formal investigation, QA monitors these issues for potential trends and will issue an ICAT if a trend is evident.

10.0 SUBCONTRACTING OF TESTS

The laboratory may subcontract tests to other laboratories if the requested testing is not routinely performed in our laboratory. To a lesser extent, samples may need to be subcontracted to an overflow laboratory to ensure hold times and/or turn-around-times (TAT) are met.

Testing is only subcontracted with the client's knowledge and approval. The <define> must notify the client in writing when any of their requested analyses will be subcontracted to another lab. Client approval must be obtained in writing before samples are shipped.

Subcontract laboratories are selected based on their qualifications and accreditations. The subcontractor is requested to sign a Laboratory Analytical Services Subcontract. If projects require a specific agency certification (i.e. individual state agencies, NELAP, DoD, PALA, ISO 17025), only an appropriately accredited laboratory is used. The client may also have a list of laboratories to be used for subcontracting. In these cases, the evaluation of the subcontract laboratory is made by the client.

Data obtained from subcontract laboratories is clearly marked as such when reported by the laboratory. The data are submitted to the client in the format obtained from the subcontractor.

10.1 Overview

For the purpose of this quality manual, the phrase subcontract laboratory refers to a laboratory external to the Eurofins Environment Testing America laboratories. The phrase "work sharing" refers to internal transfers of samples between the Eurofins Environment Testing America laboratories. The term outsourcing refers to the act of subcontracting tests.

When contracting with our clients, the laboratory makes commitments regarding the services to be performed and the data quality for the results to be generated. When the need arises to outsource testing for our clients because project scope, changes in laboratory capabilities, capacity, or unforeseen circumstances, we must be assured that the subcontractors or work sharing laboratories understand the requirements and will meet the same commitments we have made to the client. Refer to the NDSC Documents on Subcontracting Procedures (CW-L-S-004) and the Work Sharing Process (CA-C-S-001).

When outsourcing analytical services, the laboratory will assure, to the extent necessary, that the subcontract or work sharing laboratory maintains a program consistent with the requirements of this document, the requirements specified in TNI/ISO 17025 and/or the client's Quality Assurance Project Plan (QAPP). All QC guidelines specific to the client's analytical program are transmitted to the subcontractor and agreed upon before sending the samples to the subcontract facility. Additionally, work requiring accreditation will be placed with an appropriately accredited laboratory. The laboratory performing the subcontracted work will be identified in the final report, as will non-TNI accredited work where required.

PMs or other responsible Client Service members, for the Export Lab (i.e., the Eurofins Eurofins Environment Testing America laboratory that transfers samples to another laboratory) are responsible for obtaining client approval prior to subcontracting any samples. The laboratory will advise the client of a subcontract arrangement in writing and when possible approval from the client shall be obtained and retained in the project folder. Standard Eurofins Environment Testing America Terms & Conditions include the flexibility to subcontract samples within the Eurofins Environment Testing America laboratories. Therefore, additional advance notification to

clients for intra-laboratory subcontracting is not necessary unless specifically required by a client contract.

Note: In addition to the client, some regulating agencies (e.g., USDA) or contracts require notification prior to placing such work**.**

10.2 Qualifying and Monitoring Subcontractors

Whenever a PM becomes aware of a client requirement or laboratory need where samples must be outsourced to another laboratory, the other laboratory(s) shall be selected based on the following:

- Subcontractors specified by the client - In these circumstances, the client assumes responsibility for the quality of the data generated from the use of a subcontractor.
- Subcontractors reviewed by Eurofins Environment Testing America – Firms which have been reviewed by the company and are known to meet standards for accreditations (e.g., State, TNI and DoD/DOE); technical specifications; legal and financial information.

A listing of vendors is available on the Eurofins Environment Testing America intranet site.

All Eurofins Environment Testing America laboratories are pre-qualified for work sharing provided they hold the appropriate accreditations and can adhere to the project/program requirements. Client approval is not necessary unless specifically required by the contract. In these cases, the client must provide acknowledgement that the samples can be sent to that facility (an e-mail is sufficient documentation or if acknowledgement is verbal, the date, time, and name of person providing acknowledgement must be documented). The originating laboratory is responsible for communicating all technical, quality, and deliverable requirements as well as other contract needs as detailed in NDSC Document No. CA-C-S-001, Work Sharing Process.

10.2.1 When the potential subcontract laboratory has not been previously approved, Account Executives or PMs may nominate a laboratory as a subcontractor based on need. The decision to nominate a laboratory must be approved by the Client Relations Manager (CRM). The CRM requests that the PM begin the process of approving the subcontract laboratory. Refer to the NDSC Document No. CW-L-S-004, Subcontracting Procedures.

Once the appropriate accreditation and legal information is received by the laboratory, it is evaluated for acceptability and forwarded to the NDSC Quality Information Manager (QIM) for review. After the NDSC QIM reviews the documents for completeness, the information is forwarded to the Finance Department for formal signature and contracting with the laboratory. The approved vendor will be added to the approved subcontractor list on the intranet site, and the finance group is concurrently notified.

The client will assume responsibility for the quality of the data generated from the use of a subcontractor they have requested the lab to use. The qualified subcontractors on the intranet site are known to meet minimal standards. Eurofins Environment Testing America does not certify laboratories. The subcontractors on our approved list can only be recommended to the extent that we would use them.

10.3 Oversight and Reporting

The status and performance of qualified subcontractors will be monitored by NDSC and includes an annual review process (ref.: NDSC Document No. SOP CW-L-S-004. Any problems identified will be brought to the attention of NDSC.

- Complaints shall be investigated. Documentation of the complaint, investigation, and corrective action will be maintained in the subcontractor's file on the intranet site. Complaints are posted using the Vendor Performance Report.
- Information shall be updated on the intranet when new information is received from the subcontracted laboratories.
- Subcontractors in good standing will be retained on the intranet listing. Client Service personnel will notify all Eurofins Environment Testing America laboratories, NDSC and Corporate Contracts if any laboratory requires removal from the intranet site. This notification will be posted on the intranet site and e-mailed to all Client Service Personnel, Laboratory Directors, QA Managers, and Sales Personnel.

Prior to initially sending samples to the subcontracted laboratory, the PM confirms their certification status to determine if it's current and scope-inclusive. The information is documented within the project records.

The laboratory's certifications can be viewed on the company's website: https://www.eurofinsus.com/environment-testing/

10.3.1 All subcontracted samples must be accompanied by a Eurofins Edison Chain of Custody (COC). A copy of the original COC sent by the client must be available in LIMS for all samples workshared within Eurofins Environment Testing America. Client COCs are only forwarded to external subcontractors when samples are shipped directly from the project site to the subcontractor lab. Under routine circumstances, client COCs are not provided to external subcontractors.

Through communication with the subcontracted laboratory, the PM monitors the status of the subcontracted analyses, facilitates successful execution of the work, and ensures the timeliness and completeness of the analytical report.

Non-TNI accredited work must be identified in the subcontractor's report as appropriate. If TNI accreditation is not required, the report does not need to include this information.

Reports submitted from subcontractor laboratories are not altered and are included in their original form in the final project report. This clearly identifies the data as being produced by a subcontractor facility. If subcontract laboratory data is incorporated into the laboratory's EDD (i.e., imported), the report must explicitly indicate which lab produced the data for which methods and samples.

Note: The results submitted by a Eurofins Environment Testing America work sharing laboratory may be transferred electronically and the results reported by the Eurofins Environment Testing America sharing lab are identified on the final report. The report must explicitly indicate which lab produced the data for which methods and samples. The final report must include a copy of the completed COC for all work sharing reports.

10.4 Contingency Planning

The full qualification of a subcontractor may be waived to meet emergency needs. This decision and justification must be documented in the project files, and the 'Purchase Order Terms And Conditions For Subcontracted Laboratory Services' must be sent with the samples and COC.

In the event this provision is utilized, the laboratory (e.g., PM) will be required to verify and document the applicable accreditations of the subcontractor. All other quality and accreditation

requirements will still be applicable, but the subcontractor need not have signed a subcontract agreement with Eurofins Environment Testing America at this time.

The use of any emergency subcontractor will require the PM to complete a New Vendor Add Form in order to process payment to the vendor and add them to LIMS. This form requires the user to define the subcontractor's category/s of testing and the reason for testing.

10.5 Use of NELAP and A2LA Logo

It is not laboratory policy to use these logos on any company letterhead, including analytical reports.

11.0 PURCHASING SERVICES AND SUPPLIES

11.1 Overview

Evaluation and selection of suppliers and vendors is performed, in part, on the basis of the quality of their products, their ability to meet the demand for their products on a continuous and short term basis, the overall quality of their services, their past history, and competitive pricing. This is achieved through evaluation of objective evidence of quality furnished by the supplier, which can include certificates of analysis, recommendations, and proof of historical compliance with similar programs for other clients. To ensure that quality critical consumables and equipment conform to specified requirements, which may affect quality, all purchases from specific vendors are approved by a member of the supervisory or management staff. Capital expenditures are made in accordance with NDSC Document No. CW-F-S-007, Eurofins Environment Testing America Fixed Asset Acquisition, Retention and Safeguarding

Contracts will be signed in accordance with NDSC Document No. CW-F-P-002, Company-Wide Authorization Matrix Policy. Request for Proposals (RFP's) will be issued where more information is required from the potential vendors than just price. Process details are available in the NDSC Document No. CW-F-P-004, Guidance on Procurement and Contracts Policy. RFP's allow the laboratory to determine if a vendor is capable of meeting requirements such as supplying all of the laboratory facilities, meeting required quality standards and adhering to necessary ethical and environmental standards. The RFP process also allows potential vendors to outline any additional capabilities they may offer.

11.2 Glassware

Glassware used for volumetric measurements must be Class A or verified for accuracy according to laboratory procedure. Pyrex (or equivalent) glass should be used where possible. For safety purposes, thick-wall glassware should be used where available.

11.3 Reagents, Standards & Supplies

Purchasing guidelines for equipment, consumables, and reagents must meet the requirements of the specific method and testing procedures for which they are being purchased. Solvents and acids are pre-tested in accordance with NDSC Document No. CA-Q-S-001, Solvent & Acid Lot Testing & Approval. Approval information for the solvents and acids tested under Document No. CA-Q-S-001 is stored on the laboratory's SharePoint site, under Solvent Approvals. A master list of all tested materials, as well as the certificates of analysis for the materials, is stored in the same location.

11.3.1 Purchasing

Chemical reagents, solvents, glassware, and general supplies are ordered as needed to maintain sufficient quantities on hand. Materials used in the analytical process must be of a known quality. The wide variety of materials and reagents available makes it advisable to specify recommendations for the name, brand, and grade of materials to be used in any determination. This information is contained in the method SOP. The analyst completes the Material Request Sheet when requesting reagents, standards, or supplies. The analyst may check the item out of the on-site consignment system that contains items approved for laboratory use.

The analyst must provide the master item number (from the master item list that has been approved by the Technical Manager), item description, package size, catalogue page number, and the quantity needed. If an item being ordered is not the exact item requested, approval must be obtained from the Technical Manager prior to placing the order. The purchasing manager places the order.

11.3.2 Receiving

It is the responsibility of the analyst or department manager to receive the shipment. It is the responsibility of the analyst who ordered the materials to document the date materials were received. Once the ordered reagents or materials are received, the analyst compares the information on the label or packaging to the original order to ensure that the purchase meets the quality level specified. This is documented through the addition of the received date and initials to the information present on the daily order log.

The analyst or department manager verifies the lot numbers of received solvents and acids against the pre-approval lists. If a received material is listed as unapproved, or is not listed, it is sequestered and returned to the vendor. Alternatively, the laboratory may test the material for the intended use, and if it is acceptable, document the approval on the approval list. Records of any testing performed locally are maintained on the shared “public” folder on the computer network.

Materials may not be released for use in the laboratory until they have been inspected, verified as suitable for use, and the inspection/verification has been documented.

Safety Data Sheets (SDSs) are available online through the Company’s intranet website. Anyone may review these for relevant information on the safe handling and emergency precautions of on-site chemicals.

11.3.3 Specifications

Methods used in the laboratory specify the grade of reagent that must be used in the procedure. If the quality of the reagent is not specified, analytical reagent grade will be used. It is the responsibility of the analyst to check the procedure carefully for the suitability of grade of reagent.

Chemicals must not be used past the manufacturer’s expiration date and must not be used past the expiration time noted in a method SOP. If expiration dates are not provided, the laboratory may contact the manufacturer to determine an expiration date.

The laboratory assumes a five year expiration date on inorganic dry chemicals and solvents unless noted otherwise by the manufacturer or by the reference source method. Chemicals/solvents should not be used past the manufacturer's or SOP expiration date unless verified as outlined below.

- An expiration date **cannot** be extended if the dry chemical/solvent is discolored or appears otherwise physically degraded. In this case, the dry chemical/solvent must be discarded.
- Expiration dates can be extended if the dry chemical/solvent is found to be satisfactory based on acceptable performance of quality control samples (Continuing Calibration Verification (CCV), Blanks, Laboratory Control Sample (LCS), etc.).
- If the dry chemical/solvent is used for the preparation of standards, the expiration dates can be extended 6 months if the dry chemical/solvent is compared to an unexpired independent source in performing the method and the performance of the dry chemical/solvent is found to be satisfactory. The comparison must show that the dry chemical/solvent meets CCV limits. The comparison must show that the dry chemical/solvent meets CCV limits. Any such comparison studies will be maintained on-file and available for review in the QA Manager's office.

Wherever possible, standards must be traceable to national or international standards of measurement or to national or international reference materials. Records to that effect are available to the user.

Compressed gases in use are checked for pressure and secure positioning daily. To prevent a tank from going to dryness, or introducing potential impurities, the pressure should be closely watched as it decreases to approximately 15% of the original reading, at which point it should be replaced. For example, a standard sized laboratory gas cylinder containing 3,000 psig of gas should be replaced when it drops to approximately 500 psig. The quality of the gases must meet method or manufacturer specification or be of a grade that does not cause any analytical interference.

Water used in the preparation of samples, standards or reagents must have a specific conductivity of less than 1-µmho/cm (or specific resistivity of greater than 1.0 megohm-cm) at 25ºC. The specific conductivity is checked and recorded daily. If the water's specific conductivity is greater than the specified limit, the Facility Manager and appropriate Technical Managers must be notified immediately in order to notify all departments, decide on cessation (based on intended use) of activities, and make arrangements for correction.

The laboratory may purchase reagent grade (or other similar quality) water for use in the laboratory. This water must be certified clean by the supplier for all target analytes or otherwise verified by the laboratory prior to use. This verification is documented.

Standard lots are verified before first time use if the laboratory switches manufacturers or has historically had a problem with the type of standard.

Purchased bottleware used for sampling must be certified clean and the certificates must be maintained. If uncertified sampling bottleware is purchased, all lots must be verified clean prior to use. This verification must be maintained.

Records of manufacturer's certification and traceability statements are maintained in files or binders in each laboratory section. These records include date of receipt, lot number (when applicable), and expiration date (when applicable). Incorporation of the item into the record indicates that the analyst has compared the new certificate with the previous one for the same purpose and that no difference is noted, unless approved and so documented by the Technical Manager or QA Manager.

11.3.4 Storage

Reagent and chemical storage is important from the aspects of both integrity and safety. Light-sensitive reagents may be stored in brown-glass containers. Storage conditions are per the NDSC Environmental Health & Safety Manual, Document No. CW-E-M-001, the local laboratory EH&S manual addendum and method SOPs or manufacturer instructions.

11.4 Purchase of Equipment / Instruments / Software

When a new piece of equipment is needed, either for additional capacity or for replacing inoperable equipment, the analyst or supervisor makes a supply request to the Technical Manager and/or the Laboratory Director. If they agree with the request, the procedures outlined in NDSC Document No. CA-T-P-001 Qualified Products List are followed. A decision is made as to which piece of equipment can best satisfy the requirements. The appropriate written requests are completed and purchasing places the order

Upon receipt of a new or used piece of equipment, an identification name is assigned and added to the equipment list. IT must also be notified so that they can synchronize the instrument for back-ups. Its capability is assessed to determine if it is adequate or not for the specific application. For instruments, a calibration curve is generated, followed by MDLs, Demonstration of Capabilities (DOCs), and other relevant criteria (refer to Section 20). For software, its operation must be deemed reliable and evidence of instrument verification must be retained by the IT Department or QA Department. Software certificates supplied by the vendors are filed with the LIMS Administrator. The manufacturer's operation manual is retained at the bench.

11.5 Services

Service to analytical instruments (except analytical balances) is performed on an as needed basis. Routine preventative maintenance is discussed in Section 22. The need for service is determined by analysts and/or Technical Managers. The service providers that perform the services are approved by the Technical Manager.

Analytical balances are serviced and calibrated annually in accordance with SOP No.ED-GEN-010, Calibration of Analytical Balances. The calibration and maintenance services are performed on-site, and the balances are returned to use immediately following successful calibration. Calibration certificates are filed for reference. If the calibration was unsuccessful, the balance is immediately removed from service and segregated pending either further maintenance or disposal.

Calibration services for support equipment such as thermometers, weight sets, autopipettors, etc., are obtained from vendors with current and valid ISO 17025 accreditation for calibration of the specific piece of equipment. Prior to utilizing the vendor's services, the vendor's accreditation status is verified. Once the equipment has been calibrated, the calibration certificates are reviewed by the QA department, and documentation of the review is filed with the calibration certificates. The equipment is then returned to service within the laboratory

11.6 Suppliers

The laboratory selects vendors through a competitive proposal / bid process, strategic business alliances or negotiated vendor partnerships (contracts).

This process is defined in the NDSC Document No. CW-F-P-004, Procurement & Contracts Policy. The level of control used in the selection process is dependent on the anticipated

spending amount and the potential impact on the laboratory's business. Vendors that provide test and measuring equipment, solvents, standards, certified containers, instrument related service contracts or subcontract laboratory services shall be subject to more rigorous controls than vendors that provide off-the-shelf items of defined quality that meet the end use requirements. The purchasing system includes all suppliers/vendors that have been approved for use.

Evaluation of suppliers is accomplished by ensuring the supplier ships the product or material ordered and that the material is of the appropriate quality. This is documented by signing off on packing slips or other supply receipt documents. The purchasing documents contain the data that adequately describe the services and supplies ordered.

Any issues of vendor performance are to be reported immediately by the laboratory staff to the Purchasing Group by completing a Vendor Performance Report.

The Purchasing Group will work through the appropriate channels to gather the information required to clearly identify the problem and will contact the vendor to report the problem and to make any necessary arrangements for exchange, return authorization, credit, etc.

Suppliers are subject to re-evaluation, as deemed appropriate, through the use of Vendor Performance Reports used to summarize and review to determine corrective action necessary, or service improvements required by vendors

The laboratory has access to a listing of all approved suppliers of critical consumables, supplies and services. This information is provided through the purchasing system.

11.6.1 New Vendor Procedure

Laboratory employees who wish to request the addition of a new vendor must complete a Vendor Add Request Form.

New vendors are evaluated based upon criteria appropriate to the products or services provided as well as their ability to provide those products and services at a competitive cost. Vendors are also evaluated to determine if there are ethical reasons or potential conflicts of interest with laboratory employees that would make it prohibitive to do business with them as well as their financial stability. The QA Department and/or the Technical Services Director are consulted with vendor and product selection that have an impact on quality.

12.0 COMPLAINTS

12.1 Overview

The laboratory considers an effective client complaint handling processes to be of significant business and strategic value. Listening to and documenting client concerns captures client knowledge that enables our operations to continually improve processes and client satisfaction. An effective client complaint handling process also provides assurance to the data user that the laboratory will stand behind its data, service obligations and products.

A client complaint is any expression of dissatisfaction with any aspect of our business services (e.g., communications, responsiveness, data, reports, invoicing and other functions) expressed by any party, whether received verbally or in written form. Client inquiries, complaints or noted discrepancies are documented, communicated to management, and addressed promptly and thoroughly.

The laboratory has procedures for addressing both external and internal complaints with the goal of providing satisfactory resolution to complaints in a timely and professional manner.

The nature of the complaint is identified, documented and investigated, and an appropriate action is determined and taken. In cases where a client complaint indicates that an established policy or procedure was not followed, the QA Department must evaluate whether a special audit must be conducted to assist in resolving the issue. A written confirmation or letter to the client, outlining the issue and response taken is recommended as part of the overall action taken.

The process of complaint resolution and documentation utilizes the procedures outlined in Section 14 (Corrective Actions) and is documented following SOP No. ED-GEN-003, Control of Non-Conformances and Corrective Action. A copy of this procedure will be made available to any interested party on request.

12.2 External Complaints

An employee that receives a complaint initiates the complaint resolution process by first documenting the complaint according SOP No. ED-GEN-003, Control of Non-Conformances and Corrective Action.

Complaints fall into two categories: correctable and non-correctable. An example of a correctable complaint would be one where a report re-issue would resolve the complaint. An example of a non-correctable complaint would be one where a client complains that their data was repeatedly late. Non-correctable complaints should be reviewed for preventive action measures to reduce the likelihood of future occurrence and mitigation of client impact.

The general steps in the complaint handling process are:

- Receiving and documenting complaints
- Acknowledging receipt of complaint, whenever possible
- Complaint investigation and service recovery
- Process improvement

The laboratory shall inform the initiator of the complaint of the results of the investigation and the corrective action taken, if any.

12.3 Internal Complaints

Internal complaints include, but are not limited to: errors and non-conformances, training issues, internal audit findings, and deviations from methods. Corrective actions may be initiated by any staff member who observes a nonconformance and shall follow the procedures outlined in Section 14. In addition, Executive Management, Sales and Marketing and IT may initiate a complaint by contacting the laboratory or through the corrective action system described in Section 14.

12.4 Management Review

The number and nature of client complaints is reported by the QA Manager to the Laboratory Director and Quality Director in the QA Monthly report. Monitoring and addressing the overall level and nature of client complaints and the effectiveness of the solutions is part of the Annual Management Systems Review (Section 18).

13.0 CONTROL OF NON-CONFORMING WORK

13.1 Overview

When data discrepancies are discovered or deviations and departures from laboratory SOPs, policies and/or client requests have occurred, corrective action is taken immediately. First, the laboratory evaluates the significance of the nonconforming work. Then, a corrective action plan is initiated based on the outcome of the evaluation. If it is determined that the nonconforming work is an isolated incident, the plan could be as simple as adding a qualifier to the final results and/or making a notation in the case narrative. If it is determined that the nonconforming work is a systematic or improper practices issue, the corrective action plan could include a more in depth investigation and a possible suspension of an analytical method. In all cases, the actions taken are documented using the laboratory's corrective action system (refer to Section 14).

Due to the frequently unique nature of environmental samples, sometimes departures from documented policies and procedures are needed. When an analyst encounters such a situation, the problem is presented to the supervisor for resolution. The supervisor may elect to discuss it with the Technical Manager or have a representative contact the client to decide on a logical course of action. Once an approach is agreed upon, the analyst documents it using the laboratories corrective action system described in Section 14. This information can then be supplied to the client in the form of a footnote or a case narrative with the report.

Project Management may encounter situations where a client may request that a special procedure be applied to a sample that is not standard lab practice. Based on a technical evaluation, the lab may accept or opt to reject the request based on technical or ethical merit. An example might be the need to report a compound that the lab does not normally report. The lab would not have validated the method for this compound following the procedures in Section 21. The client may request that the compound be reported based only on the calibration. Such a request would need to be approved by the Technical Manager and QA Manager,, documented and included in the project folder. Deviations **must** also be noted on the final report with a statement that the compound is not reported in compliance with TNI (or the analytical method) requirements and the reason. Data being reported to a non-TNI state would need to note the change made to how the method is normally run

13.2 Responsibilities and Authorities

Under certain circumstances, the Laboratory Director, a Technical Manager, or a member of the QA team may authorize departures from documented procedures or policies. The departures may be a result of procedural changes due to the nature of the sample; a one-time procedure for a client; QC failures with insufficient sample to reanalyze, etc. In most cases, the client will be informed of the departure prior to the reporting of the data. Any departures must be well documented using the laboratory's corrective action procedures. This information may also be documented in logbooks and/or data review checklists as appropriate**.** Any impacted data must be referenced in a case narrative and/or flagged with an appropriate data qualifier.

Any misrepresentation or possible misrepresentation of analytical data discovered by any laboratory staff member must be reported to facility Senior Management within 24-hours. The Senior Management staff is comprised of the Laboratory Director, the QA Manager, and the Technical Managers. The reporting of issues involving alleged violations of the company's Data Integrity or Manual Integration procedures must be conveyed to an ECO (e.g., the VP-QA/EHS) and the laboratory's Quality Director within 24 hours of discovery.

Whether an inaccurate result was reported due to calculation or quantitation errors, data entry errors, improper practices, or failure to follow SOPs, the data must be evaluated to determine the possible effect.

The Laboratory Director, QA Manager, ECOs, VP of for due cause as well as authorize the resumption of work.

13.3 Evaluation of Significance and Actions Taken

For each nonconforming issue reported, an evaluation of its significance and the level of management involvement needed is made. This includes reviewing its impact on the final data, whether or not it is an isolated or systematic issue, and how it relates to any special client requirements.

The NDSC Document entitled Data Recalls (CW-Q-S-005) is the procedure to be followed when it is discovered that erroneous or biased data may have been reported to clients or regulatory agencies.

The NDSC Document entitled Internal Investigations (CW-L-S-002) is the procedure to be followed for investigation and correction of situations involved alleged incidents of misconduct or violation of the company's ethics policy.

Laboratory level decisions are documented and approved using the laboratory's standard nonconformance/corrective action reporting in lieu of the data recall determination form contained in NDSC Document No.CW-Q-S-005.

13.4 Prevention of Nonconforming Work

If it is determined that the nonconforming work could recur, further corrective actions must be made following the laboratory's corrective action system. Periodically as defined by the laboratory's preventive action schedule, the QA Department evaluates non-conformances to determine if any nonconforming work has been repeated multiple times. If so, the laboratory's corrective action process may be followed.

13.5 Method Suspension / Restriction (Stop Work Procedures)

In some cases, it may be necessary to suspend/restrict the use of a method or target analyte which constitutes significant risk and/or liability to the laboratory. Suspension/restriction procedures can be initiated by any of the persons noted in Section 13.2, Paragraph 5.

Prior to suspension/restriction, confidentiality will be respected, and the problem with the required corrective and preventive action will be stated in writing and presented to the Laboratory Director.

The Laboratory Director shall arrange for the appropriate personnel to meet with the QA Manager as needed. This meeting shall be held to confirm that there is a problem, that suspension/restriction of the method is required and will be concluded with a discussion of the steps necessary to bring the method/target or test fully back on line. In some cases, that may not be necessary if all appropriate personnel have already agreed there is a problem and there is agreement on the steps needed to bring the method, target or test fully back on line. The QA Manager will also initiate a corrective action report as described in Section 14 if one has not already been started. A copy of any meeting notes and agreed upon steps should be e-mailed by the laboratory to their Business Unit President; VP-QA & EHS. This e-mail acts as notification of the incident.

After suspension/restriction, the lab will hold all reports to clients pending review. No faxing, mailing or distributing through electronic means may occur. The report must not be posted for viewing on the internet. It is the responsibility of the Laboratory Director to hold all reporting and to notify all relevant laboratory personnel regarding the suspension/restriction (e.g., Project Management, Log-in, etc.). Clients will NOT generally be notified at this time. Analysis may proceed in some instances depending on the non-conformance issue.

Within 72 hours, the QA Manager will determine if compliance is now met and reports can be released, OR determine the plan of action to bring work into compliance, and release work. A team, with all principals involved (e.g., Laboratory Director, Technical Manager, QA Manager) can devise a start-up plan to cover all steps from client notification through compliance and release of reports. Project Management and the Directors of Client Services and Sales and Marketing must be notified if clients must be notified or if the suspension/restriction affects the laboratory's ability to accept work. The QA Manager must approve start-up or elimination of any restrictions after all corrective action is complete.

14.0 CORRECTIVE ACTION

14.1 Overview

A major component of the laboratory's (QA) Program is the problem investigation and feedback mechanism designed to keep the laboratory staff informed on quality related issues and to provide insight to problem resolution. When nonconforming work or departures from policies and procedures in the quality system or technical operations are identified, the corrective action procedure provides a systematic approach to assess the issues, restore the laboratory's system integrity, and prevent reoccurrence. The laboratory employs two systems to manage non-conformances. Issues suspected of being systematic in nature and for which root cause analysis and a formal Corrective Action Report (CAR) are documented in the Incident Corrective Action Tracking (ICAT) database. Routine batch non-conformances, events that are understood to be isolated in nature, are documented in the LIMS non-conformance memo (NCM) system.

14.2 General

Problems within the quality system or within analytical operations may be discovered in a variety of ways, such as QC sample failures, internal or external audits, proficiency testing (PT) performance, client complaints, staff observation, etc.

The purpose of a corrective action system is to:

- Identify non-conformance events and assign responsibility for investigating.
- Resolve non-conformance events and assign responsibility for any required corrective action.
- Identify systematic problems before they become serious.
- Identify and track client complaints and provide resolution.

14.2.1 Non-Conformance Memo (NCM)

NCMs are used to document the following types of corrective actions:

- Deviations from an established procedure or SOP
- QC outside of limits

- Isolated reporting / calculation errors
- Client complaints
- Discrepancies in materials / goods received vs. manufacturer packing slips (Forms of documentation other than NCMs in LIMS are also acceptable)

14.2.2 Corrective Actions Documented In the ICAT Database

- Internal and external audit findings
- Failed or unacceptable PT results
- Identified poor process or method performance trends
- Systematic reporting / calculation errors
- Data recall investigations
- Questionable trends that are found in the review of NCMs.
- Client complaints
- Excessive revised reports

The ICAT database is used to document background information, track the results of corrective action investigations and root cause analysis, and to provide reports of corrective action plans.

14.3 Closed Loop Corrective Action Process

Any employee in the company can initiate a corrective action. There are four main components to a closed-loop corrective action process once an issue has been identified: Cause Analysis, Selection and Implementation of Corrective Actions (both short and long term), Monitoring of the Corrective Actions, and Follow-up.

14.3.1 Cause Analysis

- Upon discovery of a non-conformance event, the event must be defined and documented. An entry into the ICAT system must be initiated, someone is assigned to investigate the issue and the event is investigated for cause. Table 12-1 provides some general guidelines on determining responsibility for assessment.
- The cause analysis step is the key to the process as a long term corrective action cannot be determined until the cause is determined.
- If the cause is not readily obvious, the Technical Manager, Laboratory Director, or QA Manager (or QA designee) is consulted.

14.3.2 Selection and Implementation of Corrective Actions

- Where corrective action is needed, the laboratory shall identify potential corrective actions. The action(s) most likely to eliminate the problem and prevent recurrence are selected and implemented. Responsibility for implementation is assigned.
- Corrective actions shall be to a degree appropriate to the magnitude of the problem identified through the cause analysis.
- Whatever corrective action is determined to be appropriate, the laboratory shall document and implement the changes. The ICAT record is used for this documentation.

14.3.3 Root Cause Analysis

Root Cause Analysis is a class of problem solving (investigative) methods aimed at identifying the basic or causal factor(s) that underlie variation in performance or the occurrence of a significant failure. The root cause may be buried under seemingly innocuous events, many steps preceding the perceived failure. At first glance, the immediate response is typically directed at a symptom and not the cause. Typically, root cause analysis would be best with three or more incidents to triangulate a weakness. NDSC Document No. CA-Q-S-009, Root Cause Analysis, provides guidance on this procedure

Systematically analyze and document the root causes of the more significant problems that are reported. Identify, track, and implement the corrective actions required to reduce the likelihood of recurrence of significant incidents. Trend the root cause data from these incidents to identify root causes that, when corrected, can lead to dramatic improvements in performance by eliminating entire classes of problems.

Identify the one event associated with problem and ask why this event occurred. Brainstorm the root causes of failures; for example, by asking why events occurred or conditions existed; and then why the cause occurred consecutive times until you get to the root cause. For each of these sub events or causes, ask why it occurred. Repeat the process for the other events associated with the incident.

Root cause analysis does not mean the investigation is over. Look at technique or other systems outside the normal indicators. Often creative thinking will find root causes that ordinarily would be missed and continue to plague the laboratory or operation.

14.3.4 Monitoring of the Corrective Actions

- The Technical Manager and QA Manager are responsible to ensure that the corrective action taken was effective.
- Ineffective actions are documented and re-evaluated until acceptable resolution is achieved. Technical Managers are accountable to the Laboratory Director to ensure final acceptable resolution is achieved and documented appropriately.
- The QA Manager reviews monthly NCM and ICAT records for trends. Highlights are included in the QA monthly report (refer to Section 18). If a significant trend develops that adversely affects quality, an audit of the area is performed and corrective action implemented.
- Any out-of-control situations that are not addressed acceptably at the laboratory level may be reported to the NDSC Quality Director by the QA Manager, indicating the nature of the out-of-control situation and problems encountered in solving the situation.

14.3.5 Follow-up Audits

- Follow-up audits may be initiated by the QA Manager and shall be performed as soon as possible when the identification of a nonconformance casts doubt on the laboratory's compliance with its own policies and procedures, or on its compliance with state or federal requirements.
- These audits often follow the implementation of the corrective actions to verify effectiveness. An additional audit would only be necessary when a critical issue or risk to business is discovered.

(Also refer to Section 17.1.4, Special Audits.)

14.4 Technical Corrective Actions

In addition to providing acceptance criteria and specific protocols for technical corrective actions in the method SOPs, the laboratory has general procedures to be followed to determine when departures from the documented policies and procedures and quality control have occurred (refer to Section 13). The documentation of these procedures is through the use of an NCM or record in the ICAT system.

Table 14-1 includes examples of general technical corrective actions. For specific criteria and corrective actions, refer to the analytical methods or specific method SOPs. The laboratory may also maintain Work Instructions on these items that are available upon request.

Table 14-1 provides some general guidelines for identifying the individual(s) responsible for assessing each QC type and initiating corrective action. The table also provides general guidance on how a data set should be treated if associated QC measurements are unacceptable. Specific procedures are included in Method SOPs, Work Instructions, QA Manual Sections 19 and 20. All corrective actions are reviewed monthly, at a minimum, by the QA Manager and highlights are included in the QA monthly report.

To the extent possible, samples shall be reported only if all quality control measures are acceptable. If the deficiency does not impair the usability of the results, data will be reported with an appropriate data qualifier and/or the deficiency will be noted in the case narrative. Where sample results may be impaired, the PM is notified by an NCM and appropriate corrective action (e.g., reanalysis) is taken and documented**.**

14.5 Basic Corrections

When mistakes occur in records, each mistake shall be crossed-out, [not obliterated (e.g. no white-out)], and the correct value entered alongside. All such corrections shall be initialed (or signed) and dated by the person making the correction. In the case of records stored electronically, the original uncorrected file must be maintained intact and a second corrected file is created. This same process applies to adding additional information to a record. All additions made later than the initial must also be initialed (or signed) and dated. When corrections are due to reasons other than obvious transcription errors, the reason for the corrections (or additions) shall also be documented.

Table 14-1. Example – General Corrective Action Procedures

QC Activity ***(Individual Responsible for Initiation/Assessment)***	**Acceptance Criteria**	**Recommended Corrective Action**
Initial Instrument Blank *(Analyst)*	- Instrument response < MDL.	- Prepare another blank. - If same response, determine cause of contamination: reagents, environment, instrument equipment failure, etc..
Initial Calibration Standards *(Analyst, Technical Manager(s))*	- Correlation coefficient > 0.99 or standard concentration value. - % Recovery within acceptance range. - See details in Method SOP.	- Reanalyze standards. - If still unacceptable, remake standards and recalibrate instrument.

QC Activity *(Individual Responsible for Initiation/Assessment)*	Acceptance Criteria	Recommended Corrective Action
Independent Calibration Verification (Second Source) *(Analyst, Technical Manager(s))*	- % Recovery within control limits.	- Remake and reanalyze standard. - If still unacceptable, then remake calibration standards or use new primary standards and recalibrate instrument.
Continuing Calibration Standards *(Analyst, Data Reviewer)*	% Recovery within control limits.	- Reanalyze standard. - If still unacceptable, then recalibrate and rerun affected samples.
Matrix Spike / Matrix Spike Duplicate (MS/MSD) *(Analyst, Data Reviewer)*	- % Recovery within limits documented in TALS Method Limit Group	- If the acceptance criteria for duplicates or matrix spikes are not met because of matrix interferences, the acceptance of the analytical batch is determined by the validity of the LCS. - If the LCS is within acceptable limits the batch is acceptable. - The results of the duplicates, matrix spikes and the LCS are reported with the data set. - For matrix spike or duplicate results outside criteria the data for that sample shall be reported with qualifiers.
Laboratory Control Sample (LCS) *(Analyst, Data Reviewer)*	- % Recovery within limits specified in TALS Method Limit Group	- Batch must be re-prepared and re-analyzed. This includes any allowable marginal exceedance. When not using marginal exceedances, the following exceptions apply: 1) when the acceptance criteria for the positive control are exceeded high (i.e., high bias) and there are associated samples that are non-detects, then those non-detects may be reported with data qualifying codes; 2) when the acceptance criteria for the positive control are exceeded low (i.e., low bias), those sample results may be reported if they exceed a maximum regulatory limit/decision level with data qualifying codes. **Note:** If there is insufficient sample or the holding time cannot be met, contact client and report with flags.
Surrogates *(Analyst, Data Reviewer)*	- % Recovery within limits of method or within three standard deviations of the historical mean.	- Individual sample must be repeated. Place comment in LIMS. - Surrogate results outside criteria shall be reported with qualifiers.

QC Activity *(Individual Responsible for Initiation/Assessment)*	Acceptance Criteria	Recommended Corrective Action
Method Blank (MB) *(Analyst, Data Reviewer)*	< Reporting Limit [1]	- Reanalyze blank. - If still positive, determine source of contamination. If necessary, reprocess (i.e. digest or extract) entire sample batch. Report blank results. - Qualify the result(s) if the concentration of a targeted analyte in the MB is at or above the reporting limit AND is > 1/10 of the amount measured in the sample.
Proficiency Testing (PT) Samples *(QA Manager, Technical Manager(s))*	- Criteria supplied by PT Supplier.	- Any failures or warnings must be investigated for cause. Failures may result in the need to repeat a PT sample to show the problem is corrected.
Internal / External Audits *(QA Manager, Technical Manager(s), Laboratory Director)*	- Defined in Quality System documentation such as SOPs, QAM, etc..	- Non-conformances must be investigated through CAR system and necessary corrections must be made.
Reporting / Calculation Errors (Depends on issue – possible individuals include: Analysts, Data Reviewers, Project Managers, Technical Managers, QA Manager, NDSC-QA, Executive Management)	- NDSC Document No. CW-Q-S-005, Data Recall **or** local SOP No.	- Corrective action is determined by type of error. Follow the procedures in NDSC Document No. CW-L-S-002 **or** in lab SOP No. ED-GEN-003, Non-Conformances and Corrective Action.
Client Complaints *(Project Managers, Lab Director/Manager, Sales and Marketing)*	-	- Corrective action is determined by the type of complaint. For example, a complaint regarding an incorrect address on a report will result in the report being corrected and then follow-up must be performed on the reasons the address was incorrect (e.g., database needs to be updated).
QA Monthly Report (Refer to Section 16 for an example) (QA Manager, Lab Director/Manager, *Technical Manager(s)*)	- QAM, SOPs.	- Corrective action is determined by the type of issue. For example, CARs for the month are reviewed and possible trends are investigated.

QC Activity *(Individual Responsible for Initiation/Assessment)*	Acceptance Criteria	Recommended Corrective Action
Health and Safety Violation (Safety Officer, Lab Director/Manager, *Technical Manager(s)*)	- Environmental Health and Safety (EHS) Manual.	- Non-conformance is investigated and corrected through CAR system.

Note:
1. Except as noted below for certain compounds, the method blank should be below the reporting limit. Concentrations up to five times the reporting limit will be allowed for the ubiquitous laboratory and reagent contaminants: methylene chloride, toluene, acetone, 2-butanone and phthalates **provided** they appear in similar levels in the reagent blank and samples. This allowance presumes that the detection limit is significantly below any regulatory limit to which the data are to be compared and that blank subtraction will not occur. For analytes for which regulatory limits are extremely close to the detection limit, the method blank must be below the method detection limit

15.0 PREVENTIVE ACTION / IMPROVEMENT

15.1 Overview

The laboratory's preventive action programs improve or eliminate potential causes of nonconforming product and/or nonconformance to the quality system. This preventive action process is a proactive and continuous process of improvement activities that can be initiated through feedback from clients, employees, business providers, and affiliates. The QA Department has the overall responsibility to ensure that the preventive action process is in place, and that relevant information on actions is submitted for management review.

Dedicating resources to an effective preventive action system emphasizes the laboratory's commitment to its QA Program. It is beneficial to identify and address negative trends before they develop into complaints, problems and corrective actions. Additionally, the laboratory continually strives to improve customer service and client satisfaction through continuous improvements to laboratory systems.

Opportunities for improvement may be discovered through any of the following:

- review of the monthly QA Metrics Report,
- trending NCMs,
- review of control charts and QC results,
- trending proficiency testing (PT) results,
- performance of management system reviews,
- trending client complaints,
- review of processing operations, or
- staff observations.

The monthly Management Systems Metrics Report shows performance indicators in all areas of the laboratory and quality system. These areas include revised reports, corrective actions, audit findings, internal auditing and data authenticity audits, client complaints, PT samples, holding time violations, SOPs, ethics training, etc. The metrics report is reviewed monthly by the laboratory management, NDSC QA Team, Local and Executive Management. These metrics are used ~~to~~ in evaluating the management and quality system performance on an ongoing basis and provide a tool for identifying areas for improvement.

Items identified as continuous improvement opportunities to the management system may be issued as goals from the annual management systems review, recommendations from internal audits, white papers, Lessons Learned, Technical Services audit report, Technical Best Practices, or as Executive or management initiatives.

The laboratory's corrective action process is integral to implementation of preventive actions. A critical piece of the corrective action process is the implementation of actions to prevent further occurrence of a non-compliance event. Historical review of corrective action and non-conformances provides a valuable mechanism for identifying preventive action opportunities.

15.1.1 The following elements are part of a preventive action/process improvement system:

- Identification of an opportunity for preventive action or process improvement.
- Process for the preventive action or improvement.
- Define the measurements of the effectiveness of the process once undertaken.
- Execution of the preventive action or improvement.
- Evaluation of the plan using the defined measurements.
- Verification of the effectiveness of the preventive action or improvement.
- Close-Out by documenting any permanent changes to the Quality System as a result of the Preventive Action or Process Improvement. Documentation of Preventive Action/process Improvement is incorporated into the monthly QA reports, corrective action process and management review.

15.1.2 Any preventive actions/process improvement undertaken or attempted shall be taken into account during the annual Management Systems Review (Section 16). A highly detailed report is not required; however, a summary of successes and failures within the preventive action program is sufficient to provide management with a measurement for evaluation.

16.0 CONTROL OF RECORDS

The laboratory maintains a records management system appropriate to its needs and that complies with applicable standards or regulations as required. The system produces unequivocal, accurate records that document all laboratory activities. The laboratory retains all original observations, calculations and derived data, calibration records and a copy of the analytical report for a minimum of five years after it has been issued. Exceptions for programs with longer retention requirements are discussed in Section 14.1.2.

16.1 Overview

The laboratory has established procedures for identification, collection, indexing, access, filing, storage, maintenance and disposal of quality and technical records. A record index is listed in Table 14-1. More detailed information on retention of specific records is provided in NDSC

Document Nos. CW-L-P-001, Records Retention Policy, CW-L-WI-001,Records Retention/Storage Schedule and local SOP No. ED-GEN-024, Records Storage and Retention.>. Quality records are maintained by the QA department in a database, which is backed up as part of the regular laboratory backup. Records are of two types; either electronic or hard copy paper formats depending on whether the record is computer or hand generated (some records may be in both formats). Technical records are maintained by department supervisors.

Table 16-1. Record Index

	Record Types [1]:	**Retention Time:**
Technical Records	- Raw Data - Logbooks[2] - Standards - Certificates - Analytical Records - MDLs/IDLs/DOCs - Lab Reports	5 Years from analytical report issue*
Official Documents	- Quality Assurance Manual (QAM) - Work Instructions - Policies - SOPs - Policy Memorandums - Manuals - Published Methods	Indefinitely
QA Records	- Certifications - Method and Software Validation / Verification Data	Indefinitely
QA Records	- Internal & External Audits/Responses - Corrective/Preventive Actions - Management Reviews - Data Investigation	5 Years from archival* **Data Investigation:** 5 years or the life of the affected raw data storage whichever is greater (beyond 5 years if ongoing project or pending investigation)
Project Records	- Sample Receipt & COC Documents - Contracts and Amendments - Correspondence - QAPP - SAP - Telephone Logbooks - Lab Reports	5 Years from analytical report issue*
Administrative Records	Financial and Business Operations	Refer to NDSC Doc. No. CW-L-WI-001
	EH&S Manual, Permits	Indefinitely
	Disposal Records	Indefinitely
	Employee Handbook	Indefinitely
	Personnel files, Employee Signature & Initials, Administrative Training Records (e.g., Ethics)	Refer to HR Manual
	Administrative Policies	Indefinitely

	Record Types [1]:	Retention Time:
	Technical Training Records	7 years
	Legal Records	Indefinitely
	HR Records	Refer to NDSC Doc. No. CW-L-WI-001
	IT Records	Refer to NDSC Doc. No. CW-L-WI-001
	NDSC Governance Records	Refer to NDSC Doc. No. CW-L-WI-001
	Sales & Marketing	5 years
	Real Estate	Indefinitely

[1] Record Types encompass hardcopy and electronic records.
[2] Examples of Logbook types: Maintenance, Instrument Run, Preparation (standard and samples), Standard and Reagent Receipt, Archiving, Balance Calibration, Temperature (hardcopy or electronic records).
* Exceptions listed in Table 16-2.

16.1.1 All records are stored and retained in such a way that they are secure and readily retrievable at the laboratory facility that provides a suitable environment to prevent damage or deterioration and to prevent loss**.** All records shall be protected against fire, theft, loss, environmental deterioration, and vermin. In the case of electronic records, electronic or magnetic sources, storage media are protected from deterioration caused by magnetic fields and/or electronic deterioration.

Electronic data backups are performed as described in the Eurofins Data Back-Up Policy (CW-I-P-010, current revision). Backups occur at a frequency that ensures that no more than one business day of critical data will be lost in the event of a system failure. The Edison laboratory uses Citrix as part of its data management system. All laboratory data is copied offsite to the Eurofins Datacenter, which is located in Colorado. The Edison facility maintains a local instrument fileserver to which all local data is copied before it is transmitted to the Datacenter and a backup virtual server as redundancy. All instrument data files are located on the local file server, EDI0011 and each evening at 6:00pm ET entire instrument data folder structure is copied to the redundant virtual server EDICV01. Every evening starting at 10:00pm ET the data is then replicated from EDICV01 to the Datacenter in Colorado where it's backed up to tape.

Access to the data is limited to laboratory and company employees and shall be documented with an access log**.** Records archived off-site are stored in a secure location where a record is maintained of any entry into the storage facility. Whether on-site or off-site storage is used, logs are maintained in each storage box to note removal and return of records. Retention of records are maintained on-site at the laboratory for the full length of the required storage time. Records are maintained for a minimum of five years unless otherwise specified by a client or regulatory requirement.

For raw data and project records, record retention shall be calculated from the date the project report is issued. For other records, such as NDSC and/or KGD Documents, QA, or Administrative Records, the retention time is calculated from the date the record is formally retired. Records related to the programs listed in Table 16-2 have lengthier retention requirements and are subject to the requirements in Section 16.1.3.

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16.2 Programs with Longer Retention Requirements

Some regulatory programs have longer record retention requirements than the standard record retention time. These are detailed in Table 16-2 with their retention requirements. In these

cases, the longer retention requirement is enacted. If special instructions exist such that client data cannot be destroyed prior to notification of the client, the container or box containing that data is marked as to who to contact for authorization prior to destroying the data.

Table 16-2. Example: Special Record Retention Requirements

Program	[1]Retention Requirement
Drinking Water – All States	10 years (lab reports and raw data) 10 years - Radiochemistry (project records)
Drinking Water Lead and Copper Rule	12 years (project records)
Commonwealth of MA – All environmental data 310 CMR 42.14	10 years
OSHA	30 years

[1]Note: Extended retention requirements must be noted with the archive documents or addressed in facility-specific records retention procedures.

16.2.1 The laboratory has procedures to protect and back-up records stored electronically and to prevent unauthorized access to or amendment of these records. All analytical data is maintained as hard copy or in a secure readable electronic format. For analytical reports that are maintained as copies in PDF format, refer to Section 19.15.1 for more information.

16.2.2 The record keeping system allows for historical reconstruction of all laboratory activities that produced the analytical data, as well as rapid recovery of historical data. The history of the sample from when the laboratory took possession of the samples must be readily understood through the documentation. This shall include inter-laboratory transfers of samples and/or extracts.

- The records include the identity of personnel involved in sampling, sample receipt, preparation, or testing. All analytical work contains the initials (at least) of the personnel involved. The laboratory's copy of the COC is stored with the invoice and the work order sheet generated by LIMS. The chain of custody would indicate the name of the sampler. If any sampling notes are provided with a work order, they are kept with this package.
- All information relating to the laboratory facilities' equipment, analytical test methods, and related laboratory activities, such as sample receipt, sample preparation, or data verification are documented.
- The record keeping system facilitates the retrieval of all working files and archived records for inspection and verification purposes (e.g., set format for naming electronic files, set format for what is included with a given analytical data set). Instrument data is stored sequentially by instrument. A given day's analyses are maintained in the order of the analysis. Run logs are maintained for each instrument or method; a copy of each day's run log or instrument sequence is stored with the data to aid in re-constructing an analytical sequence. Where an analysis is performed without an instrument, bound logbooks or bench sheets are used to record and file data. Standard and reagent information is recorded in logbooks or entered into LIMS for each method as required.
- Changes to hardcopy records shall follow the procedures outlined in Section 12 and 19. Changes to electronic records in LIMS or instrument data are recorded in audit trails.
- The reason for a signature or initials on a document is clearly indicated in the records such as "sampled by," "prepared by," "reviewed by", or "analyzed by".

- All generated data except those that are generated by automated data collection systems, are recorded directly, promptly and legibly in permanent dark ink.
- Hard copy data may be scanned into PDF format for record storage as long as the scanning process can be verified in order to ensure that no data is lost and the data files and storage media must be tested to verify the laboratory's ability to retrieve the information prior to the destruction of the hard copy that was scanned. The procedure for this verification can be found in SOP No. ED-GEN-021, Data Review.
- Also refer to Section 19.15.1 'Computer and Electronic Data Related Requirements'.

16.3 Technical and Analytical Records

16.3.1 The laboratory retains records of original observations, derived data and sufficient information to establish an audit trail, calibration records, staff records and a copy of each analytical report issued, for a minimum of five years unless otherwise specified by a client or regulatory requirement. The records for each analysis shall contain sufficient information to enable the analysis to be repeated under conditions as close as possible to the original. The records shall include the identity of laboratory personnel responsible for the sampling, performance of each analysis and reviewing results.

16.3.2 Observations, data and calculations are recorded real-time and are identifiable to the specific task.

16.3.3 Changes to hardcopy records shall follow the procedures outlined in Section 12 and 19. Changes to electronic records in LIMS or instrument data are recorded in audit trails.

The essential information to be associated with analysis, such as strip charts, tabular printouts, computer data files, analytical notebooks, and run logs, include:

- Laboratory sample ID code;
- Date of analysis; time of analysis is also required if the holding time is seventy-two (72) hours or less, or when time critical steps are included in the analysis (e.g., drying times, incubations, etc.); instrumental analyses have the date and time of analysis recorded as part of their general operations. Where a time critical step exists in an analysis, location for such a time is included as part of the documentation in a specific logbook or on a benchsheet.
- Instrumentation identification and instrument operating conditions/parameters. Operating conditions/parameters are typically recorded in instrument maintenance logs where available.
- analysis type;
- all manual calculations and manual integrations;
- analyst's or operator's initials/signature;
- sample preparation including cleanup, separation protocols, incubation periods or subculture, ID codes, volumes, weights, instrument printouts, meter readings, calculations, reagents;
- test results;
- standard and reagent origin, receipt, preparation, and use;
- calibration criteria, frequency and acceptance criteria;
- data and statistical calculations, review, confirmation, interpretation, assessment and

reporting conventions;

- quality control protocols and assessment;
- electronic data security, software documentation and verification, software and hardware audits, backups, and records of any changes to automated data entries; and
- Method performance criteria including expected quality control requirements. These are indicated both in LIMS and on specific analytical report formats.

16.3.4 All logbooks used during receipt, preparation, storage, analysis, and reporting of samples or monitoring of support equipment shall undergo a periodic, documented supervisory or peer review.

16.4 Laboratory Support Activities

In addition to documenting all the above-mentioned activities, the following are retained QA records and project records (previous discussions in this section relate where and how these data are stored):

- all original raw data, whether hard copy or electronic, for calibrations, samples and quality control measures, including analysts' work sheets and data output records (chromatograms, strip charts, and other instrument response readout records);
- a written description or reference to the specific test method used which includes a description of the specific computational steps used to translate parametric observations into a reportable analytical value;
- copies of final reports;
- archived SOPs;
- correspondence relating to laboratory activities for a specific project;
- all corrective action reports, audits and audit responses;
- proficiency test results and raw data; and
- results of data review, verification, and crosschecking procedures

16.4.1 Sample Handling Records

Records of all procedures to which a sample is subjected while in the possession of the laboratory are maintained. These include but are not limited to records pertaining to:

- sample preservation including appropriateness of sample container and compliance with holding time requirement;
- sample identification, receipt, acceptance or rejection and login;
- sample storage and tracking including shipping receipts, sample transmittal / COC forms; and
- procedures for the receipt and retention of samples, including all provisions necessary to protect the integrity of samples.

16.5 Administrative Records

The laboratory also maintains the administrative records in either electronic or hard copy form.

Refer to Table 16-1.

16.6 Records Management, Storage and Disposal

All records (including those pertaining to test equipment), certificates and reports are safely stored, held secure and in confidence to the client. Certification related records are available upon request.

All information necessary for the historical reconstruction of data is maintained by the laboratory. Records that are stored only on electronic media must be supported by the hardware and software necessary for their retrieval.

Records that are stored or generated by computers or personal computers have hard copy, write-protected backup copies, or an electronic audit trail controlling access**.**

The laboratory has a record management system (a.k.a., document control) for control of laboratory notebooks, instrument logbooks, standards logbooks, and records for data reduction, validation, storage and reporting. Laboratory notebooks are issued on a per analysis basis, and are numbered sequentially. All data are recorded sequentially within a series of sequential notebooks. Bench sheets are filed sequentially. Standards are maintained in LIMS – no logbooks are used to record that data**.** Records are considered archived when noted as such in the records management system (a.k.a., document control).

16.6.1 Transfer of Ownership

In the event that the laboratory transfers ownership or goes out of business, the laboratory shall ensure that the records are maintained or transferred according to client's instructions. Upon ownership transfer, record retention requirements shall be addressed in the ownership transfer agreement and the responsibility for maintaining archives is clearly established. In addition, in cases of bankruptcy, appropriate regulatory and state legal requirements concerning laboratory records must be followed. In the event of the closure of the laboratory, all records will revert to the control of NDSC. Should the entire company cease to exist, as much notice as possible will be given to clients and the accrediting bodies who have worked with the laboratory during the previous 5 years of such action.

16.6.2 Records Disposal

Records are removed from the archive and destroyed after 5 years unless otherwise specified by a client or regulatory requirement. On a project specific or program basis, clients may need to be notified prior to record destruction. Records are destroyed in a manner that ensures their confidentiality such as shredding, mutilation or incineration. (Refer to Tables 14-1 and 14-2).

Electronic copies of records must be destroyed by erasure or physically damaging off-line storage media so no records can be read.

If a third party records management company is hired to dispose of records, a "Certificate of Destruction" is required.

17.0 AUDITS

17.1 Internal Audits

Internal audits are performed to verify that laboratory operations comply with the requirements of the lab's quality system and with the external quality programs under which the laboratory operates. Audits are planned and organized by the QA staff. Personnel conducting the audits should be independent of the area being evaluated. Auditors will have sufficient authority, access to work areas, and organizational freedom necessary to observe all activities affecting quality and to report the assessments to laboratory management and, when requested, to Executive Management..

Audits are conducted and documented as described in the NDSC Document No. on performing Internal Auditing, SOP No. CW-Q-S-003. The types and frequency of routine internal audits are described in Table 17-1. Special or ad hoc assessments may be conducted as needed under the direction of the QA staff.

Table 17-1. Types of Internal Audits and Frequency

Description	Performed by	Frequency
Quality Systems Audits	QA Department, QA approved designee, or NDSC QA	All areas of the laboratory annually
Method Audits QA Technical Audits	Joint responsibility: a) QA Manager or designee b) Technical Manager or Designee (Refer to NDSC CW-Q-S-003)	QA Technical Audits Frequency: 50% of methods annually
SOP Method Compliance	Joint responsibility: a) QA Manager or designee b) Technical Manager or Designee (Refer to NDSC CW-Q-S-003)	SOP Compliance Review Frequency: • Every 2 years • Every 1 year (Drinking Water Methods)
Special	QA Department or Designee	Surveillance or spot checks performed as needed, e.g., to confirm corrective actions from other audits.
Performance Testing	Analysts with QA oversight	Two successful per year for each TNI-field of testing or as dictated by regulatory requirements

17.1.1 Annual Quality Systems Audit

An annual quality systems audit is required to ensure compliance to analytical methods and SOPs, Eurofins Data Integrity and Ethics Policies (see Sec. 7.2), TNI quality systems, client and state requirements, and the effectiveness of the internal controls of the analytical process, including but not limited to data review, quality controls, preventive action and corrective action. The completeness of earlier corrective actions is assessed for effectiveness & sustainability.

The audit is divided into sections for each operating or support area of the lab, and each section is comprehensive for a given area. The area audits may be performed on a rotating schedule throughout the year to ensure adequate coverage of all areas. This schedule may change as situations in the laboratory warrant.

17.1.2 QA Technical Audits

QA technical audits assess data authenticity and analyst integrity. These audits are based on client projects, associated sample delivery groups, and the methods performed. Reported results are compared to raw data to verify the authenticity of results. The validity of calibrations and QC results are compared to data qualifiers, footnotes, and case narratives. Documentation is assessed by examining run logs and records of manual integrations. Manual calculations are checked. Where possible, electronic audit miner programs (e.g., Chrom AuditMiner) are used to identify unusual manipulations of the data deserving closer scrutiny. QA technical audits will include all methods within a two-year period.

17.1.3 SOP Method Compliance

Compliance of all SOPs with the source methods and compliance of the operational groups with the SOPs will be assessed by the Technical Manager or qualified designee at least every two years. It is also recommended that the work of each newly hired analyst is assessed within 3 months of working independently, (e.g., completion of method IDOC). In addition, as analysts add methods to their capabilities, (new IDOC) reviews of the analyst work products will be performed within 3 months of completing the documented training.

17.1.4 Special Audits

Special audits are conducted on an as needed basis, generally as a follow up to specific issues such as client complaints, corrective actions, PT results, data audits, system audits, validation comments, regulatory audits or suspected ethical improprieties. Special audits are focused on a specific issue, and report format, distribution, and timeframes are designed to address the nature of the issue.

17.1.5 Performance Testing

The laboratory participates semi-annually in performance audits conducted through the analysis of PT samples provided by a third party. The laboratory generally participates in the following types of PT studies: Drinking Water, Non-potable Water and Soil.

It is Eurofins policy that PT samples be treated as typical samples in the production process. Furthermore, where PT samples present special or unique problems, in the regular production process they may need to be treated differently, as would any special or unique request submitted by any client. The QA Manager must be consulted and in agreement with any decisions made to treat a PT sample differently due to some special circumstance.

Written investigations for unacceptable PT results are required. In some cases it may be necessary for blind QC samples to be submitted to the laboratory to show a return to control.

17.2 External Audits

External audits are performed when certifying agencies or clients conduct on-site inspections or submit performance testing samples for analysis. It is Eurofins policy to cooperate fully with regulatory authorities and clients. The laboratory makes every effort to provide the auditors with

access to personnel, documentation, and assistance. Laboratory supervisors are responsible for providing corrective actions to the QA Manager who coordinates the response. Audit responses are due in the time allotted by the client or agency performing the audit.

The laboratory cooperates with clients and their representatives to monitor the laboratory's performance in relation to work performed for the client. The client may only view data and systems related directly to the client's work. All efforts are made to keep other client information confidential.

17.2.1 Confidential Business Information (CBI) Considerations

During on-site audits, auditors may come into possession of information claimed as business confidential. A business confidentiality claim is defined as "a claim or allegation that business information is entitled to confidential treatment for reasons of business confidentiality or a request for a determination that such information is entitled to such treatment." When information is claimed as business confidential, the laboratory must place on (or attach to) the information at the time it is submitted to the auditor, a cover sheet, stamped or typed legend or other suitable form of notice, employing language such as "trade secret", "proprietary" or "company confidential". Confidential portions of documents otherwise non-confidential must be clearly identified. CBI may be purged of references to client identity by the responsible laboratory official at the time of removal from the laboratory. However, sample identifiers may not be obscured from the information. Additional information regarding CBI can be found in within the 2009 and 2016 TNI standards.

17.3 Audit Findings

Audit findings are documented using the corrective action process and database (see Section 14). The laboratory's corrective action responses may include action plans that could not be completed within a predefined timeframe. In these instances, a completion date must be set and agreed to by operations management and the QA Manager.

Developing and implementing corrective actions to findings is the responsibility of the Technical Manager where the finding originated. Findings that are not corrected by specified due dates are reported monthly to management in the QA monthly report.

If any audit finding casts doubt on the effectiveness of the operations or on the correctness or validity of the laboratory's test results, the laboratory shall take timely corrective action, and shall notify clients in writing if the investigations show that the laboratory results have been affected. Once corrective action is implemented, a follow-up audit is scheduled to ensure that the problem has been corrected.

Clients must be notified promptly in writing of any event such as the identification of defective measuring or test equipment that casts doubt on the validity of results given in any test report or amendment to a test report. The investigation must begin within 24 hours of discovery of the problem and all efforts are made to notify the client within two weeks after the completion of the investigation.

18.0 MANAGEMENT REVIEWS

18.1 Quality Assurance Report

The QA Department is responsible for preparing a comprehensive monthly metrics report to Management to keep them apprised of current quality issues. This report fosters

communication, review, and refinement of the QA system to evaluate the suitability of policies and procedures to meet both regulatory and laboratory quality objectives.

The NDSC QA team compiles information from all of the Environment Testing laboratories monthly metrics reports for the Executive Management team. This report includes notable information and concerns regarding the laboratories QA program and a listing of new regulations that may potentially impact the laboratories.

18.2 Annual Management Review

The Laboratory Management team (Laboratory Director, Technical Managers, QA Manager) conducts a review annually of its quality systems to ensure its continuing suitability and effectiveness in meeting client and regulatory requirements and to introduce any necessary changes or improvements. It will also provide a platform for defining goals, objectives and action items that feed into the laboratory planning system. NDSC personnel is be included in this meeting at the discretion of the Laboratory Director. The LIMS review consists of examining any audits, complaints or concerns that have been raised through the year that are related to LIMS. The laboratory will summarize any critical findings that cannot be solved by the lab and report them to Corporate

This management systems review NDSC Document No. CW-Q-S-004, Work Instruction No. CW-Q-WI-003 and local SOP No. ED-GEN-004, Quality Management Review uses information generated during the preceding year to assess the “big picture” by ensuring that routine actions taken and reviewed on a monthly basis are not components of larger systematic concerns. The monthly review should keep the quality systems current and effective, therefore, the annual review is a formal senior management process to review specific existing documentation. Significant issues from the following documentation are compiled or summarized by the QA Manager prior to the review meeting:

- Matters arising from the previous annual review.
- Prior Monthly QA Reports issues.
- Laboratory QA Metrics.
- Review of report reissue requests.
- Review of client feedback and complaints.
- Issues arising from any prior management or staff meetings.
- Minutes from prior senior lab management meetings. Issues that may be raised from these meetings include:
    - Adequacy of staff, equipment and facility resources.
    - Adequacy of policies and procedures.
    - Future plans for resources and testing capability and capacity.
- The annual internal double blind PT program sample performance (if performed),
- Compliance to the Ethics Policy and Data Integrity Plan. Including any evidence/incidents of inappropriate actions or vulnerabilities related to data Integrity.
- For labs analyzing radioactive samples, also include the following:
    - Radiation health and safety

- Radioactive hazardous waste management
- Radioactive materials management

- Evaluation of overall risk, including risks to impartiality, confidentiality, reporting statements of conformity, and nonconforming work.

A report is generated by the QA Manager and management. The report is distributed to the, President of the Business Unit, the Lab Director/Business Unit Manager, and Quality Director. The report includes, but is not limited to:

- The date of the review and the names and titles of participants.
- A reference to the existing data quality related documents and topics that were reviewed.
- Quality system or operational changes or improvements that will be made as a result of the review [e.g., an implementation schedule including assigned responsibilities for the changes (Action Table)].

Changes to the quality systems requiring update to the laboratory QA Manual shall be included in the next revision of the QA Manual.

18.3 Potential Integrity Related Managerial Reviews

Potential integrity issues (data or business related) must be handled and reviewed in a confidential manner until such time as a follow-up evaluation, full investigation, or other appropriate actions have been completed and issues clarified. The NDSC Document No. CW-L-S-002, Internal Investigations shall be followed. All investigations that result in finding of inappropriate activity are documented and include any disciplinary actions involved, corrective actions taken, and all appropriate notifications of clients.

The Eurofins Environment Testing America Presidents, Business Unit Managers, Laboratory Directors and NDSC Team are informed any current data integrity or data recall investigations.

19.0 TEST METHODS AND METHOD VALIDATION

19.1 Overview

The laboratory uses methods that are appropriate to meet our clients' requirements and that are within the scope of the laboratory's capabilities. These include sampling, handling, transport, storage and preparation of samples, and, where appropriate, an estimation of the measurement of uncertainty as well as statistical techniques for analysis of environmental data.

Instructions are available in the laboratory for the operation of equipment as well as for the handling and preparation of samples. All instructions, Standard Operating Procedures (SOPs), reference methods and manuals relevant to the working of the laboratory are readily available to all staff. Deviations from published methods are documented (with justification) in the laboratory's approved SOPs. SOPs are submitted to clients for review at their request. Significant deviations from published methods require client approval and regulatory approval where applicable.

19.2 Standard Operating Procedures (SOPs)

The laboratory maintains SOPs that accurately reflect all phases of the laboratory such as assessing data integrity, corrective actions, handling customer complaints as well as all analytical methods and sampling procedures. The method SOPs are derived from the most

recently promulgated/approved, published methods and are specifically adapted to the laboratory facility. Modifications or clarifications to published methods are clearly noted in the SOPs. All SOPs are controlled in the laboratory.

- All SOPs contain a revision number, effective date, and appropriate approval signatures. Controlled copies are available to all staff.
- Procedures for writing an SOP are incorporated by reference to NDSC Document No. CW-Q-S-002, Writing a Standard Operating Procedure.
- SOPs are reviewed at a minimum of every 2 years (annually for Drinking Water and DoD/DOE SOPs), and where necessary, revised to ensure continuing suitability and compliance with applicable requirements.

19.3 Laboratory Methods Manual

For each test method, the laboratory shall have available the published referenced method as well as the laboratory developed SOP.

Note: If more stringent standards or requirements are included in a mandated test method or regulation than those specified in this manual, the laboratory shall demonstrate that such requirements are met. If it is not clear which requirements are more stringent, the standard from the method or regulation is to be followed. Any exceptions or deviations from the referenced methods or regulations are noted in the specific analytical SOP.

The laboratory maintains an SOP Index for both technical and non-technical SOPs. Technical SOPs are maintained to describe a specific test method. Non-technical SOPs are maintained to describe functions and processes not related to a specific test method.

19.4 Selection of Methods

Since numerous methods and analytical techniques are available, continued communication between the client and laboratory is imperative to assure the correct methods are utilized. Once client methodology requirements are established, this and other pertinent information is summarized by the PM. These mechanisms ensure that the proper analytical methods are applied when the samples arrive for log-in. For non-routine analytical services (e.g., special matrices, non-routine compound lists), the method of choice is selected based on client needs and available technology. The methods selected should be capable of measuring the specific parameter of interest, in the concentration range of interest, and with the required precision and accuracy.

19.4.1 Sources of Methods

Routine analytical services are performed using standard EPA-approved methodology. In some cases, modification of standard approved methods may be necessary to provide accurate analyses of particularly complex matrices. When the use of specific methods for sample analysis is mandated through project or regulatory requirements, only those methods shall be used.

When clients do not specify the method to be used or methods are not required, the methods used will be clearly validated and documented in an SOP and available to clients and/or the end user of the data. Refer to Appendix 6 for a list of the currently accepted U.S. EPA analytical method references used by the laboratory.

The laboratory reviews updated versions to all the aforementioned references for adaptation based upon capabilities, instrumentation, etc., and implements them as appropriate. As such, the laboratory strives to perform only the latest versions of each approved method as regulations allow or require.

Other reference procedures for non-routine analyses may include methods established by specific states (e.g., Underground Storage Tank methods), ASTM or equipment manufacturers. Sample type, source, and the governing regulatory agency requiring the analysis will determine the method utilized.

The laboratory shall inform the client when a method proposed by the client may be inappropriate or out of date. After the client has been informed, and they wish to proceed contrary to the laboratory's recommendation, it will be documented.

19.4.1.1 Client Supplied Methods

Most of the client-supplied method requirements presented to us involve achieving specific quality control criteria, limits of quantitation (LOQ), and/or method detection limits (MDL) using standard EPA methods. These requirements are communicated to the appropriate technical groups prior to the project start up. Each technical group evaluates the scope of work and the requirements to ensure the criteria can be met using the standard EPA method. The data is monitored to ensure the criteria are met throughout the project. The PM notifies the client if there is a more appropriate method available or if the client's criteria cannot be achieved on a certain sample matrix (i.e., due to matrix or dilutions).

Occasionally, we are asked to transfer a non-standardized method from a client into our lab or to develop a new method, when one is not available. In the case of a method transfer, we set up the client's method and perform some initial evaluation. After the initial evaluation, we may make recommendations on how to improve method performance. If the method appears to be adequate, we determine linearity, specificity, precision, accuracy, MDL, and LOQ by performing calibrations, analyzing method blanks, and carrying out method detection limit and IDOC studies.

In the case of method development, we work with the client and/or data user to determine the level of validation required ensuring that the method meets its intended purpose. In addition to the elements above, we also determine standard and sample stability and robustness depending on the scope of the project. Typically, a standard operating procedure is written and submitted to the client with the results of the validation. These steps are completed prior to analysis of field samples. Data related to the setup of the method are archived.

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19.4.1.2 Procedural Deviations

Analysts are required to follow a documented method for all tests performed; and any deviations from analytical methods must be documented, approved, and justified in an appropriate and consistent manner. We classify method deviations as either being a planned deviation or an unplanned deviation. In general, the following information is captured to document both types of situations:

- Description of the situation
- Reason or justification for the deviation
- Impact the deviation had on the testing

- Signature/date of analyst performing the test
- Signature/date of QA and Laboratory management approving the deviation
- Signature/date of client approval, if necessary

Deviations to written procedures are documented in raw data records or through the ICAT system. Both types of documentation require management and QA review and approval.

19.4.2 Demonstration of Capability

Before the laboratory may institute a new method and begin reporting results, the laboratory shall confirm that it can properly operate the method. In general, this demonstration does not test the performance of the method in real world samples, but in an applicable and available clean matrix sample. If the method is for the testing of analytes that are not conducive to spiking, demonstration of capability may be performed on quality control samples.

A demonstration of capability (DOC) (Lab SOP ED-GEN-022, Training) is performed whenever there is a change in instrument type (e.g., new instrumentation), matrix, method or personnel (e.g., analyst has not performed the test within the last 12 months).

Note: The laboratory shall have a DOC for all analytes included in the methods that the laboratory performs, and proficiency DOCs for each analyst shall include all analytes that the laboratory routinely performs. Addition of non-routine analytes does not require new DOCs for all analysts if those analysts are already qualified for routine analytes tested using identical chemistry and instrument conditions.

The initial demonstration of capability must be thoroughly documented and approved by the Technical Manager and QA Manager prior to independently analyzing client samples. All associated documentation must be retained in accordance with the laboratory's archiving procedures.

The laboratory must have an approved SOP, demonstrate satisfactory performance, and conduct an MDL study (when applicable). There may be other requirements as stated within the published method or regulations (e.g., retention time window study).

Note: In some instances, a situation may arise where a client requests that an unusual analyte be reported using a method where this analyte is not normally reported. If the analyte is being reported for regulatory purposes, the method must meet all procedures outlined within this QA Manual (SOP, MDL, and Demonstration of Capability). If the client states that the information is not for regulatory purposes, the result may be reported as long as the following criteria are met:

- The instrument is calibrated for the analyte to be reported using the criteria for the method and ICV/CCV criteria are met (unless an ICV/CCV is not required by the method or criteria are per project DQOs).
- The laboratory's nominal or default reporting limit (RL) is equal to the quantitation limit (QL), must be at or above the lowest non-zero standard in the calibration curve and must be reliably determined. Project RLs are client specified reporting levels which may be higher than the QL. Results reported below the QL must be qualified as estimated values. Also see Section 19.6.1.3, Relationship of Limit of Detection (LOD) to Quantitation Limit (QL).
- The client request is documented and the lab informs the client of its procedure for working with unusual compounds. The final report must be footnoted: *Reporting Limit based on the low standard of the calibration curve.*

19.4.3 Initial Demonstration of Capability (IDOC) Procedures

Note: IDOC procedures are further detailed in Lab SOP ED-GEN-022, Training.

The spiking standard used must be prepared independently from those used in instrument calibration.

The analyte(s) shall be diluted in a volume of clean matrix sufficient to prepare four aliquots at the concentration specified by a method or the laboratory SOP.

19.4.3.1 At least four aliquots shall be prepared (including any applicable clean-up procedures) and analyzed according to the test method (either concurrently or over a period of days).

19.4.3.2 Using all of the results, calculate the mean recovery in the appropriate reporting units and the standard deviations for each parameter of interest.

19.4.3.3 When it is not possible to determine the mean and standard deviations, such as for presence, absence and logarithmic values, the laboratory will assess performance against criteria described in the Method SOP.

19.4.3.4 Compare the information obtained above to the corresponding acceptance criteria for precision and accuracy in the test method (if applicable) or in laboratory generated acceptance criteria (LCS or interim criteria) if there is no mandatory criteria established. If any one of the parameters do not meet the acceptance criteria, the performance is unacceptable for that parameter.

19.4.3.5 When one or more of the tested parameters fail at least one of the acceptance criteria, the analyst must proceed according to either option listed below:

- Locate and correct the source of the problem and repeat the test for all parameters of interest beginning with 19.4.3.3 above.
- Beginning with 19.4.3.3 above, repeat the test for all parameters that failed to meet criteria. Repeated failure, however, will confirm a general problem with the measurement system. If this occurs, locate and correct the source of the problem and repeat the test for all compounds of interest beginning with 19.4.3.1 above.

Note: Results of successive LCS analyses can be used to fulfill the DOC requirement.

Methods on line prior to the effective date of this Section shall be updated to the procedures outlined above as new analysts perform their demonstration of capability. A copy of the new record will replace that which was used for documentation in the past. At a minimum, the precision and accuracy of four mid-level laboratory control samples must have been compared to the laboratory's quality control acceptance limits.

19.5 Laboratory Developed Methods and Non-Standard Methods

Any new method developed by the laboratory must be fully defined in an SOP and validated by qualified personnel with adequate resources to perform the method. Method specifications and the relation to client requirements must be clearly conveyed to the client if the method is a non-standard method (not a published or routinely accepted method). The client must also be in agreement to the use of the non-standard method.

19.6 Validation of Methods

Validation is the confirmation by examination and the provision of objective evidence that the particular requirements for a specific intended use are fulfilled.

All non-standard methods, laboratory designed/developed methods, standard methods used outside of their scope, and major modifications to published methods must be validated to confirm they are fit for their intended use. The validation will be as extensive as necessary to meet the needs of the given application. The results are documented with the validation procedure used and contain a statement as to the fitness for use.

19.6.1 Method Validation and Verification Activities for All New Methods

While method validation can take various courses, the following activities can be required as part of method validation. Method validation records are designated QC records and are archived accordingly.

When changes are made to any validated methods, the influence of such changes shall be documented and, if appropriate, a new validation shall be performed.

19.6.1.1 **Determination of Method Selectivity** – Method selectivity is the demonstrated ability to discriminate the analyte(s) of interest from other compounds in the specific matrix or matrices from other analytes or interference. In some cases to achieve the required selectivity for an analyte, a confirmation analysis is required as part of the method.

19.6.1.2 **Determination of Method Sensitivity** – Sensitivity can be both estimated and demonstrated. Whether a study is required to estimate sensitivity depends on the level of method development required when applying a particular measurement system to a specific set of samples. Detection limit studies are conducted as described in Section 19.7. Where other protocols for estimations and/or demonstrations of sensitivity are required by regulation or client agreement, these shall be followed.

19.6.1.3 **Relationship of Limit of Detection (LOD) to the Limit of Quantitation (LOQ)** – An important characteristic of expression of sensitivity is the distinction between the LOD and the LOQ. The LOD is the minimum level at which the presence of an analyte can be reliably concluded. The LOQ is the minimum concentration of analyte that can be quantitatively determined with acceptable precision and bias, equivalent to the laboratory's routine reporting limit (RL). For most instrumental measurement systems, there is a region where semi-quantitative data is generated around the LOD (both above and below the estimated MDL or LOD) and below the LOQ. In this region, detection of an analyte may be confirmed but quantification of the analyte is unreliable within the accuracy and precision guidelines of the measurement system. When an analyte is detected below the LOQ, and the presence of the analyte is confirmed by meeting the qualitative identification criteria for the analyte, the analyte can be reliably reported, but the amount of the analyte can only be estimated. If data is to be reported in this region, it must be done so with a qualification that denotes the semi-quantitative nature of the result.

19.6.1.4 **Determination of Interferences** – A determination that the method is free from interferences in a blank matrix is performed.

19.6.1.5 **Determination of Range** – Where appropriate to the method, the quantitation range is determined by comparison of the response of an analyte in a curve to established or targeted criteria. Generally the upper quantitation limit is defined by highest acceptable calibration concentration. The lower quantitation limit or QL cannot be lower than the lowest non-zero calibration level, and can be constrained by required levels of bias and precision.

19.6.1.6 **Determination of Accuracy and Precision** – Accuracy and precision studies are generally performed using replicate analyses, with a resulting percent recovery and measure of

reproducibility (standard deviation, relative standard deviation) calculated and measured against a set of target criteria.

19.6.1.7 **Documentation of Method** – The method is formally documented in an SOP. If the method is a minor modification of a standard laboratory method that is already documented in an SOP, an SOP Attachment describing the specific differences in the new method is acceptable in place of a separate SOP.

19.6.1.8 **Continued Demonstration of Method Performance** – Continued demonstration of method performance is addressed in the SOP. Continued demonstration of method performance is generally accomplished by batch specific QC samples such as LCS, method blanks or PT samples.

19.7 Method Detection Limit (MDL) / Limits of Detection (LOD)

The MDL is the minimum measured quantity of a substance that can be reported with 99% confidence that the concentration is distinguishable from method blank results, consistent with 40CFR Part 136 Appendix B, August, 2017. The MDL is equivalent to the TNI LOD or DL, and is also equivalent to the DoD/DOE Quality Systems Manual (QSM) DL. The working or final MDL is the higher of the MDL value determined from spikes (MDLs) and the MDL value determined from blanks (MDLb). An initial MDL study shall be performed during the method validation process and when the method is altered in a way that can reasonably be expected to change its sensitivity. On-going data are collected during each quarter in which samples are being analyzed. At least once every 13 months the MDLs and MDLb are re-calculated and re-evaluated using data collected during the preceding period. Details of the laboratory's procedure for conducting MDL studies are given in NDSC Document No.CA-Q-S-006.

19.8 Verification of Detection Limits

If it is found during the re-evaluation of detection limit results that more than 5% of the spiked samples do not return positive numeric results that meet all method qualitative identification criteria, then then spiking level shall be increased and the initial MDL study pre-performed at the new spiking concentration..

19.9 Instrument Detection Limits (IDL)

The IDL is sometimes used to assess the reasonableness of the MDL or in some cases required by the analytical method or program requirements. IDLs are most commonly used in metals analyses but may be useful in demonstration of instrument performance in other areas.

IDLs are calculated to determine an instrument's sensitivity independent of any preparation method. IDLs are calculated either using 7 replicate spike analyses, like MDL but without sample preparation, or by the analysis of 10 instrument blanks and calculating 3 x the absolute value of the standard deviation.

19.10 Limit of Quantitation

The LOQ shall be at a concentration equivalent to the lowest calibration standard concentration, with the exception of methods using a single-point calibration, and shall be greater than the MDL. The LOQ is verified by preparing and analyzing spikes at concentrations 1-2X the selected LOQ, employing the complete analytical process.

When the laboratory establishes a quantitation limit, it must be initially verified by the analysis of a low level standard or QC sample at 1-2 times the reporting limit or by a DL check samples at or below the LOQ. The LOQ is verified annually thereafter. The annual requirement is waived for methods that have an annually verified MDL. The laboratory will comply with any regulatory requirements

19.11 Retention Time Windows

Most organic analyses and some inorganic analyses use chromatography techniques for qualitative and quantitative determinations. For every chromatography analysis or as specified in the reference method, each analyte will have a specific time of elution from the column to the detector. This is known as the analyte's retention time. The variance in the expected time of elution is defined as the retention time window. As the key to analyte identification in chromatography, retention time windows must be established on every column for every analyte used for that method. These records are kept on-file and available for review. Complete details are available in the laboratory SOPs.

19.12 Evaluation of Selectivity

The laboratory evaluates selectivity by following the checks within the applicable analytical methods, which include mass spectral tuning, second column confirmation, ICP interelement interference checks, chromatography retention time windows, sample blanks, spectrochemical, atomic absorption or fluorescence profiles, co-precipitation evaluations and specific electrode response factors.

19.13 Estimation of Uncertainty of Measurement

19.13.1 Uncertainty is "a parameter associated with the result of a measurement, that characterizes the dispersion of the values that could reasonably be attributed to the measurand" (as defined by the International Vocabulary of Basic and General Terms in Metrology, ISO Geneva, 1993, ISBN 92-67-10175-1). Knowledge of the uncertainty of a measurement provides additional confidence in a result's validity. Its value accounts for all the factors which could possibly affect the result, such as adequacy of analyte definition, sampling, matrix effects and interferences, climatic conditions, variances in weights, volumes, and standards, analytical procedure, and random variation. Some national accreditation organizations require the use of an "expanded uncertainty" defined as the range within which the value of the measurand is believed to lie within at least a 95% confidence level with the coverage factor k=2.

19.13.2 Uncertainty is not error. Error is a single value (i.e., the difference between the true result and the measured result). On environmental samples, the true result is never known. The measurement is the sum of the unknown true value and the unknown error. Unknown error is a combination of systematic error, or bias, and random error. Bias varies predictably, constantly, and independently from the number of measurements. Random error is unpredictable, assumed to be Gaussian in distribution, and reducible by increasing the number of measurements.

19.13.3 The minimum uncertainty associated with results generated by the laboratory can be determined by using the Laboratory Control Sample (LCS) accuracy range for a given analyte. The LCS limits are used to assess the performance of the measurement system since they take into consideration all of the laboratory variables associated with a given test over time (except for variability associated with the sampling and the variability due to matrix effects). The percent

recovery of the LCS is compared either to the method-required LCS accuracy limits or to the statistical, historical, in-house LCS accuracy limits.

19.13.4 To calculate the uncertainty for the specific result reported, multiply the result by the decimal of the lower end of the LCS range percent value for the lower end of the uncertainty range, and multiply the result by the decimal of the upper end of the LCS range percent value for the upper end of the uncertainty range. These calculated values represent uncertainties at approximately the 99% confidence level with a coverage factor of k = 3. As an example, for a reported result of 1.0 mg/L with an LCS recovery range of 50 to 150%, the estimated uncertainty in the result would be 1.0 +/- 0.5 mg/L.

19.13.5 In the case where a well-recognized test method specifies limits to the values of major sources of uncertainty of measurement (e.g., 524.2, 525, etc.) and specifies the form of presentation of calculated results, no further discussion of uncertainty is required.

19.14 Sample Reanalysis Guidelines

Because there is a certain level of uncertainty with any analytical measurement, a sample re-preparation (where appropriate) and subsequent analysis (hereafter referred to as 'reanalysis') may result in either a higher or lower value from an initial sample analysis. There are also variables that may be present (e.g., sample homogeneity, analyte precipitation over time, etc.) that may affect the results of a reanalysis. Based on the above comments, the laboratory will reanalyze samples at a client's request with the following caveats. **Client specific Contractual Terms & Conditions for reanalysis protocols may supersede the following items.**

Homogenous samples: If a reanalysis agrees with the original result to within the RPD limits for MS/MSD or Duplicate analyses, or within $\pm$ 1 reporting limit for samples $\leq$ 5x the reporting limit, the original analysis will be reported. At the client's request, both results may be reported on the same report but not on two separate reports.

- If the reanalysis does not agree (as defined above) with the original result, then the laboratory will investigate the discrepancy and reanalyze the sample a third time for confirmation if sufficient sample is available.
- Any potential charges related to reanalysis are discussed in the contract terms and conditions or discussed at the time of the request. The client will typically be charged for reanalysis unless it is determined that the lab was in error.
- Due to the potential for increased variability, reanalysis may not be applicable to Non-homogenous, Encore, and Sodium Bisulfate preserved samples. See the Area Supervisor or Laboratory Director if unsure.

19.15 Control of Data

The laboratory has policies and procedures in place to ensure the authenticity, integrity, and accuracy of the analytical data generated by the laboratory.

19.15.1 Computer and Electronic Data Related Requirements

The three basic objectives of our computer security procedures and policies are shown below.

The laboratory is currently using the Eurofins LIMS system, which has been highly customized to meet the needs of the laboratory.

19.15.1.1 Maintain the Database Integrity – Assurance that data is reliable and accurate through data verification (review) procedures, password-protecting access, anti-virus protection, data change requirements, as well as an internal LIMS permissions procedure.

- LIMS Database Integrity is achieved through data input validation, internal user controls, documentation of system failures and corrective actions taken, and data change requirements.
- Spreadsheets and other software developed in-house must be verified with documentation through hand calculations prior to use. Cells containing calculations must be lock-protected and controlled.
- Instrument hardware and software adjustments are safeguarded through maintenance logs, audit trails and controlled access.

19.15.1.2 Ensure Information Availability – Protection against loss of information or service is ensured through scheduled back-ups, stable file server network architecture, secure storage of media, line filter, Uninterruptible Power Supply (UPS), and maintaining older versions of software as revisions are implemented.

19.15.1.3 Maintain Confidentiality – Ensure data confidentiality through physical access controls such as password protection or website access approval when electronically transmitting data.

19.15.2 Data Reduction

The complexity of the data reduction depends on the analytical method and the number of discrete operations involved (e.g., extractions, dilutions, instrument readings and concentrations). The analyst calculates the final results from the raw data or uses appropriate computer programs to assist in the calculation of final reportable values.

For manual data entry, e.g., Wet Chemistry, the data is reduced by the analyst and then verified by the Department Manager or alternate analyst prior to updating the data in LIMS. The spreadsheets, or any other type of applicable documents, are signed by both the analyst and alternate reviewer to confirm the accuracy of the manual entry(s).

Manual integration of peaks will be documented and reviewed and the raw data will be flagged using NDSC Document No. CA-Q-S-002, Acceptable Manual Integration Practices.

Analytical results are reduced to appropriate concentration units specified by the analytical method, taking into account factors such as dilution, sample weight or volume, etc. Blank correction will be applied only when required by the method or per manufacturer's indication; otherwise, it should not be performed. Calculations are independently verified by appropriate laboratory staff. Calculations and data reduction steps for various methods are summarized in the respective analytical SOPs or program requirements.

19.15.2.1 All raw data must be retained in the worklist folder, computer file (if appropriate), and/or runlog. All criteria pertinent to the method must be recorded. The documentation is recorded at the time observations or calculations are made and must be signed or initialed/dated (month/day/year). It must be easily identifiable who performed which tasks if multiple people were involved.

19.15.2.2 In general, concentration results are reported in milligrams per liter (mg/l) or micrograms per liter (µg/l) for liquids and milligrams per kilogram (mg/kg) or micrograms per kilogram (µg/kg) for solids. For values greater than 10,000 mg/l, results can be reported in percent, i.e., 10,000 mg/l = 1**%.** Units are defined in each lab SOP.

19.15.2.3 In reporting, the analyst or the instrument output records the raw data result using values of known certainty plus one uncertain digit. If final calculations are performed external to LIMS, the results should be entered in LIMS with at least three significant figures. In general, results are reported to 2 significant figures on the final report.

19.15.2.4 For those methods that do not have an instrument printout or an instrumental output compatible with the LIMS, the raw results and dilution factors are entered directly into LIMS by the analyst, and the software calculates the final result for the analytical report. LIMS has a formatter for significant figure criterion for each analyte.

19.15.2.5 The laboratory strives to import data directly from instruments or calculation spreadsheets to ensure that the reported data are free from transcription and calculation errors. For those analyses with an instrumental output compatible with the LIMS, the raw results and dilution factors are transferred into LIMS, electronically after reviewing the quantitation report, and removing unrequested or poor spectrally-matched compounds. The analyst prints a copy of what has been entered to check for errors. This printout and the instrument's printout of calibrations, concentrations, retention times, chromatograms, and mass spectra, if applicable, are retained with the data file. The data file is stored in a monthly folder on the instrument computer; periodically, this file is transferred to the server and, eventually, to a tape file.

19.15.3 Logbook / Worksheet Use Guidelines

Logbooks and worksheets are filled out 'real time' and have enough information on them to trace the events of the applicable analysis/task. (e.g. calibrations, standards, analyst, sample ID, date, time on short holding time tests, temperatures when applicable, calculations are traceable, etc.)

- Corrections are made following the procedures outlined in Section 14.
- Logbooks are controlled by the QA department. A record is maintained of all logbooks in the lab.
- Unused portions of pages must be "Z"'d out, signed and dated.
- Worksheets are created with the approval of the Technical Manager/QA Manager at the facility. The QA Manager controls all worksheets following the procedures in Section 6.

19.15.4 Review / Verification Procedures

Review procedures are outlined in Lab SOP ED-GEN-021, Data Review to ensure that reported data are free from calculation and transcription errors, that QC parameters have been reviewed and evaluated before data is reported. The laboratory also utilizes NDSC Document No. CA-Q-S-002, Acceptable Manual Integration Practices to ensure the authenticity of the data . The general review concepts are discussed below, more specific information can be found in the SOPs.

19.15.4.1 Log-In Review - The data review process starts at the sample receipt stage. Sample control personnel review chain-of-custody forms and project instructions from the project management group. This is the basis of the sample information and analytical instructions entered into the LIMS. The log-in instructions are reviewed by the personnel entering the information, and a second level review is conducted by the project management staff.

19.15.4.2 First Level Data Review - The next level of data review occurs with the analysts. As data are generated, analysts review their work to ensure that the results meet project and SOP requirements. First level reviews include inspection of all raw data (e.g., instrument output for

continuous analyzers, chromatograms, spectra, and manual integrations), evaluation of calibration/calibration verification data in the day's analytical run, evaluation of QC data, and reliability of sample results. The analyst transfers data into LIMS, data qualifiers are added as needed. All first level reviews are documented.

19.15.4.3 Second Level Data Review – All analytical data are subject to review by a second qualified analyst or supervisor. Second level reviews include inspection of all raw data (e.g., instrument output, chromatograms, and spectra) including 100% of data associated with any changes made by the primary analyst, such as manual integrations or reassignment of peaks to different analytes, or elimination of false negative analytes. The second review also includes evaluation of initial calibration/calibration verification data in the day's analytical run, evaluation of QC data, reliability of sample results, qualifiers and NCM narratives. Manual calculations are checked in second level review. All second level reviews are documented.

Issues that deem further review include the following:

- QC data are outside the specified control limits for accuracy and precision
- Reviewed sample data does not match with reported results
- Unusual detection limit changes are observed
- Samples having unusually high results
- Samples exceeding a known regulatory limit
- Raw data indicating some type of contamination or poor technique
- Inconsistent peak integration
- Transcription errors
- Results outside of calibration range

19.15.4.4 Unacceptable analytical results may require reanalysis of the samples. Any problems are brought to the attention of the Laboratory Director, Project Manager, Quality Director/Manager, Technical Manager, or Supervisor for further investigation. Corrective action is initiated whenever necessary.

19.15.4.5 The results are then entered or directly transferred into the computer database and a .pdf is printed for the client.

19.15.4.6 As a final review prior to the release of the report, the Project Manager reviews the results for appropriateness and completeness. This review and approval ensures that client requirements have been met and that the final report has been properly completed. The process includes, but is not limited to, verifying that the COC is followed, cover letters / narratives are present, flags are appropriate, and project specific requirements are met. The Project Manager may also evaluate the validity of results for different test methods given expected chemical relationships.

19.15.4.7 Any project that requires a data package is subject to a tertiary data review for transcription errors and acceptable quality control requirements. The Project Manager then signs the final report. The accounting personnel also check the report for any clerical or invoicing errors. When complete, the report is sent out to the client.

19.15.4.8 A brief overview of sample flow and information through the laboratory, as well as data review and validation, is presented in Figure 19-1.

19.15.5 Manual Integrations

Computerized data systems provide the analyst with the ability to re-integrate raw instrument data in order to optimize the interpretation of the data. Though manual integration of data is an invaluable tool for resolving variations in instrument performance and some sample matrix problems, when used improperly, this technique would make unacceptable data appear to meet quality control acceptance limits. Improper re-integrations lead to legally indefensible data, a poor reputation, or possible laboratory decertification. Because guidelines for re-integration of data are not provided in the methods and most methods were written prior to widespread implementation of computerized data systems, the laboratory trains all analytical staff on proper manual integration techniques using NDSC Document No. CA-Q-S-002, Acceptable Manual Integration Practices.

19.15.5.1 The analyst must adjust baseline or the area of a peak in some situations, for example when two compounds are not adequately resolved or when a peak shoulder needs to be separated from the peak of interest. The analyst must use professional judgment to determine when manual integrating is required. Analysts are encouraged to ask for assistance from a senior analyst or manager when in doubt.

19.15.5.2 Analysts shall not increase or decrease peak areas for the sole purpose of achieving acceptable QC recoveries that would have otherwise been unacceptable. The intentional recording or reporting of incorrect information (or the intentional omission of correct information) is against company principles and policy and is grounds for immediate termination.

19.15.5.3 Client samples, performance evaluation samples, and quality control samples are all treated equally when determining whether or not a peak area or baseline should be manually adjusted.

19.15.5.4 All manual integrations receive a second level review. Manual integrations must be indicated on ~~an~~ expanded scale “before” and “after” chromatograms such that the integration performed can be easily evaluated during data review. Expanded scale “before” chromatograms are also required for all manual integrations on QC parameters (calibrations, calibration verifications, laboratory control samples, internal standards, surrogates, etc.) unless the laboratory has another documented NDSC approved procedure in place that can demonstrate an active process for detection and deterrence of improper integration practices.

Figure 19-1. Example – Work Flow

Action	Personnel Involved
Bottle orders generated upon request - Bottles packed and shipped to the client under CoC	- Client Service Personnel / PM - Bottle Preparation Personnel
Samples received at laboratory - Unpacked and reconciled against client paperwork or CoC - Samples acceptance documented against completed (CoC)[1]	Sample Login Personnel
Samples are entered into LIMS - Job # assigned - Methods/Analytes linked from project - Sample labels generated - LIMS backlogs updated immediately	Sample Login Personnel

Action	Personnel Involved
Sample Storage - Short TATs and hold-time samples are delivered directly to the laboratory for analysis - Samples stored in assigned locations (refrigerated walk-in coolers, freezers, VOA refrigerators; etc.)	Sample Login Personnel
Sample Receipt Acknowledgement sent to client (when requested)	Sample Login Personnel
Sample Acquisition/Testing - Analysts collect samples from assigned storage. - Legal CoC samples are signed in-and-out of storage - Remaining sample returned to storage.	Analysts / Login Personnel
Preparation and analysis is performed according to selected analytical method and applicable Project notes - Raw data recorded/reviewed[2] - Data imported to LIMS	Analysts
LIMS performs calculations: - Data reviewed by primary/secondary analyst - Data approved as final	Data Process (LIMS / Analyst)
Generation/review/release of reports (automated through LIMS) - Electronic copy saved in LIMS	Project Management Assistants / Reporting Group
Electronic Data Deliverables (EDDs) generated (as applicable)	EDD Group
Hard copy and electronic batch raw data is archived according to data archiving procedures.	Analysts / IT

[1]Refer to Sec. 23.3.
[2]Analyses requiring the analyst's interpretation may involve manual data reduction before entry into LIMS.

20.0 EQUIPMENT and CALIBRATIONS

20.1 Overview

The laboratory purchases the most technically advanced analytical instrumentation for sample analyses. Instrumentation is purchased on the basis of accuracy, dependability, efficiency and sensitivity. Each laboratory is furnished with all items of sampling, preparation, analytical testing and measurement equipment necessary to correctly perform the tests for which the laboratory has capabilities. Each piece of equipment is capable of achieving the required accuracy and complies with specifications relevant to the method being performed. Before being placed into use, the equipment (including sampling equipment) is calibrated and checked to establish that it meets its intended specification. The calibration routines for analytical instruments establish the range of quantitation. Calibration procedures are specified in laboratory SOPs. A list of available laboratory instrumentation is presented in Table 20-1.

Equipment is only operated by authorized and trained personnel. Manufacturer's instructions for equipment use are readily accessible to all appropriate laboratory personnel.

20.2 Preventive Maintenance

The laboratory follows a well-defined maintenance program to ensure proper equipment operation and to prevent the failure of laboratory equipment or instrumentation during use. This program of preventive maintenance helps to avoid delays due to instrument failure.

Routine preventive maintenance procedures and frequency, such as cleaning and replacements, should be performed according to the procedures outlined in the manufacturer's manual. Qualified personnel must also perform maintenance when there is evidence of degradation of peak resolution, a shift in the calibration curve, loss of sensitivity, or failure to continually meet one of the quality control criteria.

Scheduled routine maintenance is defined in each method SOP. It is the responsibility of each Technical Manager to ensure that instrument maintenance logs are kept for all equipment in his/her department. Preventative maintenance procedures are also outlined in analytical SOPs or instrument manuals. (Note: for some equipment, the log used to monitor performance is also the maintenance log. Multiple pieces of equipment may share the same log as long as it is clear as to which instrument is associated with an entry.)

Instrument maintenance logs are controlled and are used to document instrument problems, instrument repair and maintenance activities. Maintenance logs shall be kept for all major pieces of equipment. Instrument maintenance logs may also be used to specify instrument parameters.

- Documentation must include all major maintenance activities such as contracted preventive maintenance and service and in-house activities such as the replacement of electrical components, lamps, tubing, valves, columns, detectors, cleaning and adjustments.
- Each entry in the instrument log includes the analyst's initials, the date, a detailed description of the problem (or maintenance needed/scheduled), a detailed explanation of the solution or maintenance performed, and a verification that the equipment is functioning properly (state what was used to determine a return to control. e.g. CCV run on *'date'* was acceptable, or instrument recalibrated on 'date' with acceptable verification, etc.) must also be documented in the instrument records.
- When maintenance or repair is performed by an outside agency, service receipts detailing the service performed can be affixed into the logbooks adjacent to pages describing the maintenance performed. This stapled in page must be signed across the page entered and the logbook so that it is clear that a page is missing if only half a signature is found in the logbook.

If an instrument requires repair (subjected to overloading or mishandling), gives suspect results, or otherwise has shown to be defective or outside of specified limits it shall be taken out of operation and tagged as out-of-service or otherwise isolated until such a time as the repairs have been made and the instrument can be demonstrated as operational by calibration and/or verification or other test to demonstrate acceptable performance. The laboratory shall examine the effect of this defect on previous analyses.

In the event of equipment malfunction that cannot be resolved, service shall be obtained from the instrument vendor manufacturer, or qualified service technician, if such a service can be tendered. If on-site service is unavailable, arrangements shall be made to have the instrument shipped back to the manufacturer for repair. Back up instruments, which have been approved, for the analysis shall perform the analysis normally carried out by the malfunctioning instrument. If the back-up is not available and the analysis cannot be carried out within the needed timeframe, the samples shall be subcontracted.

At a minimum, if an instrument is sent out for service or transferred to another facility, it must be recalibrated and the laboratory MDL verified (using an MDLV) prior to return to lab operations.

20.3 Support Equipment

This section applies to all devices that may not be the actual test instrument, but are necessary to support laboratory operations. These include but are not limited to: balances, ovens, refrigerators, freezers, incubators, water baths, field sampling devices, temperature measuring devices, thermal/pressure sample preparation devices and volumetric dispensing devices if quantitative results are dependent on their accuracy, as in standard preparation and dispensing or dilution into a specified volume. All raw data records associated with the support equipment are retained to document instrument performance.

20.3.1 Weights and Balances

The accuracy of the balances used in the laboratory is checked every working day, before use. All balances are placed on stable counter tops.

Each balance is checked prior to initial serviceable use with at least two certified ASTM type 1 weights spanning its range of use (weights that have been calibrated to ASTM type 1 weights may also be used for daily verification). ASTM type 1 weights used only for calibration of other weights (and no other purpose) are inspected for corrosion, damage or nicks at least annually and if no damage is observed, they are calibrated at least every 5 years by an outside calibration laboratory. Any weights (including ASTM Type 1) used for daily balance checks or other purposes are recalibrated/recertified annually to NIST standards (this may be done internally if laboratory maintains “calibration only” ASTM type 1 weights).

All balances are serviced annually by a qualified service representative, who supplies the laboratory with a certificate that identifies traceability of the calibration to the NIST standards.

All of this information is recorded in logs, and the recalibration/recertification certificates are kept on file as detailed in lab SOP ED-GEN-010, Calibration of Analytical Balances.

20.3.2 pH, Conductivity, and Turbidity Meters

The pH meters used in the laboratory are accurate to ± 0.1 pH units, and have a scale readability of at least 0.05 pH units. The meters automatically compensate for the temperature, and are calibrated with at least two working range buffer solutions before each use.

Conductivity meters used in the laboratory are capable or measuring conductivity with an error not exceeding 1% or one umhos/cm, whichever is greater. The meters are also calibrated before each use with a known standard.

Turbidity meters are also calibrated before each use. All of this information is documented in logs.

Consult pH, Conductivity, and Turbidity SOPs for further information.

20.3.3 Thermometers

All thermometers are calibrated on an annual basis with a NIST-traceable thermometer.

- If the temperature measuring device is used over a range of 10°C or less, then a single point verification within the range of use is acceptable;
- If the temperature measuring device is used over a range of greater than 10°C, then the verification must bracket the range of use.

IR thermometers, digital probes and thermocouples are calibrated quarterly. IR Thermometers should be calibrated over the full range of use, including ambient, iced (4°C) and frozen (0°C to -5°C), per the Drinking Water Manual.

The digital NIST thermometer is recalibrated every five years (unless the thermometer has been exposed to temperature extremes or apparent separation of internal liquid) by an approved outside service and the provided certificate of traceability is kept on file. The NIST thermometer(s) have increments of 1 degree (0.5 degree or less increments are required for drinking water microbiological laboratories), and have ranges applicable to method and certification requirements. The NIST traceable thermometer is used for no other purpose than to calibrate other thermometers.

All of this information is documented in logbooks. Monitoring method-specific temperatures, including incubators, heating blocks, water baths, and ovens, is documented in method-specific logbooks. More information on this subject can be found in the lab SOP ED-GEN-014, Thermometer Calibration..

20.3.4 Refrigerators/Freezer Units, Water baths, Ovens and Incubators

The temperatures of all refrigerator units and freezers used for sample and standard storage are monitored each working day.

Ovens, water baths and incubators are monitored on days of use.

All of this equipment has a unique identification number, and is assigned a thermometer for monitoring.

Sample storage refrigerator temperatures are kept between > 0ºC and ≤ 6 ºC.

Specific temperature settings/ranges for other refrigerators, ovens, water baths, and incubators can be found in method specific SOPs.

All of this information is documented in Daily Temperature Logbooks and method-specific logbooks.

20.3.5 Autopipettors, Dilutors, and Syringes

Mechanical volumetric dispensing devices including burettes (except Class A Glassware and glass microliter syringes) are given unique identification numbers and the delivery volumes are verified gravimetrically, at a minimum, on a quarterly basis.

For those dispensers that are not used for measurements, a label shall be applied to the device stating that it is not calibrated. Any device not regularly verified cannot be used for any quantitative measurements. Lab SOP ED-GEN-011, Calibration and Use of Lab Pipettes, contains detailed information on this topic.

Micro-syringes are purchased from Hamilton Company. Each syringe is traceable to NIST. The laboratory keeps on file an “Accuracy and Precision Statement of Conformance” from Hamilton attesting established accuracy.

20.3.6 Autoclaves

Section reserved. Autoclaves are not currently in use at Eurofins Edison.

20.3.7 Field Sampling Devices (Isco Auto Samplers)

Each Auto Sampler (ISCO) is assigned a unique identification number in order to keep track of the calibration. This number is also recorded on the sampling documentation.

The Auto Sampler is calibrated monthly by setting the sample volume to 100ml and recording the volume received. The results are filed in a logbook/binder. The Auto Sampler is programmed to run three (3) cycles and each of the three cycles is measured into a graduated cylinder to verify 100ml are received.

If the RSD (Relative Standard Deviation) between the 3 cycles is greater than 10%, the procedure is repeated and if the result is still greater than 10%, then the Auto Sampler is taken out of service until it is repaired and calibration verification criteria can be met. The results of this check are kept in a logbook/binder.

20.4 Instrument Calibrations

Calibration of analytical instrumentation is essential to the production of quality data. Strict calibration procedures are followed for each method. These procedures are designed to determine and document the method detection limits, the working range of the analytical instrumentation and any fluctuations that may occur from day to day.

Sufficient raw data records are retained to allow an outside party to reconstruct all facets of the initial calibration. Records contain, but are not limited to, the following: calibration date, method, instrument, analyst(s) initials or signatures, analysis date, analytes, concentration, response, and type of calibration (Avg RF, curve, or other calculations that may be used to reduce instrument responses to concentration.)

Each method that requires a standard curve must have that curve evaluated for Relative Standard Error as detailed in Eurofins document NDSC-QA-QP44940, Calibration Curves and the Selection of Calibration Points. The procedure for evaluating Relative Standard Error is documented in each applicable SOP.

Sample results must be quantitated from the initial calibration and may not be quantitated from any continuing instrument calibration verification unless otherwise required by regulation, method or program.

If the initial calibration results are outside of the acceptance criteria, corrective action is performed and any affected samples are reanalyzed if possible. If the reanalysis is not possible, any data associated with an unacceptable initial calibration will be reported with appropriate data qualifiers (refer to Section 14).

Note: Instruments are calibrated initially and as needed after that and at least annually (the annual requirement does not apply to Isotope dilution).

20.4.1 Calibration Standards

Calibration standards are prepared using the procedures indicated in the Reagents and Standards section of the determinative method SOP. If a reference method does not specify the number of calibration standards, a minimum of 3 calibration points will be used.

Standards for instrument calibration are obtained from a variety of sources. All standards are traceable to national or international standards of measurement, or to national or international standard reference materials.

The lowest concentration calibration standard that is analyzed during an initial calibration must be at or below the stated reporting limit for the method based on the final volume of extract (or sample).

The other concentrations define the working range of the instrument/method or correspond to the expected range of concentrations found in actual samples that are also within the working range of the instrument/method. Results of samples not bracketed by initial instrument calibration standards (within calibration range to at least the same number of significant figures used to report the data) must be reported as having less certainty, e.g., defined qualifiers or flags (additional information may be included in the case narrative). The exceptions to these rules. ICP and ICPMS methods which define the working range with periodic linear dynamic range studies, rather than through the range of concentrations of daily calibration standards.

All initial calibrations are verified with a standard obtained from a second source and traceable to a national standard, when available (or vendor certified different lot if a second source is not available). For unique situations, such as air analysis where no other source or lot is available, a standard made by a different analyst at a different time or a different preparation would be considered a second source. This verification occurs immediately after the calibration curve has been analyzed, and before the analysis of any samples.

20.4.1.1 Calibration Verification

The calibration relationship established during the initial calibration must be verified initially and at least daily as specified in the laboratory method SOPs in accordance with the referenced analytical methods and in the 2009 and 2016 TNI Standard. The process of calibration verification applies to both external standard and internal standard calibration techniques, as well as to linear and non-linear calibration models. Initial calibration verification (ICV) is with a standard source secondary (second source standard) to the calibration standards, but continuing calibration verifications (CCV) may use the same source standards as the calibration curve.

Note: The process of calibration verification referred to here is fundamentally different from the approach called "calibration" in some methods. As described in those methods, the calibration factors or response factors calculated during calibration are used to update the calibration factors or response factors used for sample quantitation. This approach, while employed in other EPA programs, amounts to a daily single-point calibration.

All target analytes and surrogates, including those reported as non-detects, must be included in periodic calibration verifications for purposes of retention time confirmation and to demonstrate that calibration verification criteria are being met, i.e., RPD, per 2009 and 2016 TNI Std. EL-V1M4 Sec. 1.7.2.

All samples must be bracketed by periodic analyses of standards that meet the QC acceptance criteria (e.g., calibration and retention time). The frequency is found in the determinative methods or SOPs.

Note: If an internal standard calibration is being used then bracketing calibration verification standards may not be required, only daily verifications are needed. The results from these verification standards must meet the calibration verification criteria and the retention time criteria (if applicable).

Generally, the calibrations must be verified by an ICV analyzed immediately following initial calibration and before sample analysis. The ICV may be used as the first bracketing CCV, if criteria for both are met.

A continuing instrument calibration verification (CCV) is generally analyzed at the beginning of each 12-hour analytical shift during which samples are analyzed. The 12-hour analytical shift begins with the injection of the calibration verification standard (or the MS tuning standard in MS methods). The shift ends after the completion of the analysis of the last sample, QC, or standard that can be injected within 12-hours of the beginning of the shift. For methods that have quantitation by external calibration models, a CCV is analyzed at the end of each analytical sequence. Some methods have more frequent CCV requirements. See specific SOPs. Most inorganic methods require the CCV to be analyzed after ever 10 samples or injections, including matrix or batch QC samples.

Note: If an internal standard calibration is being used (e.g., GCMS) then bracketing standards are not required, only daily verifications are needed, except as specified by program or method requirements.

If the results of a CCV are outside the established acceptance criteria and analysis of a second consecutive (and immediate) CCV fails to produce results within acceptance criteria, corrective action shall be performed. Once corrective actions have been completed and documented, the laboratory shall demonstrate acceptable instrument / method performance by analyzing two consecutive CCVs, or a new initial instrument calibration shall be performed.

Sample analyses and reporting of data may not occur or continue until the analytical system is calibrated or calibration verified. However, data associated with an unacceptable calibration verification may be fully useable and **reported based upon discussion and approval of the client** under the following special conditions:

a). when the acceptance criteria for the CCV are exceeded high (i.e., high bias) and the associated samples within the batch are non-detects, then those non-detects may be reported case narrative comment explaining the high bias. Otherwise the samples affected by the unacceptable CCV shall be re-analyzed after a new calibration curve has been established, evaluated and accepted; or

b). when the acceptance criteria for the CCV are exceeded low (i.e., low bias), those sample results may be reported if they exceed a maximum regulatory limit/decision level. Otherwise the samples affected by the unacceptable CCV shall be re-analyzed after a new calibration curve has been established, evaluated and accepted.

Samples reported by the 2 conditions identified above will be appropriately flagged.

20.4.1.2 Verification of Linear and Non-Linear Calibrations

Calibration verification for calibrations involves the calculation of the percent drift or the percent difference of the instrument response between the initial calibration and each subsequent analysis of the verification standard. (These calculations are available in the laboratory method SOPs.) Verification standards are evaluated based on the % Difference from the average CF or RF of the initial calibration or based on % Drift or % Recovery if a linear or quadratic curve is used.

Regardless of whether a linear or non-linear calibration model is used, if initial verification criterion is not met, then no sample analyses may take place until the calibration has been verified or a new initial calibration is performed that meets the specifications listed in the method SOPs. If the calibration cannot be verified after the analysis of a single verification standard, then adjust the instrument operating conditions and/or perform instrument maintenance, and analyze another aliquot of the verification standard. If the calibration cannot be verified with the second standard, then a new initial calibration is performed.

- When the acceptance criteria for the calibration verification are exceeded high, i.e., high bias, and there are associated samples that are non-detects, then those non-detects may be reported. Otherwise, the samples affected by the unacceptable calibration verification shall be reanalyzed after a new calibration curve has been established, evaluated and accepted.
- When the acceptance criteria for the calibration verification are exceeded low, i.e., low bias, those sample results may be reported if they exceed a maximum regulatory limit/decision level. Otherwise, the samples affected by the unacceptable verification shall be reanalyzed after a new calibration curve has been established, evaluated and accepted. Alternatively, a reporting limit standard may be analyzed to demonstrate that the laboratory can still support non-detects at their reporting limit.

20.5 Tentatively Identified Compounds (TICs) – GC/MS Analysis

For samples containing components not associated with the calibration standards, a library search may be made for the purpose of tentative identification. The necessity to perform this type of identification will be determined by the purpose of the analyses being conducted. For example, the RCRA permit or waste delisting requirements may require the reporting of non-target analytes. Only after visual comparison of sample spectra with the nearest library searches may the analyst assign a tentative identification. Data system library search routines should not use normalization routines that would misrepresent the library or unknown spectra when compared to each other. Additional details are provided in NDSC Document No. CA-Q-QM-001, Tentatively Identified Compounds (TICS) - GCMS Analysis.

Note: If the TIC compound is not part of the client target analyte list but is calibrated by the laboratory and is both qualitatively and/or quantitatively identifiable, it should not be reported as a TIC. If the compound is reported on the same form as true TICs, it should be qualified and/or narrated that the reported compound is qualitatively and quantitatively (if verification in control) reported compared to a known standard that is in control (where applicable).

20.6 GC/MS Tuning

Prior to any GC/MS analytical sequence, including calibration, the instrument parameters for the tune and subsequent sample analyses within that sequence must be set.

Prior to tuning/auto-tuning the mass spectrometer, the parameters may be adjusted within the specifications set by the manufacturer or the analytical method. These generally do not need any adjustment but it may be required based on the current instrument performance. If the tune verification does not pass it may be necessary to clean the source or perform additional maintenance. Any maintenance is documented in the maintenance log.

Table 20-1

Eurofins Edison– Instrumentation by Type

(see EDS-WI-002, Eurofins Edison Equipment List for full current listing)

GC	GC/MS	ICP	ICP-MS	Hg Analyzer	Auto Analyzer	IC	TOC	Spectrophoto meter
x	x	X	x	x	x	x	x	x

21.0 MEASUREMENT TRACEABILITY

21.1 Overview

Traceability of measurements shall be assured using a system of documentation, calibration, and analysis of reference standards. Laboratory equipment that are peripheral to analysis and whose calibration is not necessarily documented in a test method analysis or by analysis of a reference standard shall be subject to ongoing certifications of accuracy. At a minimum, these must include procedures for checking specifications of ancillary equipment: balances, thermometers, temperature, Deionized (DI) and Reverse Osmosis (RO) water systems, automatic pipettes and other volumetric measuring devices. (Refer to Section 20.3). With the exception of Class A Glassware and glass microliter syringes, quarterly accuracy checks are performed for all mechanical volumetric devices. Wherever possible, subsidiary or peripheral equipment is checked against standard equipment or standards that are traceable to national or international standards. Class A Glassware and glass microliter syringes should be routinely inspected for chips, acid etching or deformity (e.g., bent needle). If the Class A glassware or syringe is suspect, the accuracy of the glassware will be assessed prior to use.

21.2 NIST-Traceable Weights and Thermometers

Reference standards of measurement shall be used for calibration only and for no other purpose, unless it can be shown that their performance as reference standards would not be invalidated.

For NIST-traceable weights and thermometers, the laboratory requires that all calibrations be conducted by a calibration laboratory accredited by A2LA, NVLAP (National Voluntary Laboratory Accreditation Program), or another accreditation organization that is a signatory to a MRA (Mutual Recognition Arrangement) of one or more of the following cooperations – ILAC (International Laboratory Accreditation Cooperation) or APLAC (Asia–Pacific Laboratory Accreditation Cooperation). A calibration certificate and scope of accreditation is kept on file at the laboratory.

21.3 Reference Standards / Materials

Reference standards/materials, where commercially available, are traceable to certified reference materials. Commercially prepared reference standards, to the extent available, are purchased from vendors that are accredited to ISO Guide 34 and ISO/IEC Guide 17025. All reference standards from commercial vendors shall be accompanied with a certificate that includes at least the following information:

- Manufacturer
- Analytes or parameters calibrated
- Identification or lot number
- Calibration method
- Concentration with associated uncertainties
- Purity

If a standard cannot be purchased from a vendor that supplies a Certificate of Analysis, the purity of the standard is documented by analysis. The receipt of all reference standards must be documented. Reference standards are labeled with a unique Standard Identification Number

and expiration date. All documentation received with the reference standard is retained as a QC record and references the Standard Identification Number.

All reference, primary and working standards/materials, whether commercially purchased or laboratory prepared, must be checked regularly to ensure that the variability of the standard or material from the true value does not exceed method requirements. The accuracy of calibration standards is checked by comparison with a standard from a second source. In cases where a second standard manufacturer is not available, a vendor certified different lot is acceptable for use as a second source. For unique situations, such as air analysis where no other source or lot is available, a standard made by a different analyst would be considered a second source. The appropriate Quality Control (QC) criteria for specific standards are defined in laboratory SOPs. In most cases, the analysis of an Initial Calibration Verification (ICV) or LCS (where there is no sample preparation) is used as the second source confirmation. These checks are generally performed as an integral part of the analysis method (e.g. calibration checks, laboratory control samples).

All standards and materials must be stored and handled according to method or manufacturer's requirements in order to prevent contamination or deterioration. Refer to the–Environmental Health & Safety Manual or laboratory SOPs. For safety requirements, please refer to method SOPs and the laboratory's Environmental Health and Safety Manual.

Standards and reference materials shall not be used after their expiration dates unless their reliability is verified by the laboratory and their use is approved by the Quality Assurance Manager. The laboratory must have documented contingency procedures for re-verifying expired standards.

21.4 Documentation and Labeling of Standards, Reagents, and Reference Materials

Reagents must be at a minimum the purity required in the test method. The date of reagent receipt and the expiration date are documented. The lots for most of the common solvents and acids are tested for acceptability prior to company-wide purchase. [Refer to NDSC Document No. (CA-Q-S-001) Solvent and Acid Lot Testing and Approval]

All manufacturer or vendor supplied Certificate of Analysis or Purity must be retained, stored appropriately, and readily available for use and inspection. These records are maintained [in the TALS Reagent Module. Records must be kept of the date of receipt and date of expiration of standards, reagents and reference materials. In addition, records of preparation of laboratory standards, reagents, and reference materials must be retained, stored appropriately, and be readily available for use and inspection. For detailed information on documentation and labeling, please refer to method specific SOPs.

Commercial materials purchased for preparation of calibration solutions, spike solutions, etc.., are usually accompanied with an assay certificate or the purity is noted on the label. If the assay purity is 96% or better, the weight provided by the vendor may be used without correction. If the assay purity is less than 96% a correction will be made to concentrations applied to solutions prepared from the stock commercial material. Blended gas standard cylinders use a nominal concentration if the certified value is within +/-15%, otherwise the certified values is used for the canister concentration.

21.4.1 All standards, reagents, and reference materials must be labeled in an unambiguous manner. Standards are logged into the laboratory's LIMS, and are assigned a unique identification number. The following information is typically recorded in the electronic database within the LIMS.

- Standard ID
- Description of Standard
- Department
- Preparer's name
- Final volume and number of vials prepared
- Solvent type and lot number
- Preparation Date
- Expiration Date
- Standard source type (stock or daughter)
- Standard type (spike, surrogate, other)
- Parent standard ID (if applicable)
- Parent Standard Analyte Concentration (if applicable)
- Parent Standard Amount used (if applicable)
- Component Analytes
- Final concentration of each analyte
- Comment box (text field)

Records are maintained electronically for standard and reference material preparation. These records show the traceability to purchased stocks or neat compounds. These records also include method of preparation, date of preparation, expiration date and preparer's name or initials. Preparation procedures are provided in the Method SOPs.

21.4.2 All standards, reagents, and reference materials must be clearly labeled with a minimum of the following information:

- Expiration Date (include prep date for reagents)
- Standard ID (from LIMS)
- Special Health/Safety warnings if applicable

Records must also be maintained of the date of receipt for commercially purchased items or date of preparation for laboratory prepared items. Special Health/Safety warnings must also be available to the analyst. This information is maintained in the TALs Reagent Module.

21.4.3 In addition, the following information may be helpful:

- Date opened (for multi-use containers, if applicable)
- Description of standard (if different from manufacturer's label or if standard was prepared in the laboratory)
- Recommended Storage Conditions
- Concentration (if applicable)
- Initials of analyst preparing standard or opening container

All containers of prepared reagents must include an expiration date and an ID number to trace back to preparation.

Procedures for preparation of reagents can be found in the Method SOPs.

Standard ID numbers must be traceable through associated logbooks, worksheets and preparation/analytical batch records.

All reagents and standards must be stored in accordance to the following priority: 1) with the manufacturer's recommendations; 2) with requirements in the specific analytical methods as specified in the laboratory SOP.

22.0 SAMPLING

22.1 Overview

The laboratory provides sampling services. Sampling procedures are described in the following SOPs:

- ED-FLD-008 (Low Flow Well Purging & Sampling)
- ED-FLD-009 (Volume Average Purging & Sampling)
- ED-FLD-014 (Wastewater Sampling)

22.2 Sampling Containers

The laboratory offers clean sampling containers for use by clients. These containers are obtained from reputable container manufacturers and meet EPA specifications as required. Certificates of cleanliness for bottles and preservatives are provided by the supplier and are maintained at the laboratory. Alternatively, the certificates may be maintained by the supplier and available to the laboratory on-line.

22.2.1 Preservatives

Upon request, preservatives are provided to the client in pre-cleaned sampling containers. In some cases containers may be purchased pre-preserved from the container supplier. Whether prepared by the laboratory or bought pre-preserved, the grades of the preservatives are at a minimum:

- Hydrochloric Acid – Reagent ACS (Certified VOA Free) or equivalent
- Methanol – Purge and Trap grade
- Nitric Acid – Instra-Analyzed or equivalent
- Sodium Bisulfate – ACS Grade or equivalent
- Sodium Hydroxide – Instra-Analyzed or equivalent
- Sulfuric Acid – Instra-Analyzed or equivalent
- Sodium Thiosulfate – ACS Grade or equivalent

22.3 Definition of Holding Time

The date and time of sampling documented on the COC form establishes the day and time zero. As a general rule, when the maximum allowable holding time is expressed in days (e.g., 14 days, 28 days), the holding time is based on calendar day measured. Holding times expressed in hours (e.g., 6 hours, 24 hours, etc.) are measured from date and time zero. Holding times for

analysis include any necessary reanalysis. However, there are some programs that determine holding time compliance based on the date and specific time of analysis compared to the time of sampling regardless of how long the holding time is.

22.4 Sampling Containers, Preservation Requirements, Holding Times

The preservation and holding time criteria specified in the laboratory SOPs are derived from the source documents for the methods. If method required holding times or preservation requirements are not met, the reports will be qualified using a flag, footnote or case narrative. As soon as possible or “ASAP” is an EPA designation for tests for which rapid analysis is advised, but for which neither EPA nor the laboratory have a basis for a holding time.

22.5 Sample Aliquots / Subsampling

Taking a representative sub-sample from a container is necessary to ensure that the analytical results are representative of the sample collected in the field. The size of the sample container, the quantity of sample fitted within the container, and the homogeneity of the sample need consideration when sub-sampling for sample preparation. It is the laboratory’s responsibility to take a representative subsample or aliquot of the sample provided for analysis.

Analysts should handle each sample as if it is potentially dangerous. At a minimum, safety glasses, gloves, and lab coats must be worn when preparing aliquots for analysis.

Guidelines on taking sample aliquots and subsampling are located in lab SOP ED-GEN-007, Subsampling.

23.0 HANDLING OF SAMPLES

It is the responsibility of the client to send us representative and/or homogeneous and properly preserved samples of the system from which they are drawn. The laboratory assumes that all multiple sample containers with the same designator/description and bottle type contain a homogeneous, representative sample.

The laboratory provides the appropriate sample containers, required preservative, chain-of-custody (COC) forms, shipping containers, labels, and custody seals. The laboratory also provides trip blanks and analyte-free water for field blanks. Preparation of methanol containers for field preservation of volatile soil samples is available.

Sample management procedures at the laboratory ensure that sample integrity and custody are maintained and documented from sampling/receipt through disposal.

23.1 Chain of Custody (COC)

The COC form is the written documented history of any sample and is initiated when bottles are sent to the field, or at the time of sampling. This form is completed by the sampling personnel and accompanies the samples to the laboratory where it is received and stored under the laboratory’s custody. The purpose of the COC form is to provide a legal written record of the handling of samples from the time of collection until they are received at the laboratory. It also serves as the primary written request for analyses from the client to the laboratory. The COC form acts as a purchase order for analytical services when no other contractual agreement is in effect.

23.1.1 Field Documentation

The information the sampler needs to provide at the time of sampling on the container label is:

- Sample identification
- Date and time
- Preservative

During the sampling process, the COC form is completed and must be legible. This form includes information such as:

- Client name, address, phone number and fax number (if available)
- Project name and/or number
- The sample identification
- Date, time and location of sampling
- Sample collectors name
- The matrix description
- The container description
- The total number of each type of container
- Preservatives used
- Analysis requested
- Requested turnaround time (TAT)
- Any special instructions
- Purchase Order number or billing information (e.g. quote number) if available
- The date and time that each person received or relinquished the sample(s), including their signed name.

When the sampling personnel deliver the samples directly to the laboratory personnel, the samples are stored in a cooler with ice, as applicable, and remain solely in the possession of the client's field technician until the samples are delivered to the laboratory personnel. The sample collector must assure that each container is in his/her physical possession or in his/her view at all times, or stored in such a place and manner to preclude tampering. The field technician relinquishes the samples in writing on the COC form to the sample control personnel at the laboratory or to a laboratory courier. When sampling personnel deliver the samples through a common carrier (Fed-Ex, UPS), the CoC relinquished date/time is completed by the field personnel and samples are released to the carrier. Samples are only considered to be received by the laboratory when personnel at the fixed laboratory facility have physical contact with the samples.

Note: Independent couriers are not required to sign the COC form. The COC is usually kept in the sealed sample cooler. The receipt from the courier is stored in log-in by date; it lists all receipts each date

23.1.2 Legal / Evidentiary Chain-of-Custody

If samples are identified for legal/evidentiary purposes on the COC, login will complete the custody seal retain the shipping record with the COC, and initiate an internal COC for laboratory use by analysts and a sample disposal record.

23.2 Sample Receipt

Samples are received at the laboratory by designated sample receiving personnel and a unique laboratory project identification number is assigned. Each sample container shall be assigned a unique sample identification number that is cross-referenced to the client identification number such that traceability of test samples is unambiguous and documented. Each sample container is affixed with a durable sample identification label. Sample acceptance, receipt, tracking and storage procedures are detailed in the laboratory's SOP No. ED-SPM-001, *Procedure For Sample Receipt, Login, Identification, And Storage* and summarized in the following sections.

23.2.1 Laboratory Receipt

When samples arrive at the laboratory, sample receiving personnel inspect the coolers and samples. The integrity of each sample must be determined by comparing sample labels or tags with the COC and by visual checks of the container for possible damage. Any non-conformance, irregularity, or compromised sample receipt must be documented in a TALS Sample Receiving NCM and brought to the immediate attention of the client. The COC, shipping documents, documentation of any non-conformance, irregularity, or compromised sample receipt, record of client contact, and resulting instructions become part of the project record.

Sample Receiving personnel check and document preservation of non-volatile liquid samples after the samples have been entered into the LIMS and before they are released to the laboratory for testing or placed into storage. Any checks of volatile samples and samples for oil and grease are performed and documented at the time of analysis.

23.3 Sample Acceptance Policy

The laboratory has a written sample acceptance policy (Figure 23-1) that clearly outlines the circumstances under which samples shall be accepted or rejected. These include:

- a COC filled out completely;
- samples must be properly labeled;
- proper sample containers with adequate volume for the analysis (Sampling Guide) and necessary QC;
- samples must be preserved according to the requirements of the requested analytical method (Sampling Guide);
- sample holding times must be adhered to (Sampling Guide);
- the project manager will be notified if any sample is received in damaged condition.

Data from samples which do not meet these criteria are flagged and the nature of the variation from policy is defined.

23.3.1 After inspecting the samples, the sample receiving personnel sign and date the COC form, make any necessary notes of the samples' conditions and store them in appropriate refrigerators or storage locations.

23.3.2 Any deviations from these checks that question the suitability of the sample for analysis, or incomplete documentation as to the tests required will be resolved by consultation with the client. If the sample acceptance policy criteria are not met, the laboratory shall either:

- Retain all correspondence and/or records of communications with the client regarding the disposition of rejected samples, or
- Fully document any decision to proceed with sample analysis that does not meet sample acceptance criteria.

Once sample acceptance is verified, the samples are logged into LIMS according to SOP No. ED-SPM-001, *Procedure For Sample Receipt, Login, Identification, And Storage*.

23.4 Sample Storage

In order to avoid deterioration, contamination or damage to a sample during storage and handling, from the time of receipt until all analyses are complete, samples are stored in refrigerators, freezers or protected locations suitable for the sample matrix. Samples for metals analysis are stored unrefrigerated. In addition, samples to be analyzed for volatile organic parameters are stored in separate refrigerators designated for volatile organic parameters only. Samples are never to be stored with reagents, standards or materials that may create contamination.

To ensure the integrity of the samples during storage, refrigerator blanks are maintained in the volatile sample refrigerators and analyzed every two weeks.

Analysts and technicians retrieve the sample container allocated to their analysis from the designated refrigerator and place them on carts, analyze the sample, and return the remaining sample or empty container to the refrigerator or shelf (for metals samples) from which it originally came. All unused portions of samples, including empty sample containers, are returned to the secure sample control area. All samples are kept in the refrigerators for 30 days after delivery of the final report to the client, which meets or exceeds most sample holding times. After 30 days the samples are disposed of or, upon client request moved to a sample archive area where they are stored for an additional time period agreed upon with the client or dictated by the applicable analytical program..

Access to the laboratory is controlled such that sample storage need not be locked at all times unless a project specifically demands it. Samples are accessible to laboratory personnel only. Visitors to the laboratory are prohibited from entering the refrigerator and laboratory areas unless accompanied by an employee of Eurofins Edison.

23.5 Hazardous Samples and Foreign Soils

To minimize exposure to personnel and to avoid potential accidents, hazardous and foreign soil samples are stored in an isolated area designated for hazardous waste only.

Procedures for the handling and storage of hazardous samples are addressed in the Eurofins Safety Manual (Document No. CW-E-M-001) and in Eurofins Edison SOP No. ED-SPM-001 (*Sample Receipt, Login, Identification, and Storage*).

Procedures for the acceptance and handling of USDA regulated domestic and foreign soils are detailed in Eurofins Edison SOP No. ED-SPM-006 (*Procedure for Acceptance and Handling of Regulated Domestic and Foreign Soils).*

23.6 Sample Shipping

In the event that the laboratory needs to ship samples, the samples are placed in a cooler with enough ice to ensure the samples remain just above freezing and at or below 6.0°C during transit. The samples are carefully surrounded by packing material to avoid breakage (yet maintain appropriate temperature). A trip blank is enclosed for those samples requiring water/solid volatile organic analyses (see Note). The chain-of-custody form is signed by the sample control technician and attached to the shipping paperwork. Samples are generally shipped overnight express or hand-delivered by a Eurofins Edison courier to maintain sample integrity. All personnel involved with shipping and receiving samples must be trained to maintain the proper chain-of-custody documentation and to keep the samples intact and on ice. The laboratory's Environmental, Health and Safety Manual contains additional shipping requirements.

Note: If a client does not request trip blank analysis on the COC or other paperwork, the laboratory will not analyze the trip blanks that were supplied. However, in the interest of good client service, the laboratory will advise the client at the time of sample receipt that it was noted that they did not request analysis of the trip blank; and that the laboratory is providing the notification to verify that they are not inadvertently omitting a key part of regulatory compliance testing.

23.7 Sample Disposal

Samples should be retained for a minimum of 30 days after the project report is sent, however, provisions may be made for earlier disposal of samples once the holding time is exceeded. Some samples are required to be held for longer periods based on regulatory or client requirements (e.g., 60 days after project report is sent). The laboratory must follow the longer sample retention requirements where required by regulation or client agreement. Several possibilities for sample disposal exist: the sample may be consumed completely during analysis, the sample may be returned to the customer or location of sampling for disposal, or the sample may be disposed of in accordance with the laboratory's waste disposal procedures (SOP No. ED-SPM-008 (Laboratory Waste Disposal Procedures). All procedures in the laboratory's Environmental Health and Safety Manual are followed during disposal. Samples are normally maintained in the laboratory no longer than two months from receipt unless otherwise requested. Unused portions of samples found or suspected to be hazardous according to state or federal guidelines may be returned to the client upon completion of the analytical work.

If a sample is part of a known litigation, the affected legal authority, sample data user, and/or submitter of the sample must participate in the decision about the sample's disposal. All documentation and correspondence concerning the disposal decision process must be kept on file. Pertinent information includes the date of disposal, nature of disposal (such as sample depletion, hazardous waste facility disposal, return to client), names of individuals who conducted the arrangements and physically completed the task. The laboratory will remove or deface sample labels prior to disposal unless this is accomplished through the disposal method (e.g., samples are incinerated). A Waste Disposal Record should be completed.

Figure 23-1. Example: Sample Acceptance Policy

All incoming work will be evaluated against the criteria listed below. Where applicable, data from any samples that do not meet the criteria listed below will be noted on the laboratory report defining the nature and substance of the variation. In addition the client will be notified either by telephone, fax or e-mail ASAP after the receipt of the samples.

Per State and/or Federal Regulation, the client is responsible to ensure that samples are shipped in accordance with DOT/IATA requirements, and that radioactive materials may only be delivered to licensed facilities. Any samples containing (or suspected to contain) Source, Byproduct, or Special Nuclear Material as defined by 10 CFR should be delivered directly to facilities licensed to handle such radioactive material. Natural material or ores containing naturally occurring radionuclides may be delivered to any Eurofins facility or courier as long as the activity concentration of the material does not exceed 270 pCi/g alpha or 2700 pCi/g beta (49 CFR Part 173).

1) Samples must arrive with labels intact with a Chain of Custody filled out completely. The following information must be recorded.
    - *Client name, address, phone number and fax number (if available)*
    - *Project name and/or number*
    - *The sample identification*
    - *Date, time and location of sampling*
    - *The collectors name*
    - *The matrix description*
    - *The container description*
    - *The total number of each type of container*
    - *Preservatives used*
    - *Analysis requested*
    - *Requested turnaround time (TAT)*
    - *Any special instructions*
    - *Purchase Order number or billing information (e.g. quote number) if available*
    - *The date and time that each person received or relinquished the sample(s), including their signed name.*
    - *The date and time of receipt must be recorded between the last person to relinquish the samples and the person who receives the samples in the lab, and they must be exactly the same.*
    - Information must be legible
2) Samples must be properly labeled.
    - Use durable labels (labels provided by Eurofins are preferred)
    - Include a unique identification number
    - Include sampling date and time & sampler ID
    - Include preservative used.
    - Use indelible ink
    - Information must be legible

3) Proper sample containers with adequate volume for the analysis and necessary QC are required for each analysis requested.

4) Samples must be preserved according to the requirements of the requested analytical method

5) Most analytical methods require chilling samples to 4° C (other than water samples for metals analysis). For these methods, the criteria are met if the samples are chilled to below 6° C and above freezing (0°C). For methods with other temperature criteria (e.g. some bacteriological methods require ≤ 10 °C), the samples must arrive within ± 2° C of the required temperature or within the method specified range.

5i.) Samples that are delivered to the laboratory on the same day they are collected may not meet the requirements of Section 5. In these cases, the samples shall be considered acceptable if the samples were received on ice.

5ii.) If sample analysis is begun within fifteen (15) minutes of collection, thermal preservation is not required.

5iii.) Thermal preservation is not required in the field if the laboratory receives and refrigerates the sample within fifteen (15) minutes of collection.

- Chemical preservation (pH) will be verified prior to analysis and documented, either in sample control or at the analyst's level. The project manager will be notified immediately if there is a discrepancy. If analyses will still be performed, all affected results will be flagged to indicate improper preservation.
- For Volatile Organic analyses in drinking water (Methods 502.2 or 524.2). Residual chlorine must be neutralized prior to preservation. If there is prior knowledge that the samples are not chlorinated, state it on the COC and use the VOA vials pre-preserved with HCl. The following are other options for a sampler and laboratory where the presence of chlorine is not known:
  - 1. Test for residual chlorine in the field prior to sampling.
    - If no chlorine is present, the samples are to be preserved using HCl as usual.
    - If chlorine is present, add either ascorbic acid or sodium thiosulfate prior to adding HCl.
  - 2. Use VOA vials pre-preserved with sodium thiosulfate or ascorbic acid and add HCl after filling the VOA vial with the sample.
- **FOR WATER SAMPLES TESTED FOR CYANIDE (by Standard Methods or EPA 335)**
  - In the Field: Samples are to be tested for Sulfide using lead acetate paper prior to the addition of Sodium Hydroxide (NaOH). If sulfide is present, the sample must be treated with Cadmium Chloride and filtered prior to the addition of NaOH.
    - If the sulfide test and treatment is not performed in the field, the lab will test the samples for sulfide using lead acetate paper at the time of receipt and if sulfide is present in the sample, the client will be notified and given the option of retaking the sample and treating in the field per the method requirements or the laboratory can analyze the samples as delivered and qualify the results in the final report.
  - It is the responsibility of the client to notify the laboratory if thiosulfate, sulfite, or thiocyanate are known or suspected to be present in the sample. This notification may be on the chain of custody. The samples may need to be subcontracted to a laboratory that performs a UV digestion. If the lab does not perform the UV digestion on samples that contain these compounds, the results must be qualified in the final report.
  - The laboratory must test the sample for oxidizing agents (e.g. Chlorine) prior to analysis and treat according to the methods prior to distillation. (ascorbic acid or sodium arsenite are the preferred choice).

Sample Holding Times

- Eurofins Edison will make every effort to analyze samples within the regulatory holding time. Samples must be received in the laboratory with enough time to perform the sample analysis. Except for short holding time samples (< 48hr HT) sample must be received with at least 48 hrs (2 working days) remaining on the holding time for us to ensure analysis.
- Analyses that are designated as "field" analyses (Odor, pH, Dissolved Oxygen, Disinfectant Residual; a.k.a. Residual Chlorine, and Redox Potential) should be analyzed ASAP by the field sampler prior to delivering to the lab (within 15 minutes). However, if the analyses are to be performed in the laboratory, Eurofins Edison will make every effort to analyze the samples within 24 hours from receipt of the samples in the testing laboratory. Samples for "field" analyses received after 4:00 pm on Friday or on the weekend will be analyzed no later than the next business day after receipt (Monday unless a holiday). Samples will remain refrigerated and sealed until the time of analysis. The actual times of all "field" sample analyses are noted on the "Short Hold Time Detail Report" in the final report. Samples analyzed in the laboratory will be qualified on the final report with an 'H' to indicate holding time exceedance.

6) All samples submitted for Volatile Organic analyses must have a Trip Blank submitted at the same time. Eurofins Edison will supply a blank with the bottle order.

7) The project manager will be notified if any sample is received in damaged condition. Eurofins Edison will request that a sample be resubmitted for analysis.

8) Recommendations for packing samples for shipment.
   - Pack samples in Ice rather than "Blue" ice packs.
   - Soil samples should be placed in plastic zip-lock bags. The containers often have dirt around the top and do not seal very well and are prone to intrusion from the water from melted ice.
   - Water samples would be best if wrapped with bubble-wrap or paper (newspaper, or paper towels work) and then placed in plastic zip-lock bags.
   - Fill extra cooler space with bubble wrap.

24.0 ASSURING THE QUALITY OF TEST RESULTS

24.1 Overview

In order to assure our clients of the validity of their data, the laboratory continuously evaluates the quality of the analytical process. The analytical process is controlled not only by instrument calibration as discussed in Section 20, but also by routine process quality control measurements (e.g. Blanks, Laboratory Control Samples (LCS), Matrix Spikes (MS), duplicates (DUP), surrogates, Internal Standards (IS)). These quality control checks are performed as required by the method or regulations to assess precision and accuracy. Quality control samples are to be treated in the exact same manner as the associated field samples being tested. In addition to the routine process quality control samples, Proficiency Testing (PT) Samples (concentrations unknown to laboratory) are analyzed to help ensure laboratory performance.

24.2 Controls

Sample preparation or pre-treatment is commonly required before analysis. Typical preparation steps include homogenization, grinding, solvent extraction, sonication, acid digestion, distillation, reflux, evaporation, drying and ashing. During these pre-treatment steps, samples are arranged into discreet manageable groups referred to as preparation (prep) batches. Prep batches provide

a means to control variability in sample treatment. Control samples are added to each prep batch to monitor method performance and are processed through the entire analytical procedure with investigative/field samples.

24.3 Negative Controls

Table 24-1. Example – Negative Controls

Control Type	Details
Method Blank (MB)	are used to assess preparation and analysis for possible contamination during the preparation and processing steps.
	The specific frequency of use for method blanks during the analytical sequence is defined in the specific standard operating procedure for each analysis. Generally it is 1 for each batch of samples; not to exceed 20 environmental samples.
	The method blank is prepared from a clean matrix similar to that of the associated samples that is free from target analytes (e.g., Reagent water, Ottawa sand, glass beads, etc.) and is processed along with and under the same conditions as the associated samples. The method blank goes through all of the steps of the process (including as necessary: filtration, clean-ups, etc.).
	Reanalyze or qualify associated sample results when the concentration of a targeted analyte in the blank is at or above the reporting limit as established by the method or by regulation, AND is greater than 1/10 of the amount measured in the sample.
Calibration Blanks	are prepared and analyzed along with calibration standards where applicable. They are prepared using the same reagents that are used to prepare the standards. In some analyses the calibration blank may be included in the calibration curve.
Instrument Blanks	are blank reagents or reagent water that may be processed during an analytical sequence in order to assess contamination in the analytical system. In general, instrument blanks are used to differentiate between contamination caused by the analytical system and that caused by the sample handling or sample prep process. Instrument blanks may also be inserted throughout the analytical sequence to minimize the effect of carryover from samples with high analyte content.
Trip Blank [1]	are required to be submitted by the client with each shipment of samples requiring aqueous and solid volatiles analyses (or as specified in the client's project plan). Additionally, trip blanks may be prepared and analyzed for volatile analysis of air samples, when required by the client. A trip blank may be purchased (certified clean) or is prepared by the laboratory by filling a clean container with pure deionized water that has been purged to remove any volatile compounds. Appropriate preservatives are also added to the container. The trip blank is sent with the bottle order and is intended to reflect the environment that the containers are subjected to throughout shipping and handling and help identify possible sources if contamination is found. The field sampler returns the trip blank in the cooler with the field samples.
Field Blanks [1]	are sometimes used for specific projects by the field samplers. A field blank prepared in the field by filling a clean container with pure reagent water and appropriate preservative, if any, for the specific sampling activity being undertaken. (EPA OSWER)
Equipment Blanks [1]	are also sometimes created in the field for specific projects. An equipment blank is a sample of analyte-free media which has been used to rinse common sampling equipment to check effectiveness of decontamination procedures. (TNI)
Holding Blanks	also referred to as refrigerator or freezer blanks, are used to monitor the sample storage units for volatile organic compounds during the storage of VOA samples in the laboratory

[1] When known, these field QC samples should not be selected for matrix QC as it does not provide information on the behavior of the target compounds in the field samples. Usually, the client sample ID will provide information to identify the field blanks with labels such as "FB", "EB", or "TB."

Evaluation criteria and corrective action for these controls are defined in the specific standard operating procedure for each analysis.

24.4 Positive Controls

Control samples (e.g., QC indicators) are analyzed with each batch of samples to evaluate data based upon (1) Method Performance (Laboratory Control Sample (LCS) or Blank Spike (BS)), which entails both the preparation and measurement steps; and (2) Matrix Effects (Matrix Spike (MS) or Sample Duplicate (MD, DUP), which evaluates field sampling accuracy, precision, representativeness, interferences, and the effect of the matrix on the method performed. Each regulatory program and each method within those programs specify the control samples that are prepared and/or analyzed with a specific batch

Note that frequency of control samples vary with specific regulatory, methodology and project specific criteria. Complete details on method control samples are as listed in each analytical SOP.

24.4.1 Method Performance Control - Laboratory Control Sample (LCS)

The LCS measures the accuracy of the method in a blank matrix and assesses method performance independent of potential field sample matrix affects in a laboratory batch.

The LCS is prepared from a clean matrix similar to that of the associated samples that is free from target analytes (for example: Reagent water, Ottawa sand, glass beads, etc.) and is processed along with and under the same conditions as the associated samples. The LCS is spiked with verified known amounts of analytes or is made of a material containing known and verified amounts of analytes, taken through all preparation and analysis steps along with the field samples. Where there is no preparation taken for an analysis (such as in aqueous volatiles), or when all samples and standards undergo the same preparation and analysis process a calibration verification standard is reported as the LCS. In some instances where there is no practical clean solid matrix available, aqueous LCS's may be processed for solid matrices; final results may be calculated as mg/kg or ug/kg, assuming 100% solids and a weight equivalent to the aliquot used for the corresponding field samples, to facilitate comparison with the field samples.

Certified pre-made reference material purchased from a NIST/A2LA accredited vendor may also be used for the LCS when the material represents the sample matrix or the analyte is not easily spiked (e.g. solid matrix LCS for metals, TDS, etc.).

The specific frequency of use for LCS during the analytical sequence is defined in the specific standard operating procedure for each analysis. It is generally 1 for each batch of samples; not to exceed 20 environmental samples.

If the mandated or requested test method, or project requirements, do not specify the spiking components, the laboratory shall spike all reportable components to be reported in the Laboratory Control Sample (and Matrix Spike) where applicable (e.g. no spike of pH). However, in cases where the components interfere with accurate assessment (such as simultaneously spiking chlordane, Toxaphene and PCBs in Method 608.3), the test method has an extremely long list of components or components are incompatible, at a minimum, a representative number of the listed components (see below) shall be used to control the test method. The

selected components of each spiking mix shall represent all chemistries, elution patterns and masses, permit specified analytes and other client requested components. However, the laboratory shall ensure that all reported components are used in the spike mixture within a two-year time period.

- For methods that have 1-10 target analytes, spike all components.
- For methods that include 11-20 target analytes, spike at least 10 or 80%, whichever is greater.
- For methods with more than 20 target analytes, spike at least 16 components.
- Exception: Due to analyte incompatibility in pesticides, Toxaphene and Chlordane are only spiked at client request based on specific project needs.
- Exception: Due to analyte incompatibility between the various PCB aroclors, aroclors 1016 and 1260 are used for spiking as they cover the range of all of the aroclors. Specific aroclors may be used by request on a project specific basis.

24.5 Sample Matrix Controls

Table 24-3. Sample Matrix Control

Control Type	Details	
Matrix Spikes (MS)	Use	used to assess the effect sample matrix of the spiked sample has on the precision and accuracy of the results generated by the method used;
	Typical Frequency [1]	At a minimum, with each matrix-specific batch of samples processed, an MS is carried through the complete analytical procedure. Unless specified by the client, samples used for spiking are randomly selected and rotated between different client projects. If the mandated or requested test method does not specify the spiking components, the laboratory shall spike all reportable components to be reported in the Laboratory Control Sample and Matrix Spike. Refer to the method SOP for complete details
	Description	essentially a sample fortified with a known amount of the test analyte(s).
Surrogate	Use	Measures method performance to sample matrix (organics only).
	Typical Frequency [1]	Are added to all samples, standards, and blanks, for all organic chromatography methods except when the matrix precludes its use or when a surrogate is not available. The recovery of the surrogates is compared to the acceptance limits for the specific method. Poor surrogate recovery may indicate a problem with sample composition and shall be reported, with data qualifiers, to the client whose sample produced poor recovery.
	Description	Are similar to matrix spikes except the analytes are compounds with properties that mimic the analyte of interest and are unlikely to be found in environment samples.
Duplicates[2]	Use	For a measure of analytical precision, with each matrix-specific batch of samples processed, a matrix duplicate (MD or DUP) sample, matrix spike duplicate (MSD), or LCS duplicate (LCSD) is carried through the complete analytical procedure.
	Typical Frequency [1]	Duplicate samples are usually analyzed with methods that do not require matrix spike analysis.
	Description	Performed by analyzing two aliquots of the same field sample independently or an additional LCS.
Internal Standards	Use	Are spiked into all environmental and quality control samples (including the initial calibration standards) to monitor the qualitative aspect of organic and some inorganic analytical measurements.
	Typical Frequency [1]	All organic and ICP methods as required by the analytical method.

Table 24-3. Sample Matrix Control

Control Type	Details	
	Description	Used to correct for matrix effects and to help troubleshoot variability in analytical response and are assessed after data acquisition. Possible sources of poor internal standard response are sample matrix, poor analytical technique or instrument performance.

[1] See the specific analytical SOP for type and frequency of sample matrix control samples.

[2] LCSD's are normally not performed except when regulatory agencies or client specifications require them. The recoveries for the spiked duplicate samples must meet the same laboratory established recovery limits as the accuracy QC samples. If an LCSD is analyzed both the LCS and LCSD must meet the same recovery criteria and be included in the final report. The precision measurement is reported as "Relative Percent Difference" (RPD). Poor precision between duplicates (except LCS/LCSD) may indicate non-homogeneous matrix or sampling.

24.6 Acceptance Criteria (Control Limits)

As mandated by the test method and regulation, each individual analyte in the LCS, MS, or Surrogate Spike is evaluated against the control limits published in the test method. Where there are no established acceptance criteria, the laboratory calculates in-house control limits with the use of control charts or, in some cases, utilizes client project specific control limits. When this occurs, the regulatory or project limits will supersede the laboratory's in-house limits.

Note: For methods, analytes and matrices with very limited data (e.g., unusual matrices not analyzed often), interim limits are established using available data or by analogy to similar methods or matrices.

Once control limits have been established, they are verified, reviewed, and updated if necessary on an annual basis unless the method requires more frequent updating. Control limits are established per method (as opposed to per instrument) regardless of the number of instruments utilized.

Laboratory generated % Recovery acceptance (control) limits are generally established by taking $\pm$ 3 Standard Deviations (99% confidence level) from the average recovery of a minimum of 20-30 data points (more points are preferred).

- Regardless of the calculated limit, the limit should be no tighter than the Calibration Verification (ICV/CCV). (Unless the analytical method specifies a tighter limit).
- In-house limits cannot be any wider than those mandated in a regulated analytical method. Client or contract required control limits are evaluated against the laboratory's statistically derived control limits to determine if the data quality objectives (DQOs) can be achieved. If laboratory control limits are not consistent with DQOs, then alternatives must be considered, such as method improvements or use of an alternate analytical method.
- The lowest acceptable recovery limit will be 10% (the analyte must be detectable and identifiable). Exception: The lowest acceptable recovery limit for Benzidine will be 5% and the analyte must be detectable and identifiable.
- The maximum acceptable recovery limit will be 150%.
- The maximum acceptable RPD limit will be 35% for waters and 40% for soils. The minimum RPD limit is 10%.

- If either the high or low end of the control limit changes by ≤ 5% from previous, the control chart is visually inspected and, using professional judgment, they may be left unchanged if there is no affect on laboratory ability to meet the existing limits.

24.6.1 The lab must be able to generate a current listing of their control limits and track when the updates are performed. In addition, the laboratory must be able to recreate historical control limits. Reference Eurofins Edison SOP No. ED-GEN-026 (Evaluation of Analytical Accuracy and Precision Through The Use of Control Charts).

24.6.2 A LCS that is within the acceptance criteria establishes that the analytical system is in control and is used to validate the process. Samples that are analyzed with an LCS with recoveries outside of the acceptance limits may be determined as out of control and should be reanalyzed if possible. If reanalysis is not possible, then the results for all affected analytes for samples within the same batch must be qualified when reported. The internal corrective action process (see Section 12) is also initiated if an LCS exceeds the acceptance limits. Sample results may be qualified and reported without reanalysis if:

- The analyte results are below the reporting limit and the LCS is above the upper control limit.
- If the analytical results are above the relevant regulatory limit and the LCS is below the lower control limit.

If the MS/MSDs do not meet acceptance limits, the MS/MSD and the associated spiked sample is reported with a qualifier for those analytes that do not meet limits. If obvious preparation errors are suspected, or if requested by the client, unacceptable MS/MSDs are reprocessed and reanalyzed to prove matrix interference. A more detailed discussion of acceptance criteria and corrective action can be found in the lab's method SOPs and in Section 12.

24.6.3 If a surrogate standard falls outside the acceptance limits, and if there is not obvious chromatographic matrix interference, reanalyze the sample to confirm a possible matrix effect. If the recoveries confirm or there was obvious chromatographic interference, results are reported from the original analysis and a qualifier is added. If the reanalysis meets surrogate recovery criteria, the second run is reported (or both are reported if requested by the client). Under certain circumstances, where all of the samples are from the same location and share similar chromatography, the reanalysis may be performed on a single sample rather than all of the samples and if the surrogate meets the recovery criteria in the reanalysis, all of the affected samples would require reanalysis.

24.7 Additional Procedures to Assure Quality Control

The laboratory has written and approved method SOPs to assure the accuracy of the test method including calibration (see Section 20), use of certified reference materials (see Section 21) and use of PT samples (see Section 17).

A discussion regarding MDLs, Limit of Detection (LOD) and Limit of Quantitation (LOQ) can be found in Section 19.

- Use of formulae to reduce data is discussed in the method SOPs and in Section 20.
- Selection of appropriate reagents and standards is included in Section 11 and 21.
- A discussion on selectivity of the test is included in Section 7.
- Constant and consistent test conditions are discussed in Section 6.
- The laboratories sample acceptance policy is included in Section 23.

25.0 REPORTING RESULTS

25.1 Overview

The results of each test are reported accurately, clearly, unambiguously, and objectively in accordance with State and Federal regulations as well as client requirements. Analytical results are issued in a format that is intended to satisfy customer and laboratory accreditation requirements as well as provide the end user with the information needed to properly evaluate the results. Where there is conflict between client requests and laboratory ethics or regulatory requirements, the laboratory's ethical and legal requirements are paramount, and the laboratory will work with the client during project set up to develop an acceptable solution. Refer to Section 9.

A variety of report formats are available to meet specific needs.

In cases where a client asks for simplified reports, there must be a written request from the client. There still must be enough information that would show any analyses that were out of conformance (QC out of limits) and there should be a reference to a full report that is made available to the client. Review of reported data is included in Section 19.

25.2 Test Reports

Analytical results are reported in a format that is satisfactory to the client and meets all requirements of applicable accrediting authorities and agencies. A variety of report formats are available to meet specific needs. The report is printed on laboratory letterhead, reviewed, and signed by the appropriate project manager. At a minimum, the standard laboratory report shall contain the following information:

25.2.1 A report title (e.g., Analytical Report)

25.2.2 The cover page shall include the laboratory name, address and telephone number.

25.2.3 A unique identification of the report (e.g., Eurofins Edison Job ID #) and on each page an identification in order to ensure the page is recognized as part of the report and a clear identification of the end.

Note: Page numbers of report are represented as page # of ##. Where the first number is the page number and the second is the total number of pages.

25.2.4 A copy of the chain of custody (COC).

- Any COCs involved with Subcontracting are included.

25.2.5 The name and address of client and a project name/number, if applicable.

25.2.6 Client project manager or other contact

25.2.7 Description and unambiguous identification of the tested sample(s) including the client identification code.

25.2.8 Date of receipt of sample, date and time of collection, and date(s) of test preparation and performance, and time of preparation or analysis if the required holding time for either activity is less than or equal to 72 hours.

25.2.9 Date reported or date of revision, if applicable.

25.2.10 Method of analysis including method code (EPA, Standard Methods, etc.).

25.2.11 Reporting limit.

25.2.12 Method detection limits (if requested)

25.2.13 Definition of Data qualifiers and reporting acronyms (e.g. ND).

25.2.14 Sample results.

25.2.15 QC data consisting of method blank, surrogate, LCS, and MS/MSD recoveries and control limits.

25.2.16 Condition of samples at receipt including temperature. This may be accomplished in a narrative or by attaching sample login sheets (Refer to Sec. 25.2.4 – Item 3 regarding additional addenda).

25.2.17 A statement expressing the validity of the results, that the source methodology was followed and all results were reviewed for error.

25.2.18 A statement to the effect that the results relate only to the items tested and the sample as received by the laboratory, except when information is provided by the client. When data is provided by the client there shall be a clear identification of it, and a disclaimer shall be put in the report when the client supplied data can affect the validity of the test.

25.2.19 A statement that the report shall not be reproduced except in full, without prior express written approval by the laboratory coordinator.

25.2.20 A signature and title of the person(s) accepting responsibility for the content of the report and date of issue. Authorized signatories are qualified Project Managers appointed by the Manager of Project Managers.

25.2.21 When TNI accreditation is required, the lab shall certify that the test results meet all requirements of TNI or provide reasons and/or justification if they do not.

25.2.22 The laboratory includes a cover letter.

25.2.23 Where applicable, a narrative to the report that explains the issue(s) and corrective action(s) taken in the event that a specific accreditation or certification requirement was not met.

25.2.24 When soil samples are analyzed, a specific identification as to whether soils are reported on a “wet weight” or “dry weight” basis.

25.2.25 Appropriate laboratory certification number for the state of origin of the sample, if applicable.

25.2.26 If only part of the report is provided to the client (client requests some results before all of it is complete), it must be clearly indicated on the report (e.g., partial report, or how your lab identifies it). A complete report must be sent once all of the work has been completed.

25.2.27 Any non-Eurofins subcontracted analysis results are provided as a separate report on the official letterhead of the subcontractor. All Eurofins subcontracting is clearly identified on the report as to which laboratory performed a specific analysis.

25.2.28 A Certification Summary Report, where required, will document that, unless otherwise noted, all analytes tested and reported by the laboratory were covered by the noted certifications.

Note: Refer to the NDSC Document No. CA-I-P-002, Electronic Reporting and Signature Policy. for details on internally applying electronic signatures of approval.

25.2.29 Where the laboratory is responsible for the sampling stage, in addition to the requirements listed above, reports containing the results of sampling shall include the following, where necessary for the interpretation of test results:

- the date of sampling;
- unambiguous identification of the material sampled;
- the location of sampling plan and procedures, and deviations, addition to or exclusions from the sample procedures;
- a reference to the sampling plan and procedure, and deviations, additions to or exclusions from the sample procedures;
- details of any environmental conditions during sampling that affect the interpretation of test results;
- information required to evaluate measurement uncertainty for subsequent testing

25.3 Reporting Level or Report Type

The laboratory offers four levels of quality control reporting. Each level, in addition to its own specific requirements, contains all the information provided in the preceding level. The packages provide the following information in addition to the information described above:

- Level 1 is a report with all of the elements outlined in Section 25.2 above, excluding 25.2.15 (QC data).
- Level II is a Level I report plus summary information, including results for the method blank reported to the laboratory MDL, percent recovery for laboratory control samples and matrix spike samples, and the RPD values for all MSD and sample duplicate analyses.
- Level III contains all the information supplied in Level II, but presented on the CLP-like summary forms, and relevant calibration information. A Level II report is not included, unless specifically requested. No raw data is provided.
- Level IV is the same as Level III with the addition of all raw supporting data.

In addition to the various levels of QC packaging, the laboratory also provides reports in EDD (see below). Initial reports may be provided to clients by facsimile. Procedures used to ensure client confidentiality are outlined in Section 25.6.

25.4 Electronic Data Deliverables (EDDs)

EDDs are routinely offered as part of Eurofins Edison services in addition to the test report as described in Section 25.2. When NELAP accreditation is required and both a test report and EDD are provided to the client, the official version of the test report will be the combined information of the report and the EDD. Eurofins Edison offers a variety of EDD formats including NJ Hazsite Deliverables, Excel, Dbase, GISKEY, and Text Files

EDD specifications are submitted to the IT department by the PM for review and undergo the contract review process. Once the facility has committed to providing data in a specific electronic format, the coding of the format may need to be performed. This coding is documented and validated. The validation of the code is retained by the IT staff coding the EDD.

EDDs shall be subject to a review to ensure their accuracy and completeness. If EDD generation is automated, review may be reduced to periodic screening if the laboratory can demonstrate that it can routinely generate that EDD without errors. Any revisions to the EDD format must be reviewed until it is demonstrated that it can routinely be generated without

errors. If the EDD can be reproduced accurately and if all subsequent EDDs can be produced error-free, each EDD does not necessarily require a review.

25.5 Supplemental Information for Test

The lab identifies any unacceptable QC analyses or any other unusual circumstances or observations such as environmental conditions and any non-standard conditions that may have affected the quality of a result. This is typically in the form of a footnote or a qualifier and/or a narrative explaining the discrepancy in the front of the report.

Numeric results with values outside of the calibration range, either high or low are qualified as estimated.

Where quality system requirements are not met, a statement of compliance/non-compliance with requirements and/or specifications is required, including identification of test results derived from any sample that did not meet TNI sample acceptance requirements such as improper container, holding time, or temperature.

Where applicable, a statement on the estimated uncertainty of measurements; information on uncertainty is needed when a client's instructions so require.

When, as requested by the client and agreed to by Eurofins Edison the report includes a statement of conformity to specification or standard (see Special Services, Section 7.4), the report shall clearly identify:

- to which results the statement applies,
- which specifications, standard or parts thereof are met or not, and
- the decision rule that was applied (unless the decision rule is inherent in the requested specification or standard, taking into account the level of risk (such as false accept and false reject and statistical assumptions) associated with the decision rule.

Opinions and Interpretations - The test report contains objective information, and generally does not contain subjective information such as opinions and interpretations. If such information is required by the client, the Laboratory Director will determine if a response can be prepared. If so, the Laboratory Director will designate the appropriate member of the management team to prepare a response. The response will be fully documented, and reviewed by the Laboratory Director, before release to the client. There may be additional fees charged to the client at this time, as this is a non-routine function of the laboratory.

Note: Review of data deliverable packages for submittal to regulatory authorities requires responses to non-conforming data concerning potential impact on data quality. This necessitates a limited scope of interpretation, and this work is performed by the QA Department. This is the only form of "interpretation" of data that is routinely performed by the laboratory.

When opinions or interpretations are included in the report, the laboratory provides an explanation as to the basis upon which the opinions and interpretations have been made. Opinions and interpretations are clearly noted as such and where applicable, a comment should be added suggesting that the client verify the opinion or interpretation with their regulator.

25.6 Environmental Testing Obtained From Subcontractors

If the laboratory is not able to provide the client the requested analysis, the samples would be subcontracted following the procedures outlined in the NDSC Document No. CW-L-S-004, Subcontracting.

Data reported from analyses performed by a subcontractor laboratory are clearly identified as such on the analytical report provided to the client. Results from a subcontract laboratory outside of Eurofins are reported to the client on the subcontract laboratory's original report stationary and the report includes any accompanying documentation.

25.7 Client Confidentiality

The laboratory will ensure the highest standards of quality and integrity of the data and services provided to our clients

The laboratory is responsible for maintaining in confidence all client information obtained or created. In situations involving the transmission of environmental test results by telephone, facsimile or other electronic means, client confidentiality must be maintained.

The laboratory will not intentionally divulge to any person (other than the client or any other person designated by the client in writing) any information regarding the services provided by the laboratory or any information disclosed to the laboratory by the client. Furthermore, information known to be potentially endangering to national security or an entity's proprietary rights will not be released.

Information about the client obtained from sources other than the client (e.g., complainant, regulators) shall be confidential between client and the laboratory. The source of this information shall be confidential to the laboratory and shall not be shared with the client, unless agreed by the source.

Note: This shall not apply to the extent that the information is required to be disclosed by the laboratory under the compulsion of legal process. The laboratory will, to the extent feasible, provide reasonable notice to the client before disclosing the information..

Note: Authorized representatives of an accrediting authority are permitted to make copies of any analyses or records relevant to the accreditation process, and copies may be removed from the laboratory for purposes of assessment.

25.7.1 Report deliverable formats are discussed with each new client. If a client requests that reports be e-mailed, the reports are to meet all requirements of this document.

25.8 Format of Reports

The format of reports is designed to accommodate each type of environmental test carried out and to minimize the possibility of misunderstanding or misuse.

25.9 Amendments to Test Reports

Corrections, additions, or deletions to reports are only made when justification arises through supplemental documentation. Justification is documented using the laboratory's corrective action system (refer to Section 12).

The revised report is retained on the Archive data server, as is the original report. The revised report is stored in the Archive data server under the sample number followed by "Rev (n)" where 'n' is the revision number. The revised report will have the words "Revision (n)" on the report cover page beneath the report date. Additionally, a section entitled "Revised Report" will appear on the Case Narrative page. A brief explanation of the reasons for the re-issue will be included in this section.

When the report is re-issued, a notation of "report re-issue" is placed on the cover/signature page of the report *or at the top of the narrative page* with a brief explanation of reason for the re-issue and a reference back to the last final report generated. *For Example*: *Report was revised on 3/8/20 to include toluene in sample NQA1504 per client's request. This final report replaces the final report generated on 1/27/20 at 10:47am.*

25.10 Policies on Client Requests for Amendments

25.10.1 Policy on Data Omissions or Reporting Limit Increases

Fundamentally, our policy is simply to not omit previously reported results (including data qualifiers) or to not raise reporting limits and report sample results as ND. This policy has few exceptions. Exceptions are:

- Laboratory error.
- Sample identification is indeterminate (confusion between COC and sample labels).
- An incorrect analysis (not analyte) was requested (e.g., COC lists 8315 but client wanted 8310). A written request for the change is required.
- Incorrect limits reported based on regulatory requirements.
- The requested change has absolutely *no possible* impact on the interpretation of the analytical results and there is *no possibility* of the change being interpreted as misrepresentation by anyone inside or outside of our company.

25.10.2 Multiple Reports

The laboratory does not issue multiple reports for the same work order where there is different information on each report (this does not refer to copies of the same report) unless required to meet regulatory needs and approved by QA.

Appendix 1. List of Governing Documents applicable to the QA Manual

NDSC Doc. No.	Title
CA-C-S-001	Work Sharing Process
CA-I-P-002	Electronic Reporting and Signature Policy
CA-L-P-002	Contract Compliance Policy
CA-Q-M-002	Quality Management Plan
CA-Q-QM-001	Guidance on Tentatively Identified Compounds (TICs) – GC/MS Analysis
CA-Q-S-001	Acid and Solvent-Lot Testing and Approval Program
CA-Q-S-002	Manual Integrations
CA-Q-S-006	Detection and Quantitation Limits
CA-Q-S-009	Root Cause Analysis
CA-T-P-001	Qualified Products List
CW-E-M-001	Environmental Health & Safety Manual
CW-F-P-002	Company-Wide Authorization Matrix
CW-F-P-004	Procurement and Contracts Policy

CW-F-S-007	Fixed Asset Acquisition, Retention and Safeguarding
CW-I-M-001	IT Change Control Procedure Manual
CW-I-P-001	Data Back-up Policy
CW-L-P-001	Records Retention Policy
CW-L-P-004	Ethics Policy
CW-L-S-002	Internal Investigation
CW-L-S-004	Subcontracting
CW-Q-S-001	Document Control and Archiving
CW-Q-S-002	Writing a Standard Operating Procedure (SOPs)
CW-Q-S-003	Internal Auditing
CW-Q-S-004	Management Systems Review
CW-Q-S-005	Data Recall Process
CW-Q-S-001	Document Control and Archiving
NDSC-QA-QP44940	Calibration Curves and the Selection of Calibration Points.
Local Laboratory Document No.	**Title**
ED-GEN-002	Document Control
ED-GEN-022	Training
ED-GEN-010	Calibration of Analytical Balances
ED-GEN-003	Control of Non-Conformances and Corrective Action
ED-GEN-024	Records Storage and Retention
ED-GEN-021	Data Review
ED-GEN-026	Evaluation of Analytical Accuracy and Precision Through The Use of Control Charts
ED-GEN-004	Quality Management Review
ED-GEN-014	Calibration of Laboratory Thermometers and Measurement of Temperatures in the Laboratory
ED-GEN-011	Calibration, Use, and Care of Laboratory Pipettes
ED-GEN-007	Subsampling
ED-FLD-008	Low Flow Monitoring Well Purging and Sampling
ED-FLD-009	Volume-Average Purging and Sampling
ED-FLD-014	Wastewater Sampling
ED-SPM-001	Procedure For Sample Receipt, Login, Identification, And Storage
ED-SPM-006	Procedure for Acceptance and Handling of Regulated Domestic and Foreign Soil
ED-SPM-008	Laboratory Waste Disposal Procedures

Appendix 2. List of Laboratory Certifications, Accreditations, Validations

The laboratory maintains accreditations, certifications, and approvals with numerous state and national entities. Programs vary but may include on-site audits, reciprocal agreements with another entity, performance testing evaluations, review of the QA Manual, Standard Operating Procedures, Method Detection Limits, training records, etc. At the time of this QA Manual revision, the laboratory has accreditation/ certification/licensing with the following organizations:

eurofins | Environment Testing America

Certification List

Laboratory	Program	Authority	Identification	Expiration Date
Edison	NELAP	New Jersey	12028	06/30/2023
Edison	NELAP	New York	11452	04/01/2023
Edison	NELAP	Pennsylvania	68-00522	02/28/2023
Edison	State	Connecticut	PH-0200	09/30/2022
Edison	State	DE Haz. Subst. Cleanup Act (HSCA)	N/A	01/01/2023
Edison	State	Massachusetts	M-NJ312	06/30/2023
Edison	State	Rhode Island	LAO00376	12/31/2022
Edison	US Federal Programs	USDA	P330-20-00244	11/03/2023

Appendix 3. References used to prepare the QA Manual

The QAM has been prepared to be consistent with the requirements of the following documents:

- ANSI/ASQC, E4-1994, "Specifications and Guidelines for Quality Management Systems for Environmental Data Collection and Environmental Technology Programs" (American National Standard, January 5, 1995, or most recent version)
- "EPA Requirements for Quality Management Programs" (QA/R-2) (EPA/240/B-01/002, May 31, 2006).
- EPA 600/4-88/039, *Methods for the Determination of Organic Compounds in Drinking Water,* EPA, Revised July 1991.
- EPA 600/R-95/131, *Methods for the Determination of Organic Compounds in Drinking Water,* Supplement III, EPA, August 1995.
- EPA 600/4-79-019, *Handbook for Analytical Quality Control in Water and Wastewater Laboratories,* EPA, March 1979.
- *Test Methods for Evaluating Solid Waste Physical/Chemical Methods (SW846), Third Edition, September 1986, Final Update I, July 1992, Final Update IIA, August 1993, Final Update II, September 1994; Final Update IIB, January 1995; Final Update III, December 1996; Final Update IV, January 2008; Final Update V, August 2015.*
- Federal Register, 40 CFR Parts 136, 141, 172, 173, 178, 179 and 261.
- *Manual for the Certification of Laboratories Analyzing Drinking Water (EPA 815-R-05-004, January 2005) (DW labs only)*
- *Statement of Work for Inorganics & Organics Analysis, SOM and ISM, current versions, USEPA Contract Laboratory Program Multi-media, Multi-concentration.*
- APHA, *Standard Methods for* the Examination of Water and Wastewater, 18th Edition, 19th, 20th, 21st, 22nd and on-line Editions.
- *U.S. Department of Energy* Order 414.1B, *Quality Assurance*, Approved April 29, 2004.
- *U.S. Department of Energy* Order 414.1C, *Quality Assurance*, June 17, 2005.
- *U.S. Department of Energy Order 414.1D, Quality Assurance, April, 25, 2011.*
- Nuclear Regulatory Commission (NRC) Quality Assurance Requirements.
- Marine Protection, Research, and Sanctuaries Act (MPRSA).
- Toxic Substances Control Act (TSCA).

Appendix 4. QA Manual Crosswalk with TNI and ISO/IEC 17025 Standards

Sec. No.	Title	2009 and 2016 TNI Standard Reference	ISO/IEC 17025:2005(E) Reference	ISO/IEC 17025:2017(E) Reference
-	QUALITY ASSURANCE MANUAL COVER PAGE	V1M2 Sec. 4.2.8.3		
1.0	Title Page			
2.0	Table of Contents	V1M2 Secs. 4.2.8.3-4.2.8.4		8.1.2, 8.2.1
3.0	Introduction, Scope and Applicability	V1M2 Sec. 4.2.8.4		
3.1	Introduction and Compliance References	V1M2 Secs. 1.1; 1.2; 2.0; 3.2; 4.1.2; 4.2.4	4.1.2; 4.2.4	5.3; 5.4; 8.2.4; 8.3.1
3.2	Terms and Definitions	V1M2 Secs. 3.0; 4.2.4	4.2.4	
3.3	Scope /Field of Testing	V1M2 Secs. 1.2; 4.2.4	4.1.2; 4.2.4	5.3; 5.4; 8.2.1; 8.2.4
3.4	Management of The Manual	V1M2 Secs. 4.2.1; 4.2.7; 4.3.3.2; 4.3.3.3	4.2.1; 4.2.7; 4.3.3.2; 4.3.3.3	5.3
4.0	Management and Responsibilities	V1M2 Sec. 4		8.2.4; 8.2.5
4.1	Overview	V1M2 Secs. 4.1.1, 4.1.3; 4.1.5	4.1.1; 4.1.3; 4.1.5; 4.2.6	5.1; 5.2; 5.5; 5.6 ; 6.2.1; 6.2.4
4.2	Roles and Responsibilities	V1M2 Secs. 4.1.4; 4.1.5; 4.1.6; 4.2.1; 4.2.6; 5.2.4	4.1.3; 4.1.5; 4.1.6; 4.2.1; 4.2.6; 5.2.4	4.1.1 to 4.1.3; 4.1.5; 5.5; 5.6; 6.2.1; 6.2.4; 6.2.6 8.2.2;
4.3	Business Continuity and Contingency Plan *(Prev. Deputies)*	V1M2 Secs. 4.1.5; 4.1.7.2; 4.2.7	4.1.5; 4.2.7	
5.0	PERSONNEL	V1M2 Secs. 5.2; 5.2.1	5.2.1	6.1; 6.2.3
5.1	Overview	V1M2 Secs. 5.2.2; 5.2.3; 5.2.5	5.2.2; 5.2.3; 5.2.5	6.2.2
5.2	Education and Experience Requirements for Technical Staff	V1M2 Secs. 5.2.1; 5.2.3; 5.2.4	5.2.1; 5.2.3; 5.2.4	6.2.2 to 6.2.4
5.3	Training	V1M2 Sec. 5.2.5	5.2.5	4.2.1; 6.2.2; 6.2.4; 6.2.5
5.4	Data Integrity and Ethics Training Program	V1M2 Sec. 4.2.8.1; 5.2.7		4.1.1
6.0	ACCOMMODATIONS AND ENVIRONMENTAL CONDITIONS	V1M2 Sec. 5.3		6.1; 6.3.1
6.1	Overview	V1M2 Secs. 5.3.1; 5.3.3; 5.3.4; 5.3.5	5.3.1; 5.3.3; 5.3.4; 5.3.5	6.3.1
6.2	Environment	V1M2 Secs. 5.3.1; 5.3.2; 5.3.3; 5.3.4; 5.3.5	5.3.1; 5.3.2; 5.3.3; 5.3.4; 5.3.5	6.3.1 to 6.3.5
6.3	Work Area	V1M2 Secs. 5.3.3; 5.3.4; 5.3.5	5.3.3; 5.3.4; 5.3.5	6.3.1
6.4	Responding to Emergencies			
6.5	Building Security	V1M2 Sec. 5.3.4	5.3.4	6.3.4
7.0	QUALITY SYSTEM			6.1; 8.2.4
7.1	Quality Policy	V1M2 Secs. 4.1.5; 4.2.2; 4.2.3; 4.2.8.3	4.1.5; 4.2.2; 4.2.3	8.2.3; 8.6.1
7.2	Ethics and Data Integrity	V1M2 Secs. 4.1.5; 4.16; 4.2.2; 4.2.8.1; 5.2.7	4.1.5; 4.2.2	4.1.1 to 4.1.3; 4.2.1; 6.2.1; 8.2.2; 8.2.3
7.3	Quality System Documentation	V1M2 Secs. 4.1.5; 4.2.2; 4.2.5	4.2.2; 4.2.5	8.2.4

Sec. No.	Title	2009 and 2016 TNI Standard Reference	ISO/IEC 17025:2005(E) Reference	ISO/IEC 17025:2017(E) Reference
7.4	QA/QC Objectives for the Measurement of Data	V1M2 Sec. 4.2.2	4.1.5; 4.2.2	6.2.4
7.5	Criteria for Quality Indicators			
7.6	Statistical Quality Control			
7.7	Quality System Metrics			
8.0	Document Control	V1M2 Secs. 4.2.7; 4.3.1; 4.3.2.2 ; 4.3.3.3; 4.3.3.4	4.2.7; 4.3.1; 4.3.2.2; 4.3.3.3; 4.3.3.4	8.2.4; 8.3.1
8.1	Overview			8.2.5; 8.3.1; 8.3.2
8.2	Document Approval and Issue	V1M2 Secs. 4.3.2; 4.3.2.1-4.3.2.3; 4.3.3.1	4.3.2.1; 4.3.2.2; 4.3.2.3; 4.3.3.1	8.2.5; 8.3.2
8.3	Procedures for Document Control Policy	V1M2 Secs. 4.3.2.1–4.3.2.2; 4.3.3.1	4.3.2.1; 4.3.2.2; 4.3.3.1	8.2.5; 8.3.2
8.4	Obsolete Documents	V1M2 Secs. 4.3.2.1–4.3.2.2	4.3.2.1; 4.3.2.2	8.2.5; 8.3.2
9.0	SERVICE TO THE CLIENT	V1M2 Secs. 4.4.1 - 4.4.4	4.4.1; 4.4.2; 4.4.3; 4.4.4	7.1.1; 7.1.1.4; 7.1.1.5; 7.1.1.8; 7.1.2.1
9.1	Overview	V1M2 Secs. 4.4.5; 4.5.5; 5.7.1	4.4.5; 5.7.1	
9.2	Review Sequence and Key Personnel	V1M2 Sec. 4.4.5	4.4.5	7.1.1.6
9.3	Balancing Laboratory Work Load and Capacity			
9.4	Documentation	V1M2 Sec. 5.7.1	5.7.1	
9.5	Special Services	V1M2 Secs. 4.7.1-4.7.2	4.7.1; 4.7.2	7.1.1.3; 7.1.1.7
9.6	Client Communication	V1M2 Secs. 4.7.1-4.7.2	4.7.1; 4.7.2	7.1.1.7
9.7	Reporting	V1M2 Secs. 4.7.1-4.7.2	4.7.1; 4.7.2	7.1.1.7
9.8	Client Feedback and Surveys	V1M2 Secs. 4.7.1-4.7.2	4.7.1; 4.7.2	7.1.1.7; 8.6.2
10.0	SUBCONTRACTING OF TESTS	V1M2 Secs. 4.4.3; 4.5.4	4.4.3; 4.5.4	
10.1	Overview	V1M2 Secs. 4.5.1 - 4.5.3; 4.5.5; 5.3.1	4.5.1; 4.5.2; 4.5.3; 5.3.1	6.6.1; 7.1.2.1; 7.1.2.2
10.2	Qualifying and Monitoring Subcontractors	V1M2 Secs. 4.5.1; 4.5.2; 4.5.3; 4.5.5	4.5.1; 4.5.2; 4.5.3	6.6.1; 7.1.2.1; 7.1.2.2
10.3	Oversight and Reporting	V1M2 Sec. 4.5.5		
10.4	Contingency Planning			
10.5	Use of NELAP and A2LA Logo			
11.0	PURCHASING SERVICES AND SUPPLIES	V1M2 Sec. 4.6.1	4.6.1	
11.1	Overview	V1M2 Secs. 4.6.2; 4.6.3; 4.6.4	4.6.2; 4.6.3; 4.6.4	6.6.1; 6.6.2
11.2	Glassware	V1M2 Sec. 5.5.13.1		
11.3	Reagents, Standards & Supplies	V1M2 Secs. 4.6.2; 4.6.3; 4.6.4 2016 V1M4 1.7.2.5	4.6.2; 4.6.3; 4.6.4	6.6.1 to 6.6.3
11.4	Purchase of Equipment / Instruments / Software			
11.5	Services			
11.6	Suppliers			
12.0	COMPLAINTS	V1M2 Sec. 4.8	4.8	8.6.1; 8.6.2
12.1	Overview			7.9.1 to 7.9.3 8.6.1; 8.6.2;
12.2	External Complaints			7.9.2 to 7.9.7 8.6.1; 8.6.2;
12.3	Internal Complaints			8.6.1; 8.6.2

Sec. No.	Title	2009 and 2016 TNI Standard Reference	ISO/IEC 17025:2005(E) Reference	ISO/IEC 17025:2017(E) Reference
12.4	Management Review			8.6.1; 8.6.2
13.0	CONTROL OF NON-CONFORMING WORK	V1M2 Secs. 4.9.1; 5.10.5	4.9.1; 5.10.5	7.10.1
13.1	Overview	V1M2 Secs. 4.9.1; 4.11.3; 4.11.5	4.9.1; 4.11.3; 4.11.5	7.10.1
13.2	Responsibilities and Authorities	V1M2 Secs. 4.9.1; 4.11.3; 4.11.5; 5.2.7	4.9.1; 4.11.3; 4.11.5	7.10.1
13.3	Evaluation of Significance and Action Taken	V1M2 Secs. 4.9.1; 4.11.3; 4.11.5	4.9.1; 4.11.3; 4.11.5	4.1.5; 7.10.1; 7.10.2; 8.5.3
13.4	Prevention of Non-Conforming Work	V1M2 Secs. 4.9.4; 4.11.2	4.9.2; 4.11.2	7.10.2; 7.10.3; 8.5.3
13.5	Method Suspension / Restriction (Stop Work Procedures)	V1M2 Secs. 4.9.1; 4.9.2; 4.11.5	4.9.1; 4.9.2; 4.11.5	7.10.1; 7.10.2
14.0	CORRECTIVE ACTION	V1M2 Sec. 4.11		4.1.4; 4.1.5
14.1	Overview	V1M2 Secs. 4.9.2; 4.11.1; 4.11.2	4.9.2; 4.11.1; 4.11.2; 8.7.1; 8.7.3	7.10.2 8.7.1; 8.7.3;
14.2	General	V1M2 Sec. 4.11.2; 4.11.3	4.11.2; 4.11.3	7.7.2; 8.5.3; 8.7.1;
14.3	Closed Loop Correction Action Process	V1M2 Sec. 4.11.2; 4.11.3; 4.11.4; 4.11.6; 4.11.7; 4.12.2	4.11.2; 4.11.3; 4.11.4; 4.12.2	8.5.3; 8.6.1; 8.7.2
14.4	Technical Corrective Actions	V1M2 Sec. 4.11.6		8.7.1
14.5	Basic Corrections	V1M2 Secs. 4.11.1; 4.13.2.3	4.11.1; 4.13.2.3	7.5.2; 8.7.1
15.0	PREVENTIVE ACTION / IMPROVEMENT	V1M2 Secs. 4.10; 4.12.1; 4.12.2	4.10; 4.12.1; 4.12.2	4.1.4
15.1	Overview	V1M2 Secs. 4.15.1; 4.15.2	4.15.1; 4.15.2	8.6.2
15.2	Management of Change			
16.0	CONTROL OF RECORDS	V1M2 Secs. 4.2.7; 4.13.1.1; 4.13.3	4.2.7; 4.13.1.1	8.4.2
16.1	Overview	V1M2 Secs. 4.13.1.1 - 4.13.1.4; 4.13.2.1-4.13.2.3; 4.13.3	4.13.1.1 - 4.13.1.4; 4.13.2.1-4.13.2.3	8.4.1; 8.4.2
16.2	Programs with Longer Retention Requirements			
16.3	Technical and Analytical Records	V1M2 Sec. 4.13.2.2 - 4.13.2.3	4.13.2.2; 4.13.2.3	7.5.1; 8.4.2
16.4	Laboratory Support Activities			7.5.2; 8.4.2
16.5	Administrative Records			8.4.2
16.6	Records Management, Storage and Disposal	V1M2 Sec. 4.13.3		4.2.1; 8.4.2
17.0	AUDITS			
17.1	Internal Audits	V1M2 Sec. 4.2.8.1; 4.14; 4.14.1; 4.14.2 ; 4.14.3; 4.14.5; 5.9.1; 5.9.2	4.14.1; 4.14.2; 4.14.3; 5.9.1; 5.9.2	8.6.1; 8.8.1; 8.8.2
17.2	External Audits	V1M2 Secs.4.14.2; 4.14.3	4.14.2; 4.14.3; 4.14.4	4.2.1; 8.6.1
17.3	Audit Findings	V1M2 Secs. 4.14.2; 4.14.3; 4.14.5		8.6.1
18.0	MANAGEMENT REVIEWS	V1M2 Sec. 4.1.6; 4.15; 4.15.1; 4.15.2	4.1.6; 4.15.1; 4.15.2	4.1.4; 8.5.1; 8.6.1; 8.9.1; 8.9.2
18.1	Quality Assurance Report			8.5.1

Sec. No.	Title	2009 and 2016 TNI Standard Reference	ISO/IEC 17025:2005(E) Reference	ISO/IEC 17025:2017(E) Reference
18.2	Annual Management Review	V1M2 Sec. 4.2.2; 4.15.3	4.2.2	4.1.1, 4.1.4; 4.2.1; 7.1.1.3; 8.2.2; 8.5.1 to 8.5.3; 8.6.1; 8.9.3
18.3	Potential Integrity Related Managerial Reviews			4.1.5; 8.5.1; 8.6.1
19.0	TEST METHODS AND METHOD VALIDATION	V1M2 Sec. 5.4.1	5.4.1	7.2.1.1; 8.2.5
19.1	Overview	V1M2 Sec. 5.4.1	5.4.1; 5.4.5.1	6.2.3; 7.2.1.1 to 7.2.1.3
19.2	Standard Operating Procedures (SOPs)	V1M2 Secs. 4.2.8.5; 4.3.3.1; 5.4.2	4.3.3.1; 5.4.2	7.2.1.4
19.3	Laboratory Methods Manual	V1M2 Sec. 4.2.8.5 (2019 5.4.4.2)		6.2.3
19.4	Selection of Methods	V1M2 Secs. 4.13.3; 5.4.1; 5.4.2; 5.4.3. (2019 5.4.4) V1M4 Secs. 1.4; 1.5.1; 1.6.1; 1.6.2; 1.6.2.1; 1.6.2.2	5.4.1; 5.4.2; 5.4.3; 5.4.4; 5.4.5.1; 5.4.5.2; 5.4.5.3	7.1.1.2; 7.2; 7.2.1.2; 7.2.1.3; 7.1.2.4 to 7.2.1.7; 7.2.2.1 to 7.2.1.7; 7.2.2.1
19.5	Laboratory Developed Methods and and Non-Standard Methods	V1M2 Sec. 5.4.2. (2019 5.4.4.1, 5.4.4.2) V1M4 Sec. 1.5.1	5.4.2; 5.4.4; 5.4.5.2; 5.4.5.3	7.1.1.2; 7.2.1.4 to 7.2.1.7 7.2.2.1; 7.2.2.3; 8.2.5
19.6	Validation of Methods	V1M2 Sec. 5.4.2. (2019 5.4.5.4) V1M4 Secs. 1.5.1; 1.5.2; 1.5.2.1; 1.5.2.2; 1.5.3	5.4.2; 5.4.4; 5.4.5.2; 5.4.5.3	7.1.1.2; 7.1.2.4; 7.2.1.6; 7.2.2.1 to 7.2.2.4
19.7	Method Detection Limits (MDLs) / Limit of Detection (LOD)	V1M2 Sec. 5.9.3. V1M4 Secs. 1.5.2; 1.5.2.1; 1.5.2.2	5.4.5.3	7.2.2.3
19.8	Verification of Detection Limits	V1M2 Sec. 5.9.3		
19.9	Instrument Detection Limits (IDLs)	V1M2 Sec. 5.9.3. V1M4 Sec. 1.5.2.1		
19.10	Limit of Quantitation	V1M2 Sec. 5.9.3		
19.11	Retention Windows	V1M2 Sec. 5.9.3. V1M4 Sec. 1.5.4; (2009 1.7.3.6) (2016 1.7.2.6)		
19.12	Evaluation of Selectivity	V1M2 Sec. 5.9.3. V1M4 Sec. 1.5.4; (2009 1.7.3.6) (2016 1.7.2.6)		
19.13	Estimation of Uncertainty of Measurement	V1M2 Sec. 5.1.1; 5.1.2; 5.4.6	5.1.1; 5.1.2; 5.4.6.1; 5.4.6.2; 5.4.6.3	7.6.1; 7.6.2; 7.6.3
19.14	Sample Reanalysis Guidelines	V1M2 Sec 5.9.1	5.9.1	
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24.4	Positive Controls	V1M2 Secs 5.9.2; 5.9.3. V1M4 Secs.(2009 1.7.3; 1.7.3.2; 1.7.3.2.1; 1.7.3.2.2; 1.7.3.2.3) (2016 1.7.2, 1.7.2.2, 1.7.3.2)	5.9.2	7.7.1; 7.7.3
24.5	Sample Matrix Controls	V1M2 Secs. 5.9.2; 5.9.3. V1M4 Secs. (2009 1.7.3 ; 1.7.3.3; 1.7.3.3.1; 1.7.3.3.2; 1.7.3.3.3) (2016 1.7.2, 1.7.2.3, 1.7.3.3)	5.9.2	7.7.1; 7.7.3
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24.7	Additional Procedures to Assure Quality Control	V1M2 Sec. 5.9.3. V1M4 Sec. (2009 1.7.3.4), (2016 1.7.2.4)		7.7.1; 7.7.3
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25.2	Test Reports	V1M2 Secs. 5.10.1; 5.10.2; 5.10.3.1; 5.10.3.2; 5.10.5; 5.10.6; 5.10.7; 5.10.8; 5.10.10; 5.10.11	5.10.1; 5.10.2; 5.10.3.1; 5.10.3.2; 5.10.5; 5.10.6; 5.10.7; 5.10.8	7.8.2.1; 7.8.2.2; 7.8.3; 7.8.5
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25.6	Environmental Testing Obtained from Subcontractors	V1M2 Secs. 4.5.5; 5.10.1; 5.10.6	5.10.1; 5.10.6	
25.7	Client Confidentiality	V1M2 Secs. 4.1.5; 5.10.7	4.1.5; 5.10.7	4.2.1 to 4.2.4
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Appendix 5. Terms/Glossary and Acronyms (EL-V1M2 Sec. 3.1)

Terms/Glossary:

Acceptance Criteria: Specified limits placed on characteristics of an item, process, or service defined in requirement documents. (ASQC)

Accreditation: The process by which an agency or organization evaluates and recognizes a laboratory as meeting certain predetermined qualifications or standards, thereby accrediting the laboratory.

Accuracy: The degree of agreement between an observed value and an accepted reference value. Accuracy includes a combination of random error (precision) and systematic error (bias) components which are due to sampling and analytical operations; a data quality indicator. (QAMS)

Analyst: The designated individual who performs the "hands-on" analytical methods and associated techniques and who is the one responsible for applying required laboratory practices and other pertinent quality controls to meet the required level of quality.

Analytical Uncertainty: A subset of Measurement Uncertainty that includes all laboratory activities performed as part of the analysis. (TNI)

Anomaly: A condition or event, other than a deficiency, that may affect the quality of the data, whether in the laboratory's control or not.

Assessment: The evaluation process used to measure or establish the performance, effectiveness, and conformance of an organization and/or its systems to defined criteria (to the standards and requirements of laboratory accreditation). (TNI)

Audit: A systematic and independent examination of facilities, equipment, personnel, training, procedures, record-keeping, data validation, data management, and reporting aspects of a system to determine whether QA/QC and technical activities are being conducted as planned and whether these activities will effectively achieve quality objectives. (TNI)

Batch: Environmental samples that are prepared and/or analyzed together with the same process and personnel, using the same lot(s) of reagents. A **preparation batch** is composed of one (1) to twenty (20) environmental samples of the same quality systems matrix, meeting the above mentioned criteria and with a maximum time between the start of processing of the first and last sample in the batch to be twenty-four (24) hours. An **analytical batch** is composed of prepared environmental samples (extracts, digestates or concentrates) which are analyzed together as a group. An analytical batch can include prepared samples originating from various quality system matrices and can exceed twenty (20) samples. (TNI)

Bias: The systematic or persistent distortion of a measurement process, which causes errors in one direction (i.e., the expected sample measurement is different from the sample's true value). (TNI)

Blank: A sample that has not been exposed to the analyzed sample stream in order to monitor contamination during sampling, transport, storage or analysis. The blank is subjected to the usual analytical and measurement process to establish a zero baseline or background value and is sometimes used to adjust or correct routine analytical results. (ASQC)

Calibration: A set of operations that establish, under specified conditions, the relationship between values of quantities indicated by a measuring instrument or measuring system, or values represented by a material measure or a reference material, and the corresponding values realized by standards. (TNI)

1) In calibration of support equipment the values realized by standards are established through the use of reference standards that are traceable to the International System of Units (SI).

2) In calibration according to methods, the values realized by standards are typically established through the use of Reference Materials that are either purchased by the laboratory with a certificate of analysis or purity, or prepared by the laboratory using support equipment that has been calibrated or verified to meet specifications.

Calibration Curve: The mathematical relationship between the known values, such as concentrations, of a series of calibration standards and their instrument response. (TNI)

Calibration Standard: A substance or reference material used to calibrate an instrument (QAMS)

Certified Reference Material (CRM): A reference material accompanied by a certificate, having a value, measurement uncertainty, and stated metrological traceability chain to a national metrology institute. (TNI)

Chain of Custody (COC) Form: Record that documents the possession of the samples from the time of collection to receipt in the laboratory. This record generally includes: the number and types of containers; the mode of collection; the collector; time of collection; preservation; and requested analyses. (TNI)

Compromised Samples: Those samples which are improperly sampled, insufficiently documented (chain of custody and other sample records and/or labels), improperly preserved, collected in improper containers, or exceeding holding times when delivered to a laboratory. Under normal conditions, compromised samples are not analyzed. If emergency situation require analysis, the results must be appropriately qualified.

Confidential Business Information (CBI): Information that an organization designates as having the potential of providing a competitor with inappropriate insight into its management, operation or products. TNI and its representatives agree to safeguard identified CBI and to maintain all information identified as such in full confidentiality.

Confirmation: Verification of the identity of a component through the use of an approach with a different scientific principle from the original method. These may include, but are not limited to Second Column Confirmation; Alternate wavelength; Derivatization; Mass spectral interpretation; Alternative detectors or Additional Cleanup procedures. (TNI)

Conformance: An affirmative indication or judgment that a product or service has met the requirements of the relevant specifications, contract, or regulation; also the state of meeting the requirements. (ANSI/ASQC E4-1994)

Correction: Actions necessary to correct or repair analysis specific non-conformances. The acceptance criteria for method specific QC and protocols as well as the associated corrective actions. The analyst will most frequently be the one to identify the need for this action as a result of calibration checks and QC sample analysis**.** No significant action is taken to change behavior, process or procedure.

Corrective Action: The action taken to eliminate the causes of an existing nonconformity, defect or other undesirable situation in order to prevent recurrence. (ISO 8402)

Data Integrity: The condition that exists when data are sound, correct, and complete, and accurately reflect activities and requirements. (TNI)

Data Audit: A qualitative and quantitative evaluation of the documentation and procedures associated with environmental measurements to verify that the resulting data re of acceptable quality (i.e., that they meet specified acceptance criteria).

Data Reduction: The process of transforming the number of data items by arithmetic or statistical calculations, standard curves, and concentration factors, and collation into a more useable form. (TNI)

Deficiency: An unauthorized deviation from acceptable procedures or practices, or a defect in an item (ASQC), whether in the laboratory's control or not.

Demonstration of Capability: A procedure to establish the ability of the analyst to generate analytical results of acceptable accuracy and precision. (TNI)

Document Control: The act of ensuring that documents (and revisions thereto) are proposed, reviewed for accuracy, approved for release by authorized personnel, distributed properly, and controlled to ensure use of the correct version at the location where the prescribed activity if performed. (ASQC)

Duplicate Analyses: The analyses or measurements of the variable of interest performed identically on two subsamples of the same sample. The results from duplicate analyses are used to evaluate analytical or measurement precision but not the precision of sampling, preservation or storage internal to the laboratory. (EPA-QAD)

Equipment Blank: Sample of analyte-free media which has been used to rinse common sampling equipment to check effectiveness of decontamination procedures.

External Standard Calibration: Calibrations for methods that do not utilize internal standards to compensate for changes in instrument conditions.

Field Blank: Blank prepared in the field by filing a clean container with pure de-ionized water and appropriate preservative, if any, for the specific sampling activity being undertaken (EPA OSWER)

Field of Accreditation: Those matrix, technology/method, and analyte combinations for which the accreditation body offers accreditation.

Holding Times: The maximum time that samples may be held prior to analyses and still be considered valid or not compromised. (40 CFR Part 136)

Internal Standard: A known amount of standard added to a test portion of a sample as a reference for evaluating and controlling the precision and bias of the applied analytical test method. (TNI)

Internal Standard Calibration: Calibrations for methods that utilize internal standards to compensate for changes in instrument conditions.

Instrument Blank: A clean sample (e.g., distilled water) processed through the instrumental steps of the measurement process; used to determine instrument contamination. (EPA-QAD)

Instrument Detection Limit (IDL): The minimum amount of a substance that can be measured with a specified degree of confidence that the amount is greater than zero using a specific instrument. The IDL is associated with the instrumental portion of a specific method only, and sample preparation steps are not considered in its derivation. The IDL is a statistical estimation at a specified confidence interval of the concentration at which the relative uncertainty is ± 100%. The IDL represents a range where qualitative detection occurs on a specific instrument. Quantitative results are not produced in this range.

Laboratory Control Sample (however named, such as laboratory fortified blank, spiked blank, or QC check sample): A sample matrix, free from the analytes of interest, spiked with verified known amounts of analytes or a material containing known and verified amounts of analytes, taken through all preparation and analysis steps of the procedure unless otherwise noted in a reference method. It is generally used to establish intra-laboratory or analyst specific precision and bias or to assess the performance of all or a portion of the measurement system.

An LCS shall be prepared at a minimum of 1 per batch of 20 or less samples per matrix type per sample extraction or preparation method except for analytes for which spiking solutions are not available such as total suspended solids, total dissolved solids, total volatile solids, total solids, pH, color, odor, temperature, dissolved oxygen or turbidity. The results of these samples shall be used to determine batch acceptance.

Least Squares Regression (1st Order Curve): The least squares regression is a mathematical calculation of a straight line over two axes. The y axis represents the instrument response (or Response ratio) of a standard or sample and the x axis represents the concentration. The regression calculation will generate a correlation coefficient (r) that is a measure of the "goodness of fit" of the regression line to the data. A value of 1.00 indicates a perfect fit. In order to be used for quantitative purposes, r must be greater than or equal to 0.99 for organics and 0.995 for inorganics.

Limit(s) of Detection (LOD) [a.k.a., Method Detection Limit (MDL)]: The MDL is the minimum measured quantity of a substance that can be reported with 99% confidence that the concentration is distinguishable from method blank results, consistent with 40CFR Part 136 Appendix B, August, 2017.

Limit(s) of Quantitation (LOQ) [a.k.a., Reporting Limit]: The minimum levels, concentrations, or quantities of a target variable (e.g., target analyte) that can be reported with a specified degree of confidence.

(QS) Matrix: The component or substrate that contains the analyte of interest. For purposes of batch and QC requirement determinations, the following matrix distinctions shall be used:

Aqueous: Any aqueous sample excluded from the definition of Drinking Water or Saline/Estuarine. Includes surface water, groundwater effluents, and TCLP or other extracts.

Drinking Water: Any aqueous sample that has been designated as a potable or potential potable water source.

Saline/Estuarine: Any aqueous sample from an ocean or estuary, or other salt water source such as the Great Salt Lake.

Non-Aqueous Liquid: Any organic liquid with <15% settleable solids.

Biological Tissue: Any sample of a biological origin such as fish tissue, shellfish, or plant material. Such samples shall be grouped according to origin.

Solids: Includes soils, sediments, sludges, and other matrices with >15% settleable solids.

Chemical Waste: A product or by-product of an industrial process that results in a matrix not previously defined.

Air & Emissions: Whole gas or vapor samples including those contained in flexible or rigid wall containers and the extracted concentrated analytes of interest from a gas or vapor that are collected with a sorbant tube, impinger solution, filter, or other device. (TNI)

Matrix Spike (spiked sample or fortified sample): A sample prepared, taken through all sample preparation and analytical steps of the procedure unless otherwise noted in a referenced method, by adding a known amount of target analyte to a specified amount of sample for which an independent test result of target analyte concentration is available. Matrix spikes are used, for example, to determine the effect of the matrix on a method's recovery efficiency.

Matrix Spike Duplicate (spiked sample or fortified sample duplicate): A replicate matrix spike prepared and analyzed to obtain a measure of the precision of the recovery for each analyte.

Method Blank: A sample of a matrix similar to the batch of associated samples (when available) that is free from the analytes of interest and is processed simultaneously with and under the same conditions as samples through all steps of the analytical procedures, and in which no target analytes or interferences are present at concentrations that impact the analytical results for sample analyses.

Method Detection Limit: See Limit of Detection (LOD)

Negative Control: Measures taken to ensure that a test, its components, or the environment do not cause undesired effects, or produce incorrect test results.

Non-conformance: An indication, judgment, or state of not having met the requirements of the relevant specifications, contract, or regulation.

Observation: A record of phenomena that (1) may assist in evaluation of the sample data; (2) may be of importance to the project manager and/or the client, and yet not at the time of the observation have any known effect on quality.

Performance Audit: The routine comparison of independently obtained qualitative and quantitative measurement system data with routinely obtained data in order to evaluate the proficiency of an analyst or laboratory.

Positive Control: Measures taken to ensure that a test and/or its components are working properly and producing correct or expected results from positive test subjects.

Precision: The degree to which a set of observations or measurements of the same property, obtained under similar conditions, conform to themselves; a data quality indicator. Precision is usually expressed as standard deviation, variance or range, in either absolute or relative terms. (TNI)

Preservation: Any conditions under which a sample must be kept in order to maintain chemical and/or biological integrity prior to analysis. (TNI)

Proficiency Testing: A means of evaluating a laboratory's performance under controlled conditions relative to a given set of criteria through analysis of unknown samples provided by an external source. (TNI)

Proficiency Testing Program: The aggregate of providing rigorously controlled and standardized environmental samples to a laboratory for analysis, reporting of results, statistical evaluation of the results and the collective demographics and results summary of all participating laboratories. (TNI)

Proficiency Test Sample (PT): A sample, the composition of which is unknown to the laboratory and is provided to test whether the laboratory can produce analytical results within specified acceptance criteria. (TNI)

Quality Assurance: An integrated system of management activities involving planning, implementation, assessment, reporting and quality improvement to ensure that a process, item or service is of the type of quality needed and expected by the client. (TNI)

Quality Assurance [Project] Plan (QAPP): A formal document describing the detailed quality control procedures by which the quality requirements defined for the data and decisions pertaining to a specific project are to be achieved. (EAP-QAD)

Quality Control: The overall system of technical activities that measures the attributes and performance of a process, item, or service against defined standards to verify that they meet the stated requirements established by the customer; operational techniques and activities that are used to fulfill requirements for quality; also the system of activities and checks used to ensure that measurement systems are maintained within prescribed limits, providing protection against "out of control" conditions and ensuring that the results are of acceptable quality. (TNI)

Quality Control Sample: A sample used to assess the performance of all or a portion of the measurement system. One of any number of samples, such as Certified Reference Materials, a quality system matrix fortified by spiking, or actual samples fortified by spiking, intended to demonstrate that a measurement system or activity is in control. (TNI)

Quality Manual: A document stating the management policies, objectives, principles, organizational structure and authority, responsibilities, accountability, and implementation of an agency, organization, or laboratory, to ensure the quality of its product and the utility of its product to its users. (TNI)

Quality System: A structured and documented management system describing the policies, objectives, principles, organizational authority, responsibilities, accountability, and implementation plan of an organization for ensuring quality in its work processes, products (items), and services. The quality system provides the framework for planning, implementing, and assessing work performed by the organization and for carrying out required QA and QC activities. (TNI)

Raw Data: The documentation generated during sampling and analysis. This documentation includes, but is not limited to, field notes, electronic data, magnetic tapes, untabulated sample results, QC sample results, print outs of chromatograms, instrument outputs, and handwritten records. (TNI)

Record Retention: The systematic collection, indexing and storing of documented information under secure conditions.

Reference Material: Material or substance one or more properties of which are sufficiently homogeneous and well established to be used for the calibration of an apparatus, the assessment of a measurement method, or for assigning values to materials. (TNI)

Reference Standard: Standard used for the calibration of working measurement standards in a given organization or a given location. (TNI)

Sampling: Activity related to obtaining a representative sample of the object of conformity assessment, according to a procedure.

Second Order Polynomial Curve (Quadratic): The 2nd order curves are a mathematical calculation of a slightly curved line over two axis. The y axis represents the instrument response (or Response ratio) of a standard or sample and the x axis represents the concentration. The 2nd order regression will generate a coefficient of determination (COD or r^2) that is a measure of the "goodness of fit" of the quadratic curvature the data. A value of 1.00 indicates a perfect fit. In order to be used for quantitative purposes, r^2 must be greater than or equal to 0.99.

Selectivity: The ability to analyze, distinguish, and determine a specific analyte or parameter from another component that may be a potential interferent or that may behave similarly to the target analyte or parameter within the measurement system. (TNI)

Sensitivity: The capability of a method or instrument to discriminate between measurement responses representing different levels (e.g., concentrations) of a variable of interest. (TNI)

Spike: A known mass of target analyte added to a blank, sample or sub-sample; used to determine recovery efficiency or for other quality control purposes.

Standard: The document describing the elements of laboratory accreditation that has been developed and established within the consensus principles of standard setting and meets the approval requirements of standard adoption organizations procedures and policies. (TNI)

Standard Operating Procedures (SOPs): A written document which details the method for an operation, analysis, or action, with thoroughly prescribed techniques and steps. SOPs are officially approved as the methods for performing certain routine or repetitive tasks. (TNI)

Storage Blank: A blank matrix stored with field samples of a similar matrix (volatiles only) that measures storage contribution to any source of contamination.

Surrogate: A substance with properties that mimic the analyte of interest. It is unlikely to be found in environment samples and is added to them for quality control purposes.

Surrogate compounds must be added to all samples, standards, and blanks, for all organic chromatography methods except when the matrix precludes its use or when a surrogate is not available. Poor surrogate recovery may indicate a problem with sample composition and shall be reported to the client whose sample produced poor recovery. (QAMS)

Systems Audit (also Technical Systems Audit): A thorough, systematic, qualitative on-site assessment of the facilities, equipment, personnel, training, procedures, record keeping, data validation, data management, and reporting aspects of a total measurement system. (EPA-QAD)

Technical Manager: A member of the staff of an environmental laboratory who exercises actual day-to-day supervision of laboratory operations for the appropriate fields of accreditation and reporting of results

Technology: A specific arrangement of analytical instruments, detection systems, and/or preparation techniques.

Traceability: The ability to trace the history, application, or location of an entity by means of recorded identifications. In a calibration sense, traceability relates measuring equipment to national or international standards, primary standards, basic physical constants or properties, or reference materials. In a data collection sense, it relates calculations and data generated throughout the project back to the requirements for the quality of the project. (TNI)

Trip Blank: A blank matrix placed in a sealed container at the laboratory that is shipped, held unopened in the field, and returned to the laboratory in the shipping container with the field samples.

Uncertainty: A parameter associated with the result of a measurement that characterizes the dispersion of the value that could reasonably be attributed to the measured value.

Acronyms:

CAR – Corrective Action Report

CCV – Continuing Calibration Verification

CF – Calibration Factor

CFR – Code of Federal Regulations

COC – Chain of Custody

DOC – Demonstration of Capability

DQO – Data Quality Objectives

DUP - Duplicate

EHS – Environment, Health and Safety

EPA – Environmental Protection Agency

GC - Gas Chromatography

GC/MS - Gas Chromatography/Mass Spectrometry

HPLC - High Performance Liquid Chromatography

ICP - Inductively Coupled Plasma Atomic Emission Spectroscopy

ICP/MS – ICP/Mass Spectrometry

ICV – Initial Calibration Verification

IDL – Instrument Detection Limit

IH – Industrial Hygiene

IS – Internal Standard
LCS – Laboratory Control Sample
LCSD – Laboratory Control Sample Duplicate
LIMS – Laboratory Information Management System
LOD – Limit of Detection
LOQ – Limit of Quantitation
MDL – Method Detection Limit
MDLCK – MDL Check Standard
MDLV – MDL Verification Check Standard
MRL – Method Reporting Limit Check Standard
MS – Matrix Spike
MSD – Matrix Spike Duplicate
SDS - Safety Data Sheet
NELAP - National Environmental Laboratory Accreditation Program
PT – Performance Testing
TNI – The NELAC Institute
QAM – Quality Assurance Manual
QA/QC – Quality Assurance / Quality Control
QAPP – Quality Assurance Project Plan
RF – Response Factor
RPD – Relative Percent Difference
RSD – Relative Standard Deviation
SD – Standard Deviation
SOP – Standard Operating Procedure
TAT – Turn-Around-Time
VOA – Volatiles
VOC – Volatile Organic Compound

Appendix 6. Analytical Method References

Reference methods include:

- *Guidelines Establishing Test Procedures for the Analysis of Pollutants Under the Clean Water Act; Analysis and Sampling Procedures; 40CFR Part 136 as amended by Method Update Rule; August 28, 2017.*
- *Methods for Chemical Analysis of Water and Wastes, EPA 600 (4-79-020), 1983.*
- *Methods for the Determination of Inorganic Substances in Environmental Samples, EPA-600/R-93/100, August 1993.*
- *Methods for the Determination of Metals in Environmental Samples, EPA/600/4-91/010, June 1991. Supplement I: EPA-600/R-94/111, May 1994.*
- *Methods for the Determination of Organic Compounds in Drinking Water, EPA-600/4-88-039, December 1988, Revised, July 1991, Supplement I, EPA-600-4-90-020, July 1990, Supplement II, EPA-600/R-92-129, August 1992. Supplement III EPA/600/R-95/131 - August 1995 (EPA 500 Series) (EPA 500 Series methods)*
- *Technical Notes on Drinking Water Methods, EPA-600/R94-173, October 1994*
- *Statement of Work for Inorganics & Organics Analysis, SOM and ISM, current versions, USEPA Contract Laboratory Program Multi-media, Multi-concentration.*
- *Standard Methods for the Examination of Water and Wastewater, 18th/19th /20th/ on-line edition; Eaton, A.D. Clesceri, L.S. Greenberg, A.E. Eds; American Water Works Association, Water Pollution Control Federation, American Public Health Association: Washington, D.C.*
- *Test Methods for Evaluating Solid Waste Physical/Chemical Methods (SW846), Third Edition, September 1986, Final Update I, July 1992, Final Update IIA, August 1993, Final Update II, September 1994; Final Update IIB, January 1995; Final Update III, December 1996; Final Update IV, January 2008; Final Update V, August 2015.*
- *Annual Book of ASTM Standards, American Society for Testing & Materials (ASTM), Philadelphia, PA.*
- *Manual for the Certification of Laboratories Analyzing Drinking Water (EPA 815-R-05-004, January 2005)*
- *Code of Federal Regulations (CFR) 40, Parts 136, 141, 172, 173, 178, 179 and 261*