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SAMPLING AND ANALYSIS PLAN

Vacant 7-Acre Parcel
APN: 002-770-005
Wells
Elko County
Nevada
NDEP Contract #10-008-04
Task M02-13

Prepared for:

State of Nevada
Department of Conservation and Natural Resources
Division of Environmental Protection
Bureau of Corrective Actions
901 S. Stewart Street, Suite 4001
Carson City, Nevada 89701-5249

On behalf of:

City of Wells

January 24, 2013

Sampling and Analysis Plan for:

Limited Phase II Environmental Site Assessment for
APN: 002-770-005
Wells, Nevada

January 31, 2013

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Mary Siders

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Coordinator: _____
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1. INTRODUCTION

McGinley and Associates, Inc. (MGA) has prepared this Sampling and Analysis Plan (SAP) for assessment activities to be conducted on a vacant property located in Wells, Nevada. These assessment activities are being funded by the State of Nevada Brownfields Program. This SAP was prepared in accordance with the Nevada Division of Environmental Protection (NDEP) Quality Assurance Program Plan (QA Program Plan) for the Nevada Brownfields Program (NBP) (NDEP, 2007).

This SAP addresses the field sampling, analytical, quality control, and data review procedures for the collection and analysis of soil and groundwater samples to evaluate contamination from orphaned underground storage tanks at the site and past hydrocarbon releases from former service stations and truck stops located in the vicinity.

1.1 Site Name

Vacant Property: 7-Acre Parcel.

1.2 Site Location and Description

The subject property consists of a vacant seven-acre parcel of land located in Wells, Nevada. The parcel is listed with Elko County, Nevada as Assessor's Parcel Number (APN) 002-770-005. Geographically, the subject property is located in the N ½ of the SW ¼ of Section 10, Township 37 North, Range 62 East of the Mount Diablo Base and Meridian (MDB&M). The location of the site is indicated on Figure 1.

The subject property is a vacant unoccupied parcel of land approximately seven acres in size. Access to the property can be gained from 6th Street to the southwest. The parcel has a slight grade and generally slopes to the north-northeast. The layout of the subject property is illustrated on Figure 2. Notable findings observed during a Phase I ESA site visit included: two cover plate fixtures containing buried conduit; construction material debris; several 55-gallon drums; and one abandoned rusted rectangular tank, approximately 80 gallons in volume. The debris consisted of concrete rubble, bricks, rusted pipes, apparent wood beams/columns, and other similar items that appeared to be evidence of a former building structure or multiple building structures. One area of the debris consisted of concrete sections with metal bollards near two cover plate fixtures containing buried conduit. The shape of the concrete sections with metal bollards appeared similar to construction materials used for a former fuel island.

1.3 Responsible Agency

This project is being conducted for the NDEP through State of Nevada Brownfields program (NBP). The investigation will conform to the NBP's QA Program Plan (NDEP, 2007).

1.4 Project Organization

Title/Responsibility	Name	Phone
City of Wells		
Site Contact	Jolene Supp	(775) 752-3355
NDEP		
Program Coordinator for the Nevada Brownfields Program – Project coordination, liaison with City of Wells	Jeff Collins	(775) 687-9381
Case Officer – Review SAP, quality assurance	David Friedman	(775) 687-9385
Quality Coordinator for the Nevada	Mary Siders	(775) 687-9496

Brownfields Program – Review SAP, quality assurance		
USEPA		
USEPA Project Manager – Work plan review	Eugenia Chow	(415) 415-972-3160
USEPA QA Manager	Gail Morison	(415) 972-3807
McGinley and Associates, Inc.		
Principal – Senior review	Joe McGinley	(775) 829-2245
Project Manager – Project management, regulatory liaison, coordinate field activities, data review, report preparation.	Brett Bottenberg	(702) 260-4961
Quality Manager – Oversee implementation of SAP, review QA/QC procedures, data validation.	Brian Rakvica	(702) 260-4961
Environmental Scientist – Conduct sampling activities	Justin Fike	(775) 829-2245
GIS Services – Mapping support	Mike Parenti	(702) 260-4961
Administrative Assistant – Administrative support	Linda Comstock	(775) 829-2245
Contractors/Vendors		
ESC Lab Sciences	Dave Veratti	(615) 758-5858
GPRS	Jim Cox	(775) 560-8913
Earth Probe Environmental Field Services	Patrick Casey	(801) 466-3752

1.5 Statement of the Specific Problem

The City of Wells has proposed to redevelop the subject property with a visitor center and convention center. In 2010, the property was turned over to the City of Wells by the former owner, Century Casinos Nevada. Under the State of Nevada Brownfields Program, a Phase I ESA was performed on the property in 2012.

The Phase I ESA performed by MGA on the subject property discovered the following recognized environmental conditions (RECs):

- Potential orphaned USTs at the subject property; and
- Past hydrocarbon release events at the Four-Way Truck Stop, Former Chevron Service Station, and Former Fearless Farris Stinker Station.

These RECs will need to be addressed prior to redevelopment of the property.

2. BACKGROUND

According to the Elko County, Nevada Assessor's Office, the subject property comprises an area of approximately seven acres on a single parcel of land. Available records indicate no building structures are located on the property. The subject property is listed as Assessor's Parcel Number (APN) 002-770-005 and is currently owned by the City of Wells. Prior owners were listed as Century Casino Nevada, Inc. from 2001 to 2009 and Chevron USA, Inc. from an unknown date to 2001.

Review of historical aerial photographs, depicts the subject property as undeveloped in 1980, 1994, 1999 and 2006. However, for the year 1967, several shadows observed in the photo imply one or several onsite structures existed on the subject property. Unfortunately, it is difficult to discern details of the structure(s), due to the scale and lack of clarity of this photograph. In the

1980, 1994, 1999, and 2006 aerial photographs reviewed, the subject property and surrounding properties appear consistent with observations made during the Phase I ESA site reconnaissance.

2.1 Sampling Area Description

The study area (the Site) occupies 7 acres in Wells, Nevada (Figure 1). The area is bounded on the north by commercial property with one building, the south by a gas station with convenience store, the east by commercial property with several buildings, and to the west by 6th Street with several commercial properties beyond. As shown on Figure 2, the Site is a vacant parcel.

2.2 Operational History

Based on available historical information, it appears that the site was developed in the past. Aerial photographs suggest that the site may have been developed with several structures in 1967. However, the photograph is not clear and it is difficult to discern details of the structures.

In addition, ownership information provided by the Elko County Assessor's Office appears to indicate that the property may have formerly been a gas station. Chevron USA, Inc. is listed as an owner up until 2001. The number of years owned by Chevron prior to 2001 is unknown.

2.3 Previous Investigations/Regulatory Involvement

In May of 2012, MGA conducted a Phase I ESA on the study area. The ESA was conducted in compliance with the American Society for Testing and Materials (ASTM) Standard E-1527-05 to identify any recognized environmental conditions (RECs) at the Site. The proposed sampling assessment is based on the findings of this Phase I ESA which are previously presented in Section 1.5 of this Sampling and Analysis Plan.

2.4 Geological Information

The geology of the subject property has been mapped as Quaternary Alluvium (Coats, 1975). The deposits are described as relatively thin, coarse, and poorly sorted silt, sand, and gravel. Surface soils at the subject site have been mapped as Valmy-Enko association with slopes ranging from zero to four percent. The soil unit is described as well drained with moderately low to high permeability and moderate to high water capacity (Natural Resources Conservation Service, 2011).

Groundwater conditions on the subject property have not been positively ascertained. However, information obtained from files available for review (Facility ID #6-000029) at the Nevada Division of Environmental Protection (NDEP) and an online database maintained by the Nevada Division of Water Resources (DWR), indicates that depth to groundwater ranges from approximately 5 to 15 feet below ground surface (fbgs). No wells were observed on the subject property; however, the DWR online database shows multiple groundwater monitoring wells potentially located on the subject property and at the Shell gas station to the south. Groundwater flow direction appears to be generally towards the north-northeast.

2.5 Environmental and/or Human Impact

No adverse human health effects associated with potential contamination at this site have been reported or documented. However, the potential exists for receptors to interact with the soils once disturbed.

3. PROJECT DATA QUALITY OBJECTIVES

3.1 Project Task and Problem Definition

The purpose of this investigation is to assess the soil and groundwater for contamination from historical site use, orphaned USTs, and off-site leaking underground storage tanks (LUSTs). Definitive data will be collected to determine the extent of soil and groundwater contamination, if any.

3.2 Data Quality Objectives (DQOs)

The DQO process (EPA 2006) is a systematic planning tool that is used to establish performance or acceptance criteria. These criteria, in turn, serve as the basis for designing a plan for collecting data of sufficient quality and quantity to support the goals of a study. The DQO process consists of seven iterative steps, as described in the following sections and summarized in Table 1.

3.2.1 Step 1: State the Problem

Possible historic use of the site as a gas station and evidence of a former fueling island may indicate that orphaned USTs still exist on the subject property. In addition, LUSTs on other properties located adjacent or in the immediate vicinity may have impacted the groundwater beneath the subject property. However, the nature and extent of contamination in the soils and groundwater, if any, is not known. Additional data are needed to define the nature and extent, if any, of contamination within the soil and groundwater.

3.2.2 Step 2: Identify Decisions

Analytical data for collected samples will be evaluated to determine if concentrations of contaminants of concern (COC) exceed Nevada reportable concentrations (RCs). Analytical data will be compared to reportable concentrations (RCs) as published in the State of Nevada Division of Environmental Protection (NDEP) Draft Guidelines for Discovery Events document (NAC 445A.345 to 445A.348 as modified by adopted regulation R-189-08). Results of the investigation will be used to determine if additional assessment and/or regulatory notification with subsequent clean-up are required.

3.2.3 Step 3: Identify Inputs

Information required to address project objectives includes historical data, proposed quantitative data to be collected under this study, and soil/groundwater RCs. Analytical testing of soil and groundwater samples shall be conducted by ESC Lab Sciences of Mount Juliet, Tennessee. ESC's DQOs for the analytical testing are provided in Appendix A.

3.2.4 Step 4: Define Study Boundaries

The proposed investigation shall extend from the surface to groundwater. The duration of the assessment activities described in this SAP is approximately one week.

3.2.5 Step 5: Develop Decision Rules

Decision rules are specified in Table 1, and describe actions based on qualitative and definitive data. Laboratory analytical data for the sampled media will be compared to State of Nevada RCs. For contaminants detected above RCs, Nevada statutes require that site-specific action levels be an appropriate level of concentration that is based on the protection of public health and safety and the environment as determined through the use of the Integrated Risk Information System (IRIS) adopted by the USEPA, to be used when inhalation, ingestion, or dermal exposure is the primary exposure pathway; or a background concentration.

3.2.6 Step 6: Specify Tolerable Limits on Decision Errors

This is not a statistically based study; therefore, sampling locations will be selected based on professional judgment and site knowledge.

3.2.7 Step 7: Optimize the Sampling Design

Optimization was completed via discussions with the project team and by reviewing historical information indicating locations of potential contamination. The number of samples selected is believed to be adequate to complete an initial assessment of site conditions.

The DQOs are summarized in Table 1. Analytical testing of samples shall be conducted by Alpha, as noted above. Alpha's DQOs for the analytical testing are provided in Appendix A.

3.3 Data Quality Indicators (DQIs)

Data quality indicators (precision, accuracy, representativeness, completeness, comparability and sensitivity [i.e., PARCCS parameters]) refer to quality control criteria established for various aspects of data gathering, sampling, and/or analyses. Precision is the degree of mutual agreement between or among independent measurements of a similar property (usually reported as standard deviation (SD) or relative percent difference) and relates to the analysis of duplicate laboratory or field samples. Accuracy is the degree of agreement of a measurement with a known or true value and is determined by comparing the reported laboratory value for a sample to a known or true concentration (i.e. matrix spikes, surrogate spikes, laboratory control samples and performance samples). Representativeness is the expression of the degree to which data accurately and precisely represent a characteristic of an environmental condition or population and relates to the method of collecting samples and determining sampling locations. Completeness is expressed as the percent of valid usable data obtained compared to the amount that was expected. Comparability expresses the degree of confidence with which one data set can be compared to another. Sensitivity is defined by the laboratory detection limits and are generally expressed in terms of method detection limits (MDLs) or reporting limits (RLs).

Precision and Accuracy: The measurement quality objectives (MQOs) for precision and accuracy for the analyses of the specific COCs are summarized in Table 2.

Representativeness: Sample locations will be selected using professional judgment and knowledge of site history. Sample locations will adequately represent site conditions in the area being investigated.

Completeness: Data collection may be inhibited by geologic conditions and/or underground utilities. The project goal is to obtain at least 90% of the soil samples outlined in this SAP.

Comparability: The laboratory that will be used for analytical testing of soil samples collected during this investigation (Alpha) is certified by the State of Nevada for standard analyses under the Clean Water Act and the Safe Drinking Water Act as described in Appendix A of the NBP QA Program Plan (NDEP, 2007). Relevant SOPs from Alpha for the analyses to be conducted during this investigation are provided in Appendix B.

Sensitivity: The laboratory reporting limit for each analyte is summarized in Table 3. The reporting limits are well below the action levels and provisional action levels and are adequate for this investigation

3.4 Data Review and Validation

Data verification is the process of evaluating the completeness, correctness, conformance, and compliance of a specific data set against the method, procedural, or contractual requirements. Data verification evaluates whether sampling protocols, SOPs, and analytical methods were followed during data generation. Verification also involves examining the data for errors or omissions. Field and laboratory staff will verify that the work is producing appropriate outputs.

Data validation is a systematic process for reviewing a body of data against a pre-established set of acceptance criteria defined in this plan. Data validation is an analyte- and sample-specific process that extends the evaluation of data beyond data verification and is performed to determine the analytical quality of a specific data set. Validation involves a detailed examination of the data package to determine whether MQOs for precision, accuracy, and sensitivity have been met. For this environmental assessment, the intent of the data review and validation process is to verify that the specified levels of precision, accuracy, reproducibility, completeness, comparability, and analytical sensitivity of the final results are achieved, with respect to the project MQOs, and that the data fulfill project DQOs.

MGA's QA officer will supervise or perform data quality assessment tasks. MGA will consistently evaluate and document measurement data to monitor consistency with MQOs, to quantitatively assess data quality, and to identify potential limitations to data use. MGA will review field and analytical laboratory data generated for this project, including the following:

- Chain of custody documentation;
- Laboratory batch QC frequency; and,
- Results of batch and field QC analyses;

Laboratory Data: The laboratory will generate and review all laboratory data. Each data point will be assessed as non-qualified or qualified based upon the acceptance criteria. Data may be qualified as "estimated" (J-qualified); these data are used as is. Some data may be qualified as "rejected" (R-qualified) if critical QC parameters are not met; these data are unusable for any purpose. Sample re-analysis, for data not meeting MQOs, will be considered as a possible corrective action. Third-party data validation will not be performed.

3.5 Data Management

Sampling will be conducted in accordance with MGA's standard operating procedures (SOPs). A unique identification number will be assigned to each sample. The number will be an alphanumeric sequence that serves as an acronym to identify the sample. The following format will be used for the sample designation:

3.5.1 Soil Samples

Soil samples collected for this project will be identified based on the following unique identification system:

Sample ID: LVBRN017-SS-1-5.0

LVBRN017 - MGA Project Number

SS-1 – Soil Sample Number (i.e., #1)

5.0 – Depth of soil sample (i.e., 5.0 feet below ground surface)

3.5.2 Groundwater Samples

Groundwater samples collected for this project will be identified based on the following unique identification system:

Sample ID: LVBRN017-GW-1

LVBRN017 - MGA Project Number

GW-1 – GW Sample from Boring Number (i.e., #1)

Field logs shall be maintained throughout the project. The following information shall be included on the field logs: description of activities conducted, dates and times, field observations, deviations from sampling program, names of on-site personnel, sampling locations.

Samples shall be preserved or cooled as required for each laboratory analysis. Samples shall be delivered or shipped to the laboratory under chain-of-custody protocol.

3.6 Assessment Oversight

Prior to commencing with field activities, the SAP will be reviewed by the Project Team. The MGA QA Officer will oversee QC of all field activities. If modifications to the proposed sampling program are required due to field conditions, the Project Manager shall be notified for direction. Any modifications to the sampling plan will be documented in the field logs and in the project report as "deviations from the sampling plan."

4. SAMPLING RATIONALE

Borings to groundwater are proposed to be advanced at locations within the subject property based on a ground penetration radar study that will look for orphaned tanks. Other borings will be advanced at locations most likely to be impacted by off-site sources. Groundwater samples will be collected from each boring once groundwater is encountered. In addition, soil samples will be collected from various depths within each boring based on photo ionization detector readings made during boring advancement.

An adequate number of samples will be collected to initially assess site conditions. Professional judgment shall be used to select sampling locations that are likely to provide data to address project DQOs (Table 1). Decision statements formulated in the project DQOs are largely concerned with delineating the extent and magnitude of contamination. It is estimated that a maximum of five groundwater samples and ten soil samples will be collected for this assessment.

4.1 Soil Sampling

Once groundwater boring locations are chosen, a direct push drill rig will be utilized to advance each boring to groundwater. Continuous soil samples will be collected from each boring and select depths from each boring will be chosen for sample analysis. Selection of depth will be based on PID screening performed during boring advancement. MGAs SOP for PID soil screening can be found in Appendix B. A minimum of two soil samples will be collected from each boring.

4.2 Groundwater Sampling

A direct push drill rig will be utilized to advance each boring until groundwater is encountered. At this time, a point-in-time groundwater "grab sampler" will be used to collect samples to define groundwater conditions during the single sampling event. One groundwater sample will be collected from each boring.

4.3 Sediment Sampling

Sampling of sediments is not included in the scope of this investigation.

4.4 Biological Sampling

Biological sampling is not included in the scope of this investigation.

5. REQUEST FOR ANALYSIS

Laboratory analyses for each collected sample are discussed in Section 5.1 below.

5.1 Analyses Narrative

5.1.1 Soil Samples

It is anticipated that three to five borings will be advanced on the subject property. A minimum of two soil samples will be collected from each boring for analytical testing. The soil samples will be collected as described in Section 4.1 and analyzed for the following:

- Semi-volatile organic compounds (SVOCs): EPA Method 8270C;
- Volatile organic compounds (VOCs): EPA Method 8260B; and
- Total petroleum hydrocarbons (TPH) full range: EPA Method 8015.

5.2 Analytical Laboratory

All analytical testing shall be conducted by Alpha. Analytical testing and sample handling shall be conducted in accordance with Alpha's SOPs (Appendices A and B).

6. FIELD METHODS AND PROCEDURES

6.1 Field Equipment

6.1.1 List of Equipment Needed

- Field logbook and field data sheets;
- Personal protective equipment (Level D);
- Tape measure;
- Camera;
- Direct push drilling rig;
- 30-gallon drums;
- PID instrument;
- 4-oz glass sample containers;
- Volatile organic analysis (VOA) vials;
- 2-Liter glass amber jars;
- Point-in-time groundwater "grab samplers"
- Cooler and ice;
- Sample labels;
- Pick axe;
- Shovel;
- Stainless steel bowls and scoops; and
- Decontamination supplies;

6.1.2 Calibration of Field Equipment

All field equipment will be calibrated according to the manufacturer's guidelines and specifications.

6.2 Field Screening

Field screening will not be utilized in this investigation.

6.3 Soil Sampling

6.3.1 Surface Samples

Surface samples are not anticipated to be collected for this project. However, if surface soil sampling is performed, sample collection will be conducted in accordance with MGA's SOP as presented in Appendix C.

6.3.2 Sub-surface Samples

Sub-surface soil samples will be collected in accordance with MGA's SOP as presented in Appendix C.

6.4 Sediment Sampling

Not applicable as sediment sampling is not included in the scope of this investigation.

6.5 Groundwater Sampling

Groundwater samples will be collected in accordance with MGA's SOP as presented in Appendix C.

6.6 Decontamination Procedures

All field equipment which comes in contact with potentially contaminated soil and/or groundwater will be decontaminated in accordance with MGA's SOP as presented in Appendix C. Decontamination will occur prior to and after each use of a piece of equipment.

7. SAMPLE CONTAINERS, PRESERVATION AND STORAGE

7.1 Soil Sampling

7.1.1 Soil Sample Containers

Soil samples will be collected in dedicated sample containers provided by the analytical laboratory. The soil samples will be delivered to the laboratory within an acceptable period of time. Appendix C provides MGA's SOPs for sampling.

7.1.2 Soil Sample Preservation and Storage

Collected soil samples will be chilled to 4°C within a laboratory supplied cooler upon collection and during transport to the laboratory.

7.2 Groundwater Sampling

7.2.1 Groundwater Sample Containers

Groundwater samples will be collected in dedicated sample containers provided by the analytical laboratory. The groundwater samples will be delivered to the laboratory within an acceptable period of time. Appendix C provides MGA's SOPs for sampling.

7.2.2 Groundwater Sample Preservation and Storage

Collected groundwater samples will be preserved per the requirements of each specific analysis. In addition, groundwater samples will be chilled to 4°C within a laboratory supplied cooler upon collection and during transport to the laboratory.

8. DISPOSAL OF RESIDUAL MATERIALS

Investigation-derived waste generated during this investigation will be collected in 30-gallon waste drums and disposed per federal, state, and local regulations.

9. SAMPLE DOCUMENTATION AND SHIPMENT

9.1 Field Notes

9.1.1 Field Logbooks

Field logs will be completed describing all field activities. The following information will be included in the field logs:

- Project name and location;
- Sampling location and description utilizing a survey- or mapping-grade GPS unit;
- Site plan showing sample locations;
- Sampler's name (s);
- Date and time of sample collection;
- Type of sample (e.g., soil or groundwater);
- Type of sampling equipment used;
- Field instrument readings and calibration;
- Field observations and details related to analysis or integrity of samples (e.g., noticeable odors, colors, etc.);
- Sample preservation;
- Lot number of the sample containers, sample identification numbers and explanatory codes, and chain-of-custody form numbers; and
- Name of recipient laboratory.

9.1.2 Photographs

Photographs will be taken at select sampling locations. They will serve to verify information entered in the field logbook. For each photograph taken, the following information, at a minimum, will be written in the logbook:

- Time, date, location, and weather conditions;
- Description of the subject photographed; and
- Name of person taking the photograph.

9.2 Labeling

All samples collected will be labeled in a clear and precise manner for proper identification in the field and for tracking in the laboratory. The samples will have pre-assigned, identifiable, and unique numbers. At a minimum, the sample labels will contain the following information:

- Sample location;
- Date and time of collection;
- Analytical parameter(s) requested; and
- Method of preservation.

9.3 Sample Chain-of-Custody Forms and Custody Seals

All samples shall be delivered to the laboratory under chain-of-custody protocol. A copy of Alpha's chain-of-custody form is provided in Appendix D. Laboratory supplied custody seals

shall be used to seal the screw lid of each sample container.

9.4 Packaging and Shipment

Samples shall be placed in a sturdy cooler. Bubble wrap shall be placed in the bottom of the cooler and sample containers shall be placed in containers provided by the laboratory. Ice shall be packed in zipper locked, double plastic bags. Empty space in the cooler shall be filled with bubble wrap. Appendix C provides MGA's SOP for sample packaging and shipping.

10. QUALITY CONTROL

10.1 Field Quality Control Samples

Samples will be collected in accordance with industry standard procedures. No equipment blanks will be collected during this investigation.

10.2 Background Samples

No background samples are anticipated to be collected during this investigation.

10.3 Field Screening and Confirmation Samples

No confirmation samples will be collected during this investigation.

10.4 Assessment of Field Variability (Field Duplicates or Co-located Samples)

One duplicate soil sample per 10 targeted samples will be collected for laboratory quality control purposes. When collecting a duplicate soil sample, the sample containers with the two different sample identification numbers will alternate in the filling sequence. The duplicate samples will be preserved, packaged, and sealed in the same manner as the other samples of the same matrix. A separate sample number and station number will be assigned to the duplicate sample such that it is blind to the laboratory.

10.5 Laboratory Quality Control Samples

Laboratory QC (e.g., matrix spike/matrix spike duplicate samples) samples will be analyzed to monitor the precision and accuracy of its analytical procedures.

11. FIELD VARIANCES

As conditions in the field may vary, it may become necessary to implement minor modifications to sampling as presented in this SAP. Modifications to the approved SAP will be documented in the sampling project report.

12. FIELD HEALTH AND SAFETY PROCEDURES

A site-specific Health and Safety Plan is provided in Appendix E. The HASP shall be reviewed by all on-site personnel prior to commencing with field activities.

13. SCHEDULE FOR SAMPLING ACTIVITIES

MGA will commence with the activities proposed herein upon receiving NDEP approval of this SAP. It is anticipated that field activities will be completed within three weeks of receiving SAP

approval. However, the field activities will be reliant upon amenable weather conditions. It is anticipated that a draft report of findings will be submitted prior to April 30, 2013.

14. REFERENCES

Coats, R.R., 1987. *Geology of Elko County, Nevada.* Bulletin 101. Nevada Bureau of Mines and Geology, Mackay School of Mines, University of Nevada, Reno.

Natural Resources Conservation Service. *Soil Survey Area: Elko County, Nevada, Southeast Part. Survey Area Data: Version 4, May 1, 2007.* United States Department of Agriculture.

Nevada Division of Environmental Protection, 2007. *Final Nevada Brownfields Program Quality Assurance Program Plan.*

US EPA. 2006. *Guidance on Systematic Planning using the Data Quality Objectives Process.* February. EPA QA/G-4, EPA/240/B-06/001

Table 1. DQO Summary Table for Environmental Sampling, APN: 002-770-005, Wells, Nevada
STEP 1
State the Problem
Possible historic use of the site as a gas station and evidence of a former fueling island may indicate that orphaned USTs still exist on the subject property. In addition, LUSTs on other properties located adjacent or in the immediate vicinity may have impacted the groundwater beneath the subject property. However, the nature and extent of contamination in the soils and groundwater, if any, is not known. Additional data are needed to define the nature and extent, if any, of contamination within the soil and groundwater.
STEP 2
Identify the Decisions
1) Do contaminants of concern (COC) concentrations in the soil or groundwater exceed Nevada reportable concentrations (RCs)? 2) Does the extent of the concentration of the COCs appear to be greater than three cubic yards? 3) Is further assessment required to determine the nature and extent of contamination within the study area? 4) Is regulatory notification required?
STEP 3
Identify the Inputs to the Decisions
• Analytical data for collected samples (quantitative data) • Soil RCs as published in the State of Nevada Division of Environmental Protection (NDEP) Draft Guidelines for Discover Events (Soil RCs) document (NAC 445A.345 to 445A.348 as modified by adopted regulation R-189-08)
STEP 4
Define Study Boundaries
The proposed investigation shall extend from the surface to groundwater. The duration of the assessment activities described in this SAP is approximately one week.
STEP 5
Develop Decision Rules
1) If specific COCs are reported by the analytical laboratory to be greater than reportable detection limits (RDLs) for that analyte, the corresponding RC will be used to screen the data; 2) If the concentration exceeds the RC, then a calculation to determine an approximate volume of contaminated soil will be performed. 3) If data received from the analytical laboratory suggests that the extent of contamination within the study area is still not determined, another round of soil sampling shall be proposed. 4) If contaminated soil quantities exceed 3 cubic yards, the discovery will be reported to the NDEP. 5) If groundwater concentrations exceed the State of Nevada RC, the discovery will be reported to the NDEP.
STEP 6
Specify Tolerable Limits on Errors
The number of samples to be collected is not statistically based and will be determined in the field based using professional judgment. MQOs and DQIs established for analytical data are described in the NBP QA Program Plan.
STEP 7
Optimize Sampling Design
The quantity of samples is believed to be adequate to complete an initial assessment of site conditions.

Table 2: Method Precision and Accuracy Goals for Select COCs				
Matrix Spike Compound	Soil		Groundwater	
	Recovery (%)	RPD (%)	Recovery (%)	RPD (%)
TPH Gas Range (GRO)	67-135	20	70-124	20
TPH Diesel Range (DRO)	50-150	<20	50-150	<20
Benzene	65-128	20	79-131	20
Toluene	70-120	20	68-114	20
Ethylbenzene	74-128	20	68-125	20
Total Xylenes	74-127	20	67-113	20
Acenaphthene	22-139	36	32-120	29
Acenaphthylene	33-118	35	41-112	30
Anthracene	65-119	20	48-122	26
Benzo(a)anthracene	77-123	20	52-122	22
Benzo(a)pyrene	68-118	20	45-120	24
Benzo(b)fluoranthene	68-110	20	46-118	24
Benzo(g,h,i)perylene	57-118	28	31-110	31
Benzo(k)fluoranthene	70-124	20	45-112	24
Chrysene	79-125	20	53-126	23
Dibenzo(a,h)anthracene	64-121	25	26-113	35
Fluoranthene	76-121	20	52-125	23
Fluorene	47-126	28	45-117	27
Indeno(1,2,3-c,d)pyrene	62-121	26	40-113	29
Naphthalene	11-104	49	22-105	37
Phenanthrene	63-118	20	48-122	26
Pyrene	77-125	20	53-128	24

RPD: Relative Percent Difference

Table 3. Reporting Limits and Nevada RCs for Contaminants of Concern				
Contaminant of Concern	**Laboratory Reporting Limit (RDL)**		**Nevada RCs**	
	Soil (mg/Kg)	**Groundwater (µg/L)**	**Soil[1] (mg/Kg)**	**Groundwater[1] (µg/L)**
TPH Gas Range (GRO)	1	100	100	NA
TPH Diesel Range (DRO)	40	100	100	NA
Benzene	0.001	1	0.03	5
Toluene	0.005	5	12	1000
Ethylbenzene	0.001	1	5.7	700
Total Xylenes	0.003	3	210	10,000
Acenaphthene	0.006	0.05	570	NA
Acenaphthylene	0.006	0.05	NA	NA
Anthracene	0.006	0.05	1,200,000	NA
Benzo(a)anthracene	0.006	0.05	0.15	NA
Benzo(a)pyrene	0.006	0.05	0.015	0.2
Benzo(b)fluoranthene	0.006	0.05	0.15	NA
Benzo(g,h,i)perylene	0.006	0.05	NA	NA
Benzo(k)fluoranthene	0.006	0.05	1.5	NA
Chrysene	0.006	0.05	15	NA
Dibenzo(a,h)anthracene	0.006	0.05	0.015	NA
Fluoranthene	0.006	0.05	2,300	NA
Fluorene	0.006	0.05	560	NA
Indeno(1,2,3-c,d)pyrene	0.006	0.05	0.15	NA
Naphthalene	0.006	0.25	3.9	NA
Phenanthrene	0.006	0.05	NA	NA
Pyrene	0.006	0.05	1,700	NA

[1] NDEP, 2010

NA: Not Applicable (no RC provided)

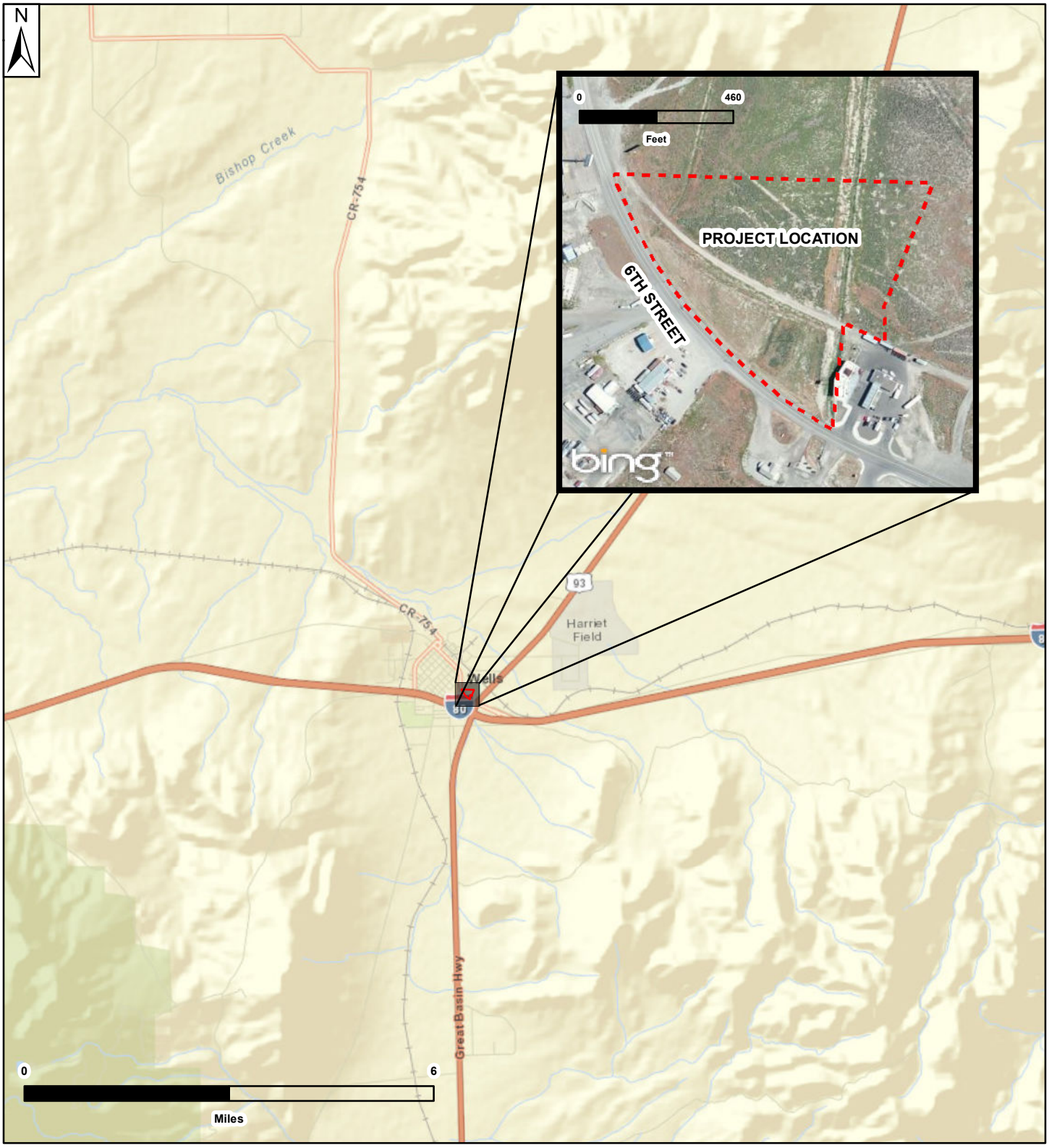

FIGURE 1

TITLE:

PROJECT LOCATION MAP

-SHOWING-

APN: 002-770-005

WELLS, NEVADA

JOB NO.: **LVBRN017**

DATE: **1/24/2013**

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Environmental Engineering and Science
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FILE: **130124_LVBRN017_figure1_msp_85x11_portrait**

COORDINATE SYSTEM: **NAD 1983 UTM Zone 11N US Feet**

REF.					REVISION:
	DESIGNED	MSP	CHECKED	MSP	-
	DRAWN	MSP	APPROVED	BB	

FIGURE 2

TITLE:

SITE MAP

-SHOWING-

APN: 002-770-005

WELLS, NEVADA

JOB NO.: **LVBRN017**

DATE: **1/24/2013**

McGinley & Associates
Environmental Engineering and Science
RENO | LAS VEGAS | www.mcgin.com

FILE: **130124_LVBRN017_figure2_msp_85x11_portrait**

COORDINATE SYSTEM: **NAD 1983 UTM Zone 11N US Feet**

REF.					REVISION:
	DESIGNED	MSP	CHECKED	MSP	-
	DRAWN	MSP	APPROVED	BB	

APPENDIX A

Laboratory Data Quality Objectives and Sample Handling Procedures

Quality Assurance Manual

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Version 9.0 4/15/11

Disclaimer

The ESC Lab Sciences Quality Assurance Manual is a living document. It is reviewed at least annually and revised when needed. The information stated herein is subject to change at any time due to updates to QC Limits, methods, operations, equipment, staff, etc. At the time of distribution the requestor will receive the most recent version of the manual and will be assigned a control number. The control number will help ESC to track what version is sent. The revision number is stated on the cover page of the manual.

Expiration

This manual expires 1 year from the date listed at the front of the manual on the “Approvals” page. If you have a copy that is not dated within this time period, please contact the laboratory and obtain the most recent version.

COMPREHENSIVE QUALITY ASSURANCE MANUAL

for

ESC LAB SCIENCES
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Prepared by

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The ESC QAM has been prepared in accordance with the following standards: AIHA (LQAP), A2LA (Env. Prog. Req.), ANSI/ISO 17025-2005, NELAC, DOD QSM.

Quality Manual: Table of Contents

Tables and Figures

1.0 GENERAL

1.1 INDEX AND REVISION STATUS

The numbering of this quality manual corresponds directly to the numbering of ISO 17025:2005 with cross-references to the 2003 NELAC Standard and the 2009 standard of The NELAC Institute (TNI).

This quality manual is only valid if all pages are at the same issue level as shown in the index of the quality manual.

Updates to this manual are made by re-issuing the relevant section of this manual and adapting the issue level in the index. New version numbers are assigned upon revision.

NOTE: This manual expires 1 year from the date listed at the beginning of the manual on the "Approvals" page.

1.2 PURPOSE

This quality manual documents the laboratory's management system and demonstrates the ability to execute the indicated tests and/or procedures and to meet regulatory requirements.

This manual establishes compliance with ISO 17025, NELAC, DOD QSM, and AIHA.

2.0 LABORATORY BACKGROUND

2.1 ACTIVITIES

2.1.1 Analytical Support and Service Areas

ESC Lab Sciences is an environmental analytical firm providing technical and support services to clients nationwide. Specific service areas include the following:

- drinking water analysis
- industrial wastewater analysis
- hazardous waste characterization and identification
- groundwater analysis
- air analysis
- regulatory document guidance
- biological assessments
- mold identification
- solid/soil analysis and characterization
- industrial hygiene/environmental lead

2.1.2 Regulatory Compliance and Quality Standards

ESC is devoted to providing reliable and accurate data recognizing the necessity to establish sound, objective, and legally defensible positions or opinions for clients regarding compliance with governing regulations. ESC maintains quality systems that are compliant with the following Quality Standards: AIHA LQAP, A2LA, ANSI/ISO 17025, NELAC, DOD QSM. The effectiveness of the quality system is measured by internal and external audits, management review meetings, internal error logs and an active preventive and corrective action system.

2.1.3 Analytical Capabilities:

Where mandated, only approved EPA procedures are used for environmental analyses. ESC utilizes a number of method sources to accomplish project requirements. For NPDES and SDWA, methodologies are taken directly from 40 CFR parts 136 and 141.

For industrial hygiene analytical procedures, ESC utilizes guidance from NIOSH and OSHA published methods.

The following list is an example of the methodology ESC routinely performs:

Routine Methodology and Programs	
PROGRAM	***METHOD SOURCE***
NPDES	***EPA 821/R-93-010-A*** *Methods for the Determination of Nonconventional Pesticides in Municipal and Industrial Wastewater, Volume I. Revision 1, August 1993.*
	EPA 821/R-02-019 *Method 1631, Revision E: Mercury in Water by Oxidation, Purge and Trap, and Cold Vapor Atomic Fluorescence Spectrometry. August 2002.*
	40 CFR part 136
	Methods for Chemical Analysis of Water and Wastes (March 1983)
	Standard Methods for the Examination of Water and Wastewater (18th, 19th, 20th editions)
AQUATIC TOXICITY	*7-Day Fathead Minnow (Pimephales promelas) Larval Survival and Growth Test; Test Method 1000.0 from "Short Term Methods for Estimating the Chronic Toxicity of Effluents and Receiving Waters to Freshwater Organisms" (EPA 821-R-02-013).*
	3-Brood Ceriodaphnia dubia Survival and Reproduction Test; Test Method 1002.0 from "Short Term Methods for Estimating the Chronic Toxicity of Effluents and Receiving Waters to Freshwater Organisms" (EPA 821-R-02-013).
	Fathead Minnow (Pimephales promelas) Acute Toxicity Test (24, 48 or 96 hour duration); referenced in "Methods for Measuring the Acute Toxicity of Effluents and Receiving Waters to Freshwater and Marine Organisms" (EPA 821-R-02-012, 10-02).
	Ceriodaphnia dubia Acute Toxicity Test (24, 48 or 96 hour duration); referenced in "Methods for Measuring the Acute Toxicity of Effluents and Receiving Waters to Freshwater and Marine Organisms" (EPA 821-R-02-012, 10-02).
SDWA	40 CFR parts 141
	Methods for Chemical Analysis of Water and Wastes (March 1983)
	Standard Methods for the Examination of Water and Wastewater (18th, 19th, 20th editions)
	Methods for the Determination of Organic Compounds in Drinking Water -EPA/600/4-88/039 - December 1988 (Revised July 1991)
	Methods for the Determination of Organic Compounds in Drinking Water Supplement I, EPA/600/4-90/020 - July 1990
	Methods for the Determination of Organic Compounds in Drinking Water Supplement II, EPA/600/R-92/129 - August 1992
	EPA. Method 1623: *Cryptosporidium and Giardia in Water by Filtration/IMS/FA, December 2005.*
RCRA	*SW-846, Test Methods for Evaluating Solid Wastes (3rd, 4th and online editions)*
IH	*NIOSH Manual of Analytical Methods (4th edition) & OSHA Sampling and Analytical Methods (online)*
AIR	*Compendium of Methods for the Determination of Toxic Organic Compounds in Ambient Air*
	Emission Measurement Center (Air Emissions Methods)
	NIOSH Manual of Analytical Methods (4th edition)
	Journal of Chromatographic Science, Vol. 36, May 1998.
CLP	*USEPA CONTRACT LABORATORY PROGRAM - STATEMENT OF WORK* FOR ORGANICS ANALYSIS Multi-Media, Multi-Concentration OLM04.3
	USEPA CONTRACT LABORATORY PROGRAM - STATEMENT OF WORK FOR INORGANIC ANALYSIS Multi-Media, Multi-Concentration ILM05.3

Routine Methodology and Programs	
PROGRAM	***METHOD SOURCE***
MOLD	*American Industrial Hygiene Association*
	NIOSH Manual of Analytical Methods (4th edition)
Miscellaneous	*American Society for Testing and Materials (ASTM)*
	State Specific Methodologies from the following: *Florida, Oregon, Iowa, Washington, Texas, Arizona, Massachusetts, North Carolina, Louisiana, Missouri, Kansas, Wisconsin, Ohio*
Miscellaneous	*Analytical Methods for the Determination of Pollutants in Pharmaceutical Manufacturing Industry Wastewater, Revision A EPA-821-B-98-016 - July 1998 (Approved at 40 CFR Part 136, Not Approved at Part 141)*

2.2 HISTORY

ESC Lab Sciences was founded in 1970 by Dr. Arthur Schulert, a professor of Biochemistry at Vanderbilt University Medical School. The laboratory's first location was a 2,000 square foot building located in Mt. Juliet, TN.

ESC initially conducted several research contracts for the National Science Foundation. EPA Clean Water and Safe Drinking Water legislation of the early 1970s provided an additional market of Tennessee utilities and industries. ESC grew slowly for several years by increasing the share of the drinking and wastewater markets in Tennessee. In the late 1980s, ESC expanded its capabilities to include Underground Storage Tank testing and Biomonitoring/Toxicity testing.

Strategic expansion of the laboratory allowed ESC to provide support to large RCRA sites and add capabilities to offer analytical support for air and mold analyses. ESC is currently the nation's largest, single-location environmental laboratory and is the only laboratory facility certified/approved to operate in all US states. Our staff of over 250 employees works out of our 87,000 square feet, nine-building facility approximately 20 minutes east of Nashville International Airport.

3.0 INTRODUCTION, SCOPE, AND DEFINITIONS

3.1 SCOPE OF CAPABILITIES

A list of approved and certified analytical capabilities can be found at the end of this section in Table 3.3b.

3.2 TABLE OF CONTENTS, REFERENCES AND APPENDICES

The table of contents is found at the beginning of this Manual. This *Quality Manual* uses the references from the 2003 NELAC Standard, Chapter 5, Appendix A.

3.3 DEFINITIONS AND TERMINOLOGY

The quality department is responsible for establishing and maintaining a list of definitions and conventions.

Table 3.3a Definitions

TERM	***DEFINITION***
Acceptance Criteria (Analytical QC Limits)	Specified limits placed on characteristics of an analytical process as defined in analytical methodology or guidance.
Accuracy	The amount of agreement between an observed value and an accepted reference value. Accuracy is represented as percent recovery.
Analytical Reagent Grade	Designation for the high purity of certain chemical reagents and solvents assigned by the American Chemical Society.
Analytical Sensitivity	The lowest concentration that can be detected by the method. (e.g., for methods involving a count = 1 raw count calculated to the reporting units). Analytical sensitivity is commonly used in Mold analysis.
Batch Analysis	Analysis of 1 – 10 or 20 samples, depending on the published method requirements, including all required QC. When there are 21 or more samples to be analyzed, the QC criteria for the next 20 samples is the same as it is with a single batch.
Batch	1 – 10 or 20 samples, depending on the published method requirements. A group of samples that behave similarly and are analyzed as a unit.
Blank	See FIELD, TRIP, METHOD, EQUIPMENT
Blind Sample	A sample submitted for analysis with a composition known only to the individual requesting the analysis. The analyst/laboratory may know the identity of the sample, but not its composition. It is used to verify the analyst or laboratory's proficiency in the execution of the analytical measurement process.

Table 3.3a Definitions	
Calibration	To determine, by measurement or comparison with a known standard, the correct value of each scale reading on a meter or other device, or the correct value for each setting of an instrument control. The levels of the applied calibration standard should bracket the range of planned or expected sample measurements.
Calibration Curve	The graphic representation of the relationship between the known values, such as concentrations of a series of calibration standards and instrument responses.
Calibration Factor	The ratio of the detector response (peak areas or peak heights) to the amount (mass) of analyte in the calibration standard. $$CF = \frac{A_s}{C_s}$$ where: A_s - Average Peak Area over the number of peaks used for quantitation C_s – Concentration of the analyte in the standard.
Continuing Calibration Blank (CCB)	The CCB is used to confirm the absence of contaminants within the analytical system prior to and during the analysis of field samples. The CCB must be <½ RL, concentrations of common laboratory contaminants cannot exceed the reporting limit. The CCB is analyzed on at regular intervals within a batch and is typically utilized in Metals and Wet Chemistry analyses.
Continuing Calibration Verification (CCV)	A standard, usually near the mid-point of the calibration curve, made from the primary stock used for the calibration curve. The CCV is used to verify the calibration stability of the instrument and must perform within method stated criteria, which is usually ±10 to 15%. The CCV must be analyzed at regular intervals within a batch.
Continuing Demonstration of Capability (CDOC)	Continuing Demonstration of Capability – Annual* verification of analyst skill. *unless required more frequently by program or method
Chain of Custody	A record that documents the possession of the samples from the time of collection to receipt by the laboratory. This record generally includes: the number and types of containers, the mode of collection; collector ID; time of collection; preservation; and requested analysis.
Corrective Action	An action taken to eliminate the causes of an existing nonconformity, defect or other undesirable situation in order to prevent recurrence.
Data Quality Objective (DQO)	A statement of the overall level of uncertainty that a data user is willing to accept in results derived from analytical data.
Duplicate	Second aliquots of field samples carried through the entire preparation and analytical process that are used as an indication of sample precision or consistency in the field sample matrix.
Equipment Blank	A sample of analyte free water (usually laboratory DI) which has been used to rinse the sampling equipment. It is collected after decontamination procedures but prior to sampling. The purpose is to demonstrate complete decontamination of the equipment.

Table 3.3a Definitions	
External Calibration Model	Comparison of instrument responses from the sample to the responses from the target compounds in the calibration standards. Sample peak areas (or peak heights) are compared to peak areas (or heights) of the corresponding analytes in calibration standards.
Field Blank	A sample of analyte free water (usually laboratory DI) is poured into the appropriate collection vessel and preserved according to method guidelines. The purpose of the field blank is to serve as a check on reagent and environmental contamination.
Initial Calibration Verification (ICV) See also SSCV	An independently prepared standard used to verify the accuracy of the initial calibration (for ongoing calibration). The ICV is used to represent the calibration efficiency of the instrument and must perform within method stated criteria, which is usually ±10 to 15%. For metals analysis, the ICV is a secondary source.
Initial Demonstration of Capability (IDOC) See also CDOC	A demonstration of capability (DOC) must be made prior to using any analytical method and any time there is a change in instrument type, personnel or testing method. Such performance must be documented and the four preparation batches following the change in personnel must not result in the failure of any batch acceptance criteria, e.g., method blank and laboratory control sample, or the demonstration of capability must be repeated.
Instrument Detection Limit (IDL)	IDL is the smallest signal above background noise that an instrument can reliably detect.
Interference Check Sample (ICS)	A series of two solutions, used in ICP and ICPMS analysis, to verify that inter-element interferences are compensated for correctly. This standard is referred to as the Spectra Interference Check (SIC) in EPA Method 200.7 ICSA – A solution containing only the interfering analytes at high concentrations. ICSAB – A solution containing interferents plus other method analytes at the level of concern, which corresponds to the project specific action limits. ICSA and ICSAB provide and adequate test of inter-element correction (IEC) factors.
Internal Calibration Model	Internal standard calibration involves the comparison of instrument responses from the target compounds in the sample to the responses of specific internal standard analytes added to the sample or sample extract prior to injection.
Internal Standards	Analytes not expected to occur naturally in field samples that are spiked to provide a consistent basis for comparison with target analytes. ISTDs are used in internal calibration models.

Table 3.3a Definitions

Laboratory Control Sample (LCS) - 2ND Source	A known matrix is spiked with known amounts of the analyte(s) of interest used to verify the efficiency of the analytical system without interference from the sample matrix. The LCS provides the best estimate of analytical system accuracy and may also be used to verify the validity of the on-going calibration. The source is usually a secondary source. The LCS matrix must closely represent the matrix of the sample batch and undergo all preparations required by the method prior to analysis. The following list are acceptable matrices for the LCS: ***Batch Matrix*** — ***LCS Matrix*** Water — Laboratory DI water Soil — Spiked Ottawa sand or Glass beads or commercially prepared LCS in a soil matrix Paint Chips — Laboratory prepared paint chip/lead mixture Commercially prepared & certified paint chip LCS Filters/Sorbent Media/Dust Wipes — Unused Industrial Hygiene sampling media that represents the substrate submitted by the client. Where possible, the media should be the same lot as that of the field samples.
Limit Of Detection (LOD)	The lowest concentration that can be determined by a single analysis to be statistically different from a blank, within a defined level of confidence. This concentration is recommended to be three standard deviations above the measured average difference between the sample and blank signals, which corresponds to the 99% confidence level. In practice, detection of an analyte by an instrument is often based on the extent to which the analyte signal exceeds peak-to-peak noise (Keith et al. 1983). Samples that do not bear residues at or above the LOD are referred to as non-detects (ND).
Limit of Quantitation (LOQ)	The minimum levels, concentrations, or quantities of a target variable (e.g., target analyte) that can be reported with a specified degree of confidence. The LOQ may be equal to the RL, MRL, or PQL. Routinely the PQL/LOQ is at least 3-5 times the statistically derived MDL/LOD.
Linear Dynamic Range (LDR)	In Inorganic analyses, the LDR is defined as the concentration range where absorbance and concentration remain directly proportional to each other. A wide linear dynamic range permits the analysis of a wide range of sample concentrations (optical densities) and reduces sample preparation (dilution) requirements.

Table 3.3a Definitions	
Matrix	The component, or substrate, which contains the analyte of interest. For purposes of batch determination, the following matrix types are used: • *Aqueous:* Any aqueous sample excluded from the definition of a drinking water matrix or saline/estuarine source. Includes surface water, groundwater, and effluents. • *Drinking Water:* Any aqueous sample that has been designated as a potable or potentially potable water source. • *Saline/Estuarine:* Any aqueous sample from an ocean or estuary, or other saltwater 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 are grouped according to origin. • *Solids:* Includes soils, sediments, sludge 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 Samples:* Media used to retain the analyte of interest from an air sample such as sorbent tubes or summa canisters. Each medium is considered as a distinct matrix. • *Solids (Other than defined above):* Includes filters, dust wipes, sorbent tubes, paint chips.
Matrix Spike (MS)	A separate aliquot of field sample spiked with a known amount of the target analyte. Accuracy is determined by comparing the recovery of the spike added to the known concentration in the sample divided by the expected analyte concentration. $$Percent Spike Recovery = \frac{O_i - O_s}{T_i} X\ 100$$ O_i = observed sample concentration with the spike added O_s = the observed value for the sample without the spike $$T_i = \frac{Spike\ Concentration\ \text{in}\ (mg/L)\ X\ Volume\ \text{of Spike in}\ (ml)}{Volume\ of\ Sample\ in\ (ml) + Volume\ of\ Spike\ in\ (ml)}$$ T_i = True value of the spike added
Matrix Spike Duplicate (MSD)	The second aliquot of the field sample spiked as the matrix spike and carried through all sample preparation/analytical steps. The MS/MSD pair are spiked with identical amounts of the target analyte and precision is calculated based on the results.
Method Detection Limit (MDL)	The minimum concentration of a substance that can be analyzed with 99% confidence that the analyte concentration is greater than zero. MDLs are performed in conjunction with 40CFR 136, Appendix B. The MDL is the absolute minimum level of reporting that is allowed. Values reported between the MDL and RL are flagged with a "J" qualifier.

Table 3.3a Definitions	
Method Blank	A laboratory produced blank is carried through each step of the analytical procedure for each batch of samples. Blanks are prepared for each preparation method and matrix (i.e., solids assay, dissolved metals, TCLP extraction, etc.). Blanks are used to confirm the absence of contaminants within the preparation and/or analytical system prior to and during the analysis of field samples.
Negative Control	Measures taken to ensure that an analytical process, its components, or the environment do not cause adverse effects or lead to incorrect quantitation.
Percent Recovery	A comparison between the observed value and the true value of a known spiked concentration, represented as a percentage. This evaluation applies to the calculation of ICV, CCV, LCS, MS/MSD, Surrogates, etc. and is calculated as follows: $$\%\ \text{Recovery} = \left[\frac{\text{Observed Value}}{\text{True Value}}\right] \text{X}\, 100$$
Positive Control	Measures taken to ensure that an analysis and/or its components are working properly and producing correct or expected results.
Post Digestion Spike	In metals analysis, a standard prepared from a previously analyzed spiked sample digestate that yielded reduced recovery for the target analyte due to a suspected matrix interferent.
Practical Detection Limit (PDL)	An in-house protocol that is used to determine a practical and real number for method detection. This is not a statistically derived number. It is a verified number that is validated using a 20% coefficient of variation.
Practical Quantitation Limit (PQL) ***See also Reporting Limit (RL)***	Generally, the lowest standard of the calibration curve. The PQL, or RL, is defined as the lowest level that can be reliably achieved within the established limits of precision and accuracy during routine laboratory operating conditions. The PQL is the default reporting limit (RL) when no other limits are required by the project. The PQL is usually a factor of 3-10 times greater than the determined MDL. The value of the PQL changes with subsequent sample dilutions and final volumes. The multiplier (dilution) of the sample is applied to the PQL for reporting. Values reported between the MDL and PQL are flagged with a "J" qualifier.
Precision	The agreement between 2 or more duplicate measurements. There is no assumption of the true value of the sample. Precision is expressed as RPD (Relative Percent Difference).
Proficiency Testing	The action of providing controlled and standardized environmental samples to a laboratory for analysis, reporting of results, statistical evaluation of the results in comparison to peer laboratories and the collective demographics and results summary of all participating laboratories.
Qualifier	A general explanation associated with deviations from established method criteria for a given analyte. The qualifiers are alpha-numeric designations that are related to specific comments. (i.e. J1 - "Surrogate recovery limits have been exceeded, values are outside of upper control limits.")
Quality Assurance	A plan for laboratory operation that specifies the measures used to produce data of known precision and bias.
Quality Control	A set of measures within a sample analysis methodology to assure that the process is operating from a controlled analytical system.

Table 3.3a Definitions	
Reference Material	A material or substance in which one or more properties are sufficiently well established to be used for the calibration of an apparatus, the assessment of a measurement method, or for assigning values to materials.
Reference Toxicant	The toxicant used in aquatic toxicity analyses to indicate the sensitivity of a test organism and to demonstrate the laboratory's ability to perform the procedure correctly and obtain consistent results.
Replicate Sample	The analytical measurement of a sample that has been split after it has been processed through the preparation stage. A replicate can also originate from a single sample that has been sub-sampled two or more times during the same analytical process time.
Reporting Limit (RL) See also PQL	The RL is equal to the PQL unless project specific limits are supplied/required by the client.
Relative Percent Difference (RPD)	$$RPD = \frac{\lvert Dup\ 1 - Dup\ 2 \rvert}{\left[\frac{(Dup\ 1 + Dup\ 2)}{2}\right]} X\ 100$$ The comparison of two values based on the mean of the two values. It is always reported as a positive number. The result is an assessment of precision. For sample duplication, the RPD is calculated using the actual analytical results of the field sample. LCS & MS calculations are also based on the actual sample result of spiked samples.
Response Factor (RF)	A measure of the relative response area of an analyte compared to its internal standard. The response factor is determined by the equation below, and if the calculated value meets the method guidelines it can be used to determine concentration for organic analyses. $$RF = \frac{(Conc._{IStd})(Area_{Analyte})}{(Conc._{analyte})(Area_{IStd})}$$ where: A_s = Response for analyte to be measured A_{is} = Response for the internal standard C_{is} = Conc. of the internal std.in ug/L C_s = Conc. of the analyte to be measured in ug/L.
Sample Blank	The purpose of a sample blank is to account for spectrophotometric interferences such as sample color, cloudiness, viscosity, etc. The sample blank must be analyzed at the same dilution as the sample. The sample blank is analyzed without any addition of reagents.
Selectivity	The capability of an analytical method or instrument to respond to a target substance or constituent in the presence of non-target substances.
Sensitivity	The capability of an analytical method or instrument to discriminate between measurement responses representing different levels (e.g., concentrations) of a property of interest.

Table 3.3a Definitions	
Secondary Source Calibration Verification (SSCV)	A mid-point or low standard made from the secondary source (lot or manufacturer) that is not used to construct the calibration curve. The SSCV is used to represent the calibration accuracy of the instrument and must perform within method stated guidelines. This sample is used to document calibration accuracy. The SSCV can be the same solution as the LCS, but is analyzed as an instrument standard, rather than a method prepared standard.
Serial Dilution	A subsequent dilution of a high concentration field sample that should agree within 10% of the original undiluted analysis. In metals analysis, a serial dilution is included in each preparation batch if target analyte concentration is at least fifty times the IDL. This is generally used as a test for matrix interferents or matrix effects.
Standard Operating Procedure (SOP)	A written document which details the method of an operation, analysis or action whose techniques and procedures are thoroughly prescribed and which is accepted as the method for performing certain routine or repetitive tasks.
Standard Reference Material	A certified reference material produced by the U.S. National Institute of Standards and Technology (NIST) and characterized for absolute content independent of analytical method.
Standards Addition	The process of spiking a known amount of analyte into an extract/digestate to observe the increase in concentration of the analyte in question. This process can be used to confirm analyte identification or suspected matrix interferences.
Surrogate	A compound that is similar to the target analytes in chemical composition and behavior and not expected to occur naturally in field samples. Surrogates are spiked by preparation/analytical personnel to assess sample preparation and analytical efficiency in each individual field sample.
Tentatively Identified Compound (TIC)	Compounds detected in samples that are not target compounds, internal standards, system monitoring compounds, or surrogates. TICs can be tentatively identified using mass spectrometers in spectral comparisons with NBS library searches. Quantitation of TICs provides a rough approximation of the concentration of these non-target analytes.
Trip Blank	A sample of analyte-free media (usually laboratory DI) that is taken from the laboratory to the sampling site and then returned unopened to the laboratory. The trip blank is used to ensure that cross contamination does not occur during shipment/storage and is used mainly for VOC analyses.

Table 3.3b
Analytical Capabilities
AE=Air Emissions, DW=Drinking Water, NPW=Non-potable Water, SCM=Solid Chemical Materials

The information listed is subject to change.
Always check with the laboratory for the most updated information.

Matrix	Approved Method	Parameter Description
AE	[EPA 3C]	Carbon Dioxide
AE	[EPA 3C]	Methane
AE	[EPA 3C]	Nitrogen
AE	[EPA 3C]	Oxygen
AE	[EPA 0040]	Hazardous organics
AE	[EPA TO-15]	Acetaldehyde
AE	[EPA TO-15]	Acetone
AE	[EPA TO-15]	Acetonitrile
AE	[EPA TO-15]	Allyl chloride
AE	[EPA TO-15]	Benzene
AE	[EPA TO-15]	Benzyl chloride
AE	[EPA TO-15]	Bromodichloromethane
AE	[EPA TO-15]	Bromoform
AE	[EPA TO-15]	Bromomethane
AE	[EPA TO-15]	Butadiene (1,3-)
AE	[EPA TO-15]	Carbon disulfide
AE	[EPA TO-15]	Carbon tetrachloride
AE	[EPA TO-15]	Chlorobenzene
AE	[EPA TO-15]	Chloroethane
AE	[EPA TO-15]	Chloroform
AE	[EPA TO-15]	Chloromethane
AE	[EPA TO-15]	Chlorotoluene (2-)
AE	[EPA TO-15]	Cyclohexane
AE	[EPA TO-15]	Dibromochloromethane
AE	[EPA TO-15]	Dibromoethane (1,2-) (EDB)
AE	[EPA TO-15]	Dichlorobenzene (1,2-)
AE	[EPA TO-15]	Dichlorobenzene (1,3-)
AE	[EPA TO-15]	Dichlorobenzene (1,4-)
AE	[EPA TO-15]	Dichlorodifluoromethane
AE	[EPA TO-15]	Dichloroethane (1,1-)
AE	[EPA TO-15]	Dichloroethane (1,2-)
AE	[EPA TO-15]	Dichloroethene (1,1-)
AE	[EPA TO-15]	Dichloroethene (cis-1,2-)
AE	[EPA TO-15]	Dichloroethene (trans-1,2-)
AE	[EPA TO-15]	Dichloropropane (1,2-)
AE	[EPA TO-15]	Dichloropropene (cis-1,3-)
AE	[EPA TO-15]	Dichloropropene (trans-1,3-)
AE	[EPA TO-15]	Dichlorotetrafluoroethane (1,2-)

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Matrix	Approved Method	Parameter Description
AE	[EPA TO-15]	Dioxane (1,4-)
AE	[EPA TO-15]	Ethylbenzene
AE	[EPA TO-15]	Ethyltoluene (4-)
AE	[EPA TO-15]	Gasoline range organic
AE	[EPA TO-15]	Hexachlorobutadiene (1,3-)
AE	[EPA TO-15]	Hexanone (2-)
AE	[EPA TO-15]	Heptane (n-)
AE	[EPA TO-15]	Hexane (n-)
AE	[EPA TO-15]	Isopropanol
AE	[EPA TO-15]	Isopropylbenzene
AE	[EPA TO-15]	Methyl alcohol (Methanol)
AE	[EPA TO-15]	Methyl ethyl ketone
AE	[EPA TO-15]	Methyl iodide
AE	[EPA TO-15]	Methyl isobutyl ketone
AE	[EPA TO-15]	Methyl methacrylate
AE	[EPA TO-15]	Methyl tert-butyl ether
AE	[EPA TO-15]	Methylene chloride (Dichloromethane)
AE	[EPA TO-15]	Naphthalene
AE	[EPA TO-15]	Propylene
AE	[EPA TO-15]	Styrene
AE	[EPA TO-15]	Trichlorobenzene (1,2,4-)
AE	[EPA TO-15]	Trimethylbenzene (1,3,5-)
AE	[EPA TO-15]	Trimethylbenzene (1,2,4-)
AE	[EPA TO-15]	Trimethylpentane (2,2,4-)
AE	[EPA TO-15]	Tert-butyl alcohol
AE	[EPA TO-15]	Tetrachloroethane (1,1,2,2-)
AE	[EPA TO-15]	Tetrachloroethene
AE	[EPA TO-15]	Tetrahydrofuran
AE	[EPA TO-15]	Toluene
AE	[EPA TO-15]	Trichloroethane (1,1,1-)
AE	[EPA TO-15]	Trichloroethane (1,1,2-)
AE	[EPA TO-15]	Trichloroethene
AE	[EPA TO-15]	Trichlorofluoromethane
AE	[EPA TO-15]	Trichloro (1,1,2-) trifluoroethane (1,2,2-)
AE	[EPA TO-15]	Vinyl acetate
AE	[EPA TO-15]	Vinyl bromide
AE	[EPA TO-15]	Vinyl chloride
AE	[EPA TO-15]	Xylene (m-)
AE	[EPA TO-15]	Xylene (o-)
AE	[EPA TO-15]	Xylene (p-)
AE	[EPA TO-15]	Xylenes (total)

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Matrix	Approved Method	Parameter Description
DW	[EPA 1622] [EPA 1623]	Cryptosporidium
DW	[EPA 180.1] [SM 2130 B]	Turbidity
DW	[EPA 353.2] [SM 4500-NO3 F]	Nitrate
DW	[EPA 300.0] [SM 4110 B]	Nitrate
DW	[EPA 353.2] [SM 4500-NO3 F]	Nitrite
DW	[EPA 300.0] [SM 4110 B]	Nitrite
DW	[EPA 300.0] [SM 4110 B]	Fluoride
DW	[SM 4500-CN C,G]	Cyanide
DW	[SM 4500-CN C,E]	Cyanide
DW	[OTHER Kelada-01]	Cyanide
DW	[EPA 335.4]	Cyanide
DW	[EPA 300.0] [SM 4110 B]	Sulfate
DW	[EPA 200.7]	Sodium
DW	[SM 2540 C]	Total dissolved solids (TDS)
DW	[EPA 200.7]	Calcium
DW	[SM 3500-Ca D (18/19th ed)] [SM 3500-Ca B (20th ed)]	Calcium-hardness
DW	[EPA 200.7]	Calcium-hardness
DW	[EPA 200.7] [SM 3120B/3111B or 2340 B]	Total hardness
DW	[SM 2340 C]	Total hardness
DW	[SM 2320 B]	Alkalinity
DW	[EPA 350.1] [SM 4500-NH3 G]	Ammonia
DW	[EPA 300.0]	Bromide
DW	[EPA 300.0] [SM 4110]	Chloride
DW	[EPA 300.0]	Chlorate
DW	[EPA 314.0]	Perchlorate
DW	[EPA 300.0] [EPA 300.1]	Chlorite (monthly)
DW	[SM 2120 B]	Color
DW	[SM 5540 C]	Foaming agents
DW	[SM 2150 B]	Odor
DW	[SM 2510 B]	Conductivity
DW	[SM 4500-P E]	Orthophosphate
DW	[SM 5310 C]	Total organic carbon (TOC)
DW	[SM 5320 B]	Total organic halides (TOX)
DW	[SM 5910B]	UV-absorbing compounds
DW	[SM 4500-Cl G]	Chlorine - residual
DW	[SM 4500-H B] [EPA 150.1]	pH
DW	[EPA 200.7]	Aluminum
DW	[EPA 200.8]	Antimony
DW	[EPA 200.8]	Arsenic
DW	[EPA 200.7]	Barium

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Matrix	Approved Method	Parameter Description
DW	[EPA 200.8]	Barium
DW	[EPA 200.7]	Beryllium
DW	[EPA 200.8]	Beryllium
DW	[EPA 200.7]	Cadmium
DW	[EPA 200.8]	Cadmium
DW	[EPA 200.7]	Chromium
DW	[EPA 200.8]	Chromium
DW	[EPA 200.7]	Copper
DW	[EPA 200.8]	Copper
DW	[EPA 200.7]	Iron
DW	[EPA 200.8]	Lead
DW	[EPA 200.7]	Magnesium
DW	[EPA 200.7]	Manganese
DW	[EPA 200.8]	Manganese
DW	[EPA 245.1]	Mercury
DW	[EPA 200.7]	Nickel
DW	[EPA 200.8]	Nickel
DW	[EPA 200.8]	Selenium
DW	[EPA 200.7]	Silver
DW	[EPA 200.8]	Silver
DW	[EPA 200.8]	Thallium
DW	[EPA 200.7]	Zinc
DW	[EPA 200.8]	Zinc
DW	[EPA 507]	Alachlor
DW	[EPA 507]	Atrazine
DW	[EPA 507]	Simazine
DW	[EPA 507]	Butachlor
DW	[EPA 507]	Metolachlor
DW	[EPA 507]	Metribuzin
DW	[EPA 504.1]	Dibromoethane (1,2-) (EDB)
DW	[EPA 504.1]	Dibromo-3-chloropropane (1,2-)
DW	[EPA 504.1]	Trichloropropane (1,2,3-)
DW	[EPA 515.1]	D (2,4-)
DW	[EPA 515.1]	Dalapon
DW	[EPA 515.1]	Dinoseb
DW	[EPA 515.1]	TP (2,4,5-) (Silvex)
DW	[EPA 515.1]	DB (2,4-)
DW	[EPA 515.1]	Dicamba
DW	[EPA 515.1]	Dichlorprop
DW	[EPA 515.1]	T (2,4,5-)
DW	[EPA 552.2]	Bromochloroacetic acid

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Matrix	Approved Method	Parameter Description
DW	[EPA 552.2]	Dibromoacetic acid
DW	[EPA 552.2]	Dichloroacetic acid
DW	[EPA 552.2]	Monobromoacetic acid (MBAA)
DW	[EPA 552.2]	Monochloroacetic acid (MCAA)
DW	[EPA 552.2]	Trichloroacetic acid
DW	[EPA 508]	Endrin
DW	[EPA 508]	Heptachlor
DW	[EPA 508]	Heptachlor epoxide
DW	[EPA 508]	Hexachlorobenzene
DW	[EPA 508]	Hexachlorocyclopentadiene
DW	[EPA 508]	Lindane (gamma BHC)
DW	[EPA 508]	Methoxychlor
DW	[EPA 508]	Chlordane (technical)
DW	[EPA 508]	Toxaphene
DW	[EPA 508]	Aldrin
DW	[EPA 508]	Alpha BHC
DW	[EPA 508]	Beta BHC
DW	[EPA 508]	Delta BHC
DW	[EPA 508]	DDD (4,4'-)
DW	[EPA 508]	DDE (4,4'-)
DW	[EPA 508]	DDT (4,4'-)
DW	[EPA 508]	Dieldrin
DW	[EPA 508]	Endosulfan I
DW	[EPA 508]	Endosulfan II
DW	[EPA 508]	Endosulfan sulfate
DW	[EPA 508]	Endrin aldehyde
DW	[EPA 508]	Endrin ketone
DW	[EPA 524.2]	Bromoform
DW	[EPA 524.2]	Chloroform
DW	[EPA 524.2]	Dibromochloromethane
DW	[EPA 524.2]	Bromodichloromethane
DW	[EPA 524.2]	Benzene
DW	[EPA 524.2]	Carbon tetrachloride
DW	[EPA 524.2]	Chlorobenzene
DW	[EPA 524.2]	Dichlorobenzene (1,2-)
DW	[EPA 524.2]	Dichlorobenzene (1,3-)
DW	[EPA 524.2]	Dichlorobenzene (1,4-)
DW	[EPA 524.2]	Dichloroethane (1,1-)
DW	[EPA 524.2]	Dichloroethane (1,2-)
DW	[EPA 524.2]	Dichloroethene (cis-1,2-)
DW	[EPA 524.2]	Dichloroethene (trans-1,2-)

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Matrix	Approved Method	Parameter Description
DW	[EPA 524.2]	Methylene chloride (Dichloromethane)
DW	[EPA 524.2]	Dichloropropane (1,2-)
DW	[EPA 524.2]	Ethylbenzene
DW	[EPA 524.2]	Methyl tert-butyl ether
DW	[EPA 524.2]	Naphthalene
DW	[EPA 524.2]	Styrene
DW	[EPA 524.2]	Tetrachloroethane (1,1,2,2-)
DW	[EPA 524.2]	Tetrachloroethene
DW	[EPA 524.2]	Trichloroethane (1,1,1-)
DW	[EPA 524.2]	Trichloroethene
DW	[EPA 524.2]	Toluene
DW	[EPA 524.2]	Trichlorobenzene (1,2,4-)
DW	[EPA 524.2]	Dichloroethene (1,1-)
DW	[EPA 524.2]	Trichloroethane (1,1,2-)
DW	[EPA 524.2]	Vinyl chloride
DW	[EPA 524.2]	Xylenes (total)
DW	[EPA 524.2]	Bromobenzene
DW	[EPA 524.2]	Bromochloromethane
DW	[EPA 524.2]	Bromomethane
DW	[EPA 524.2]	Butyl benzene (n-)
DW	[EPA 524.2]	Sec-butylbenzene
DW	[EPA 524.2]	Tert-butylbenzene
DW	[EPA 524.2]	Chloroethane
DW	[EPA 524.2]	Chloromethane
DW	[EPA 524.2]	Chlorotoluene (2-)
DW	[EPA 524.2]	Chlorotoluene (4-)
DW	[EPA 524.2]	Dibromo-3-chloropropane (1,2-)
DW	[EPA 524.2]	Dibromoethane (1,2-) (EDB)
DW	[EPA 524.2]	Dibromomethane
DW	[EPA 524.2]	Dichlorodifluoromethane
DW	[EPA 524.2]	Dichloropropane (1,3-)
DW	[EPA 524.2]	Dichloropropane (2,2-)
DW	[EPA 524.2]	Dichloropropene (1,1-)
DW	[EPA 524.2]	Dichloropropene (cis-1,3-)
DW	[EPA 524.2]	Dichloropropene (trans-1,3-)
DW	[EPA 524.2]	Hexachlorobutadiene (1,3-)
DW	[EPA 524.2]	Isopropylbenzene
DW	[EPA 524.2]	Isopropyltoluene (4-)
DW	[EPA 524.2]	Propylbenzene (n-)
DW	[EPA 524.2]	Tetrachloroethane (1,1,1,2-)
DW	[EPA 524.2]	Trichlorobenzene (1,2,3-)

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Matrix	Approved Method	Parameter Description
DW	[EPA 524.2]	Trichlorobenzene (1,3,5-)
DW	[EPA 524.2]	Trichlorofluoromethane
DW	[EPA 524.2]	Trichloropropane (1,2,3-)
DW	[EPA 524.2]	Trimethylbenzene (1,2,4-)
DW	[EPA 524.2]	Trimethylbenzene (1,3,5-)
DW	[EPA 524.2]	Acetone
DW	[EPA 524.2]	Butanone (2-)
DW	[EPA 524.2]	Carbon disulfide
DW	[EPA 524.2]	Dichloro-2-butene (trans-1,4-)
DW	[EPA 524.2]	Hexachloroethane
DW	[EPA 524.2]	Hexanone (2-)
DW	[EPA 524.2]	Methyl iodide
DW	[EPA 524.2]	Pentanone (4-methyl-2-)
NPW	[SW-846 3005A]	Metals, Total Rec and Dissolved
NPW	[SW-846 3010A]	Metals, Total
NPW	[SW-846 3020A]	Metals
NPW	[SW-846 3015A] [SW-846 3015]	Metals
NPW	[SW-846 6020A] [SW-846 6020]	Beryllium
NPW	[SW-846 7199]	Chromium (VI)
NPW	[SW-846 3510C]	Semivolatile organics
NPW	[SW-846 3511]	Semivolatile organics
NPW	[SW-846 3520C]	Semivolatile organics
NPW	[USER DEFINED 5030C] [SW-846 5030B]	Volatile organics
NPW	[OTHER J. Chrom. Sci. RSK-175]	Ethane
NPW	[OTHER J. Chrom. Sci. RSK-175]	Ethene
NPW	[OTHER J. Chrom. Sci. RSK-175]	Methane
NPW	[SW-846 9020B]	Total organic halides (TOX)
NPW	[SW-846 9050A]	Specific conductance
NPW	[SW-846 9066]	Phenols
NPW	[USER DEFINED SW-846 8330]	Nitroguanidine
NPW	[USER DEFINED EPA 353.2 Modified]	Nitrocellulose
NPW	[SM 9222 D]	Fecal coliform
NPW	[SM 9222 B]	Total coliform
NPW	[SM 9230 C]	Fecal streptococci
NPW	[SM 9215 B]	Heterotrophic plate count
NPW	[ASTM D1067] [SM 2310 B(4A)]	Acidity as CaCO3
NPW	[SM 2320 B]	Alkalinity as CaCO3
NPW	[EPA 310.2]	Alkalinity as CaCO3
NPW	[EPA 350.1] [SM 4500-NH3 B+G (19/20th ed.)]	Ammonia

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Matrix	Approved Method	Parameter Description
NPW	[SM 5210 B]	Biochemical oxygen demand
NPW	[EPA 200.7]	Boron
NPW	[EPA 300.0]	Bromide
NPW	[EPA 200.7]	Calcium
NPW	[SM 5210 B]	Carbonaceous BOD (CBOD)
NPW	[EPA 410.4] [SM 5220 D]	Chemical oxygen demand
NPW	[EPA 300.0] [SM 4110 B]	Chloride
NPW	[SM 2120 B]	Color
NPW	[SM 4500-CN C, E]	Cyanide
NPW	[OTHER Kelada-01]	Cyanide
NPW	[EPA 335.4]	Cyanide
NPW	[SM 4500-CN C,G]	Cyanide - amenable to Cl2
NPW	[OTHER Kelada-01]	Cyanide - amenable to Cl2
NPW	[EPA 300.0] [SM 4110 B]	Fluoride
NPW	[EPA 130.1]	Hardness - total as CaCO3
NPW	[SM 2340 B or C]	Hardness - total as CaCO3
NPW	[EPA 200.7]	Hardness - total as CaCO3
NPW	[SM 4500-N Org B or C + NH3 B + NH3 C (19/20th ed)]	Kjeldahl nitrogen - total
NPW	[EPA 351.2]	Kjeldahl nitrogen - total
NPW	[EPA 200.7]	Magnesium
NPW	[EPA 300.0] [SM 4110 B]	Nitrate
NPW	[EPA 353.2] [SM 4500-NO3 F]	Nitrate - nitrite
NPW	[EPA 300.0] [SM 4110 B]	Nitrate - nitrite
NPW	[EPA 300.0] [SM 4110 B]	Nitrite
NPW	[SM 5520 B]	Oil & grease - total recov
NPW	[EPA 1664A] [SM 5520 B]	Oil & grease - hem-LL
NPW	[EPA 1664A]	Oil & grease - hem-SPE
NPW	[EPA 1664A]	Oil & grease - sgt-non polar
NPW	[EPA 1664A]	Oil & grease - non polar
NPW	[SM 5310 B, C or D]	Total organic carbon (TOC)
NPW	[SM 5320 B]	Total organic halides (TOX)
NPW	[EPA 351.1,.2, .3,.4 - 350.1 .2 .3] [SM 4500-NH3 B, C, D, E, F, G, H]	Organic nitrogen
NPW	[SM 4500-P, E]	Orthophosphate
NPW	[EPA 420.1 + .4]	Phenols
NPW	[SM 4500-P B5 + E]	Phosphorus (total)
NPW	[EPA 200.7]	Potassium
NPW	[SM 2540 B]	Residue - total
NPW	[SM 2540 C]	Residue - filterable (TDS)
NPW	[SM 2540 D]	Residue - nonfilterable (TSS)
NPW	[SM 2540 F]	Residue - settleable

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Matrix	Approved Method	Parameter Description
NPW	[EPA 160.4]	Residue - volatile
NPW	[EPA 200.7]	Silica - dissolved
NPW	[EPA 200.7]	Sodium
NPW	[EPA 120.1] [SM 2510 B]	Specific conductance
NPW	[EPA 300.0] [SM 4110 B]	Sulfate
NPW	[SM 4500-S D]	Sulfides
NPW	[SM 5540 C]	Surfactants
NPW	[EPA 180.1] [SM 2130 B]	Turbidity
NPW	[SM 4500-Cl G]	Chlorine
NPW	[SM 4500-Cl G]	Chlorine
NPW	[SM 4500-O C]	Oxygen (dissolved)
NPW	[SM 4500-O G]	Oxygen (dissolved)
NPW	[SM 4500-H B]	pH
NPW	[SM 4500-SO3 B]	Sulfite - SO3
NPW	[SM 2550 B]	Temperature
NPW	[EPA 200.7]	Aluminum
NPW	[EPA 200.7]	Antimony
NPW	[EPA 200.8]	Antimony
NPW	[EPA 200.7]	Arsenic
NPW	[EPA 200.8]	Arsenic
NPW	[EPA 200.7]	Barium
NPW	[EPA 200.8]	Barium
NPW	[EPA 200.7]	Beryllium
NPW	[EPA 200.8]	Beryllium
NPW	[EPA 200.7]	Cadmium
NPW	[EPA 200.8]	Cadmium
NPW	[SM 3500-Cr D (18/19th ed)] [SM 3500-Cr B (20th ed)]	Chromium (VI)
NPW	[EPA 218.6] [SM 3500-Cr C (20th ed)]	Chromium (VI)
NPW	[EPA 200.7]	Chromium
NPW	[EPA 200.8]	Chromium
NPW	[EPA 200.7]	Cobalt
NPW	[EPA 200.7]	Copper
NPW	[EPA 200.8]	Copper
NPW	[EPA 200.7]	Iron
NPW	[SM 3500 Fe B (SM 20)]	Iron
NPW	[SM 3500-Fe B (20th ed)]	Iron, Ferrous
NPW	[EPA 200.7]	Lead
NPW	[EPA 200.8]	Lead
NPW	[EPA 200.7]	Manganese
NPW	[EPA 200.8]	Manganese
NPW	[EPA 245.1]	Mercury

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Matrix	Approved Method	Parameter Description
NPW	[EPA 1631E]	Mercury
NPW	[EPA 200.7]	Molybdenum
NPW	[EPA 200.8]	Molybdenum
NPW	[EPA 200.7]	Nickel
NPW	[EPA 200.8]	Nickel
NPW	[EPA 200.7]	Selenium
NPW	[EPA 200.8]	Selenium
NPW	[EPA 200.7]	Silver
NPW	[EPA 200.8]	Silver
NPW	[EPA 200.7]	Thallium
NPW	[EPA 200.8]	Thallium
NPW	[EPA 200.7]	Tin
NPW	[EPA 200.8]	Tin
NPW	[EPA 200.7]	Titanium
NPW	[EPA 200.7]	Vanadium
NPW	[EPA 200.8]	Vanadium
NPW	[EPA 200.7]	Zinc
NPW	[EPA 200.8]	Zinc
NPW	[EPA 602] [USER DEFINED SM 6200C 20th ED]	Benzene
NPW	[EPA 602] [USER DEFINED SM 6200C 20th ED]	Ethylbenzene
NPW	[EPA 602] [USER DEFINED SM 6200C 20th ED]	Methyl tert-butyl ether
NPW	[EPA 602] [USER DEFINED SM 6200C 20th ED]	Tert-butyl alcohol
NPW	[EPA 602] [USER DEFINED SM 6200C 20th ED]	Toluene
NPW	[EPA 602] [USER DEFINED SM 6200C 20th ED]	Xylenes (total)
NPW	[EPA 608] [SM 6630 B] [SM 6630 C]	Aldrin
NPW	[EPA 608] [SM 6630 B] [SM 6630 C]	Alpha BHC
NPW	[EPA 608] [SM 6630 C]	Beta BHC
NPW	[EPA 608] [SM 6630 C]	Delta BHC
NPW	[EPA 608] [SM 6630 B] [SM 6630 C]	Lindane (gamma BHC)
NPW	[EPA 608] [SM 6630 B] [SM 6630 C]	Chlordane
NPW	[EPA 608] [USER DEFINED SM 6630C]	Chlordane (alpha)
NPW	[EPA 608] [USER DEFINED SM 6630C]	Chlordane (gamma)
NPW	[EPA 608]	Chloroneb
NPW	[EPA 608]	Chlorothalonil

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Matrix	Approved Method	Parameter Description
NPW	[EPA 608] [SM 6630 B] [SM 6630 C]	DDD (4,4'-)
NPW	[EPA 608] [SM 6630 B] [SM 6630 C]	DDE (4,4'-)
NPW	[EPA 608] [SM 6630 B] [SM 6630 C]	DDT (4,4'-)
NPW	[EPA 608] [SM 6630 B] [SM 6630 C]	Dieldrin
NPW	[EPA 608] [SM 6630 B] [SM 6630 C]	Endosulfan I
NPW	[EPA 608] [SM 6630 B] [SM 6630 C]	Endosulfan II
NPW	[EPA 608] [SM 6630 C]	Endosulfan sulfate
NPW	[EPA 608] [SM 6630 B] [SM 6630 C]	Endrin
NPW	[EPA 608] [USER DEFINED SM 6630C]	Endrin aldehyde
NPW	[EPA 608] [USER DEFINED SM 6630C]	Endrin ketone
NPW	[EPA 608] [SM 6630 B] [SM 6630 C]	Heptachlor
NPW	[EPA 608] [SM 6630 B] [SM 6630 C]	Heptachlor epoxide
NPW	[EPA 608] [USER DEFINED SM 6630C]	Hexachlorobenzene
NPW	[EPA 608] [SM 6630 B] [SM 6630 C]	Methoxychlor
NPW	[EPA 608] [SM 6630 B] [SM 6630 C]	Toxaphene
NPW	[EPA 608]	PCB 1016
NPW	[EPA 608]	PCB 1221
NPW	[EPA 608]	PCB 1232
NPW	[EPA 608]	PCB 1242
NPW	[EPA 608]	PCB 1248
NPW	[EPA 608]	PCB 1254
NPW	[EPA 608]	PCB 1260
NPW	[EPA 610] [SM 6440 B]	Acenaphthene
NPW	[EPA 610] [SM 6440 B]	Acenaphthylene
NPW	[EPA 610] [SM 6440 B]	Anthracene
NPW	[EPA 610] [SM 6440 B]	Benzo(a)anthracene
NPW	[EPA 610] [SM 6440 B]	Benzo(a)pyrene
NPW	[EPA 610] [SM 6440 B]	Benzo(b)fluoranthene
NPW	[EPA 610] [SM 6440 B]	Benzo(ghi)perylene
NPW	[EPA 610] [SM 6440 B]	Benzo(k)fluoranthene
NPW	[EPA 610] [SM 6440 B]	Chrysene
NPW	[EPA 610] [SM 6440 B]	Dibenzo(a,h)anthracene
NPW	[EPA 610] [SM 6440 B]	Fluoranthene
NPW	[EPA 610] [SM 6440 B]	Fluorene
NPW	[EPA 610] [SM 6440 B]	Indeno(1,2,3-cd)pyrene
NPW	[EPA 610] [SM 6440 B]	Naphthalene
NPW	[EPA 610] [SM 6440 B]	Phenanthrene
NPW	[EPA 610] [SM 6440 B]	Pyrene
NPW	[EPA 624] [SM 6200 B]	Allyl chloride

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Matrix	Approved Method	Parameter Description
NPW	[EPA 624]	Amyl alcohol (n-)
NPW	[EPA 624] [USER DEFINED SM 6200 B]	Acetone
NPW	[EPA 624] [SM 6200 B]	Acrolein
NPW	[EPA 624] [SM 6200 B]	Acrylonitrile
NPW	[EPA 624] [USER DEFINED SM 6200 B]	Benzene
NPW	[EPA 624] [SM 6200 B]	Bromobenzene
NPW	[EPA 624] [SM 6200 B]	Bromochloromethane
NPW	[EPA 624] [USER DEFINED SM 6200 B]	Bromodichloromethane
NPW	[EPA 624] [SM 6200 B]	Bromoethane
NPW	[EPA 624] [USER DEFINED SM 6200 B]	Bromoform
NPW	[EPA 624] [USER DEFINED SM 6200 B]	Bromomethane
NPW	[EPA 624] [USER DEFINED SM 6200 B]	Butanone (2-)
NPW	[EPA 624] [SM 6200 B]	Butadiene (2-chloro-1,3-)
NPW	[EPA 624] [SM 6200 B]	Butyl benzene (n-)
NPW	[EPA 624] [USER DEFINED SM 6200 B]	Carbon disulfide
NPW	[EPA 624] [USER DEFINED SM 6200 B]	Carbon tetrachloride
NPW	[EPA 624] [USER DEFINED SM 6200 B]	Chlorobenzene
NPW	[EPA 624] [USER DEFINED SM 6200 B]	Chloroethane
NPW	[EPA 624] [USER DEFINED SM 6200 B]	Chloroethyl vinyl ether (2-)
NPW	[EPA 624] [USER DEFINED SM 6200 B]	Chloroform
NPW	[EPA 624] [USER DEFINED SM 6200 B]	Chloromethane
NPW	[EPA 624] [SM 6200 B]	Chlorotoluene (2-)
NPW	[EPA 624] [SM 6200 B]	Chlorotoluene (4-)
NPW	[EPA 624] [SM 6200 B]	Cyclohexanone
NPW	[EPA 624] [SM 6200 B]	Dibromo-3-chloropropane (1,2-)
NPW	[EPA 624] [USER DEFINED SM 6200 B]	Dibromochloromethane
NPW	[EPA 624] [USER DEFINED SM 6200 B]	Dibromoethane (1,2-) (EDB)
NPW	[EPA 624] [SM 6200 B]	Dibromomethane

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Matrix	Approved Method	Parameter Description
NPW	[EPA 624] [SM 6200 B]	Dichloro-2-butene (cis-1,4-)
NPW	[EPA 624] [USER DEFINED SM 6200 B]	Dichlorobenzene (1,2-)
NPW	[EPA 624] [USER DEFINED SM 6200 B]	Dichlorobenzene (1,3-)
NPW	[EPA 624] [USER DEFINED SM 6200 B]	Dichlorobenzene (1,4-)
NPW	[EPA 624] [SM 6200 B]	Dichloro-2-butene (trans-1,4-)
NPW	[EPA 624] [USER DEFINED SM 6200 B]	Dichlorodifluoromethane
NPW	[EPA 624] [USER DEFINED SM 6200 B]	Dichloroethane (1,1-)
NPW	[EPA 624] [USER DEFINED SM 6200 B]	Dichloroethane (1,2-)
NPW	[EPA 624] [USER DEFINED SM 6200 B]	Dichloroethene (1,1-)
NPW	[EPA 624] [USER DEFINED SM 6200 B]	Dichloroethene (cis-1,2-)
NPW	[EPA 624] [USER DEFINED SM 6200 B]	Dichloroethene (trans-1,2-)
NPW	[EPA 624] [USER DEFINED SM 6200 B]	Dichloropropane (1,2-)
NPW	[EPA 624] [SM 6200 B]	Dichloropropane (1,3-)
NPW	[EPA 624] [SM 6200 B]	Dichloropropane (2,2-)
NPW	[EPA 624] [SM 6200 B]	Dichloropropene (1,1-)
NPW	[EPA 624] [SM 6200 B]	Diethyl ether (Ethyl ether)
NPW	[EPA 624] [USER DEFINED SM 6200 B]	Dichloropropene (cis-1,3-)
NPW	[EPA 624] [USER DEFINED SM 6200 B]	Dichloropropene (trans-1,3-)
NPW	[EPA 624] [USER DEFINED SM 6200 B]	Ethyl acetate
NPW	[EPA 624] [USER DEFINED SM 6200 B]	Ethylbenzene
NPW	[EPA 624] [USER DEFINED SM 6200 B]	Hexane (n-)
NPW	[EPA 624] [USER DEFINED SM 6200 B]	Isopropanol
NPW	[EPA 624] [USER DEFINED SM 6200 B]	Methylene chloride (Dichloromethane)
NPW	[EPA 624] [USER DEFINED SM 6200 B]	Methyl tert-butyl ether
NPW	[EPA 624] [USER DEFINED SM 6200	Methyl isobutyl ketone

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Matrix	Approved Method	Parameter Description
	B]	
NPW	[EPA 624] [USER DEFINED SM 6200 B]	Tert-butyl alcohol
NPW	[EPA 624] [USER DEFINED SM 6200 B]	Tetrahydrofuran
NPW	[EPA 624] [USER DEFINED SM 6200 B]	Styrene
NPW	[EPA 624] [USER DEFINED SM 6200 B]	Tetrachloroethane (1,1,2,2-)
NPW	[EPA 624] [SM 6200 B]	Tetrachloroethane (1,1,1,2-)
NPW	[EPA 624] [USER DEFINED SM 6200 B]	Tetrachloroethene
NPW	[EPA 624] [USER DEFINED SM 6200 B]	Toluene
NPW	[EPA 624] [USER DEFINED SM 6200 B]	Trichloroethane (1,1,1-)
NPW	[EPA 624] [USER DEFINED SM 6200 B]	Trichloroethane (1,1,2-)
NPW	[EPA 624] [USER DEFINED SM 6200 B]	Trichloroethene
NPW	[EPA 624] [USER DEFINED SM 6200 B]	Trichlorofluoromethane
NPW	[EPA 624] [USER DEFINED SM 6200 B]	Trichloro (1,1,2-) trifluoroethane (1,2,2-)
NPW	[EPA 624] [USER DEFINED SM 6200 B]	Vinyl acetate
NPW	[EPA 624] [USER DEFINED SM 6200 B]	Vinyl chloride
NPW	[EPA 624] [USER DEFINED SM 6200 B]	Xylenes (total)
NPW	[EPA 624] [SM 6200 B]	Xylene (m-)
NPW	[EPA 624] [USER DEFINED SM 6200 B]	Xylene (o-)
NPW	[EPA 624] [SM 6200 B]	Xylene (p-)
NPW	[EPA 624] [USER DEFINED SM 6200 B]	Acetonitrile
NPW	[EPA 624]	Cyclohexane
NPW	[EPA 624] [USER DEFINED SM 6200 B]	Hexanone (2-)
NPW	[EPA 624]	Methyl acetate
NPW	[EPA 624]	Methylcyclohexane
NPW	[EPA 624] [USER DEFINED SM 6200 B]	Methyl iodide

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Matrix	Approved Method	Parameter Description
NPW	[EPA 624] [SM 6200 B]	Ethyl-tert-butyl Ether [ETBE]
NPW	[EPA 624] [SM 6200 B]	Diisopropyl Ether [DIPE]
NPW	[EPA 624] [SM 6200 B]	Dioxane (1,4-)
NPW	[EPA 624]	Butanol (1-)
NPW	[EPA 624] [SM 6200 B]	Ethanol
NPW	[EPA 624] [SM 6200 B]	Ethyl methacrylate
NPW	[EPA 624] [SM 6200 B]	Hexachlorobutadiene (1,3-)
NPW	[EPA 624] [SM 6200 B]	Iso-butyl alcohol
NPW	[EPA 624] [SM 6200 B]	Isopropylbenzene
NPW	[EPA 624] [SM 6200 B]	Isopropyltoluene (4-)
NPW	[EPA 624] [SM 6200 B]	Methacrylonitrile
NPW	[EPA 624] [SM 6200 B]	Methyl methacrylate
NPW	[EPA 624] [SM 6200 B]	Naphthalene
NPW	[EPA 624]	Octane (-n)
NPW	[EPA 624]	Nitropropane (2-)
NPW	[EPA 624] [SM 6200 B]	Propionitrile
NPW	[EPA 624] [SM 6200 B]	Pentachloroethane
NPW	[EPA 624] [SM 6200 B]	Propylbenzene (n-)
NPW	[EPA 624] [SM 6200 B]	Sec-butylbenzene
NPW	[EPA 624] [SM 6200 B]	tert-Amylmethyl ether [TAME]
NPW	[EPA 624] [SM 6200 B]	Tert-butylbenzene
NPW	[EPA 624] [SM 6200 B]	Trichlorobenzene (1,2,3-)
NPW	[EPA 624] [SM 6200 B]	Trichlorobenzene (1,2,4-)
NPW	[EPA 624] [SM 6200 B]	Trichloropropane (1,2,3-)
NPW	[EPA 624] [SM 6200 B]	Trimethylbenzene (1,2,3-)
NPW	[EPA 624] [SM 6200 B]	Trimethylbenzene (1,2,4-)
NPW	[EPA 624] [SM 6200 B]	Trimethylbenzene (1,3,5-)
NPW	[EPA 625] [SM 6410 B]	Acenaphthene
NPW	[EPA 625] [SM 6410 B]	Acenaphthylene
NPW	[EPA 625] [SM 6410 B]	Anthracene
NPW	[EPA 625] [SM 6410 B]	Benzo(a)anthracene
NPW	[EPA 625] [SM 6410 B]	Benzo(b)fluoranthene
NPW	[EPA 625] [SM 6410 B]	Benzo(k)fluoranthene
NPW	[EPA 625] [SM 6410 B]	Benzo(a)pyrene
NPW	[EPA 625] [SM 6410 B]	Benzo(ghi)perylene
NPW	[EPA 625] [SM 6410 B]	Butyl benzyl phthalate
NPW	[EPA 625] [SM 6410 B]	Bis (2-chloroethyl) ether
NPW	[EPA 625] [SM 6410 B]	Bis (2-chloroethoxy) methane
NPW	[EPA 625] [SM 6410 B]	Bis (2-ethylhexyl) phthalate
NPW	[EPA 625] [SM 6410 B]	Bis (2-chloroisopropyl) ether
NPW	[EPA 625] [SM 6410 B]	Bromophenyl-phenyl ether (4-)

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Matrix	Approved Method	Parameter Description
NPW	[EPA 625] [SM 6410 B]	Biphenylamine (4-)
NPW	[EPA 625] [SM 6410 B]	Chloronaphthalene (2-)
NPW	[EPA 625] [SM 6410 B]	Chlorophenyl-phenyl ether (4-)
NPW	[EPA 625] [SM 6410 B]	Chrysene
NPW	[EPA 625] [SM 6410 B]	Chloronaphthalene (1-)
NPW	[EPA 625] [SM 6410 B]	Dibenzo(a,h)anthracene
NPW	[EPA 625]	Dibenzofuran
NPW	[EPA 625] [SM 6410 B]	Di-n-butyl phthalate
NPW	[EPA 625] [SM 6410 B]	Dichlorobenzidine (3,3'-)
NPW	[EPA 625] [SM 6410 B]	Diethyl phthalate
NPW	[EPA 625] [SM 6410 B]	Dimethyl phthalate
NPW	[EPA 625] [SM 6410 B]	Dinitrotoluene (2,4-)
NPW	[EPA 625] [SM 6410 B]	Dinitrotoluene (2,6-)
NPW	[EPA 625] [SM 6410 B]	Di-n-octyl phthalate
NPW	[EPA 625] [SM 6410 B]	Famphur
NPW	[EPA 625] [SM 6410 B]	Fluoranthene
NPW	[EPA 625] [SM 6410 B]	Fluorene
NPW	[EPA 625] [SM 6410 B]	Hexachlorobenzene
NPW	[EPA 625] [SM 6410 B]	Hexachlorobutadiene (1,3-)
NPW	[EPA 625] [SM 6410 B]	Hexachloroethane
NPW	[EPA 625] [SM 6410 B]	Hexachlorophene
NPW	[EPA 625] [SM 6410 B]	Hexachloropropene
NPW	[EPA 625] [SM 6410 B]	Indeno(1,2,3-cd)pyrene
NPW	[EPA 625] [SM 6410B]	Isophorone
NPW	[EPA 625] [SM 6410 B]	Kepone
NPW	[EPA 625]	Methylnaphthalene (2-)
NPW	[EPA 625] [SM 6410 B]	Naphthalene
NPW	[EPA 625] [SM 6410 B]	Napththylamine (1-)
NPW	[EPA 625] [SM 6410 B]	Napththylamine (2-)
NPW	[EPA 625]	Chloroaniline (4-)
NPW	[EPA 625]	Nitroaniline (2-)
NPW	[EPA 625]	Nitroaniline (3-)
NPW	[EPA 625]	Nitroaniline (4-)
NPW	[EPA 625] [SM 6410 B]	Nitrobenzene
NPW	[EPA 625] [SM 6410 B]	N-Nitroso-di-n-propylamine
NPW	[EPA 625] [SM 6410 B]	Phenanthrene
NPW	[EPA 625] [SM 6410 B]	Pyrene
NPW	[EPA 625]	Pentachlorobenzene
NPW	[EPA 625]	Tetrachlorobenzene (1,2,4,5-)
NPW	[EPA 625] [SM 6410 B]	Trichlorobenzene (1,2,4-)
NPW	[EPA 625] [SM 6410 B]	Methyl phenol (4-chloro-3-)

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Matrix	Approved Method	Parameter Description
NPW	[EPA 625] [SM 6410 B]	Chlorophenol (2-)
NPW	[EPA 625] [SM 6410 B]	Dichlorophenol (2,4-)
NPW	[EPA 625] [SM 6410 B]	Dimethylphenol (2,4-)
NPW	[EPA 625] [SM 6410 B]	Dinitrophenol (2,4-)
NPW	[EPA 625] [SM 6410 B]	Dinitrophenol (2-methyl-4,6-)
NPW	[EPA 625] [SM 6410 B]	Nitrophenol (2-)
NPW	[EPA 625] [SM 6410 B]	Nitrophenol (4-)
NPW	[EPA 625] [SM 6410 B]	Pentachlorophenol
NPW	[EPA 625] [SM 6410 B]	Phenol
NPW	[EPA 625]	Trichlorophenol (2,4,5-)
NPW	[EPA 625] [SM 6410 B]	Trichlorophenol (2,4,6-)
NPW	[EPA 625] [SM 6410 B]	Benzoic acid
NPW	[SM 6410 B] [EPA 625]	Methylphenol (4-)
NPW	[EPA 625] [SM 6410 B]	Acetophenone
NPW	[EPA 625] [SM 6410 B]	Alpha - terpineol
NPW	[EPA 625] [SM 6410 B]	Aniline
NPW	[EPA 625] [SM 6410 B]	Benzidine
NPW	[EPA 625] [SM 6410 B]	Carbazole
NPW	[EPA 625] [SM 6410 B]	Dichloroaniline (2,3-)
NPW	[EPA 625]	Diphenylhydrazine (1,2-)
NPW	[EPA 625] [SM 6410 B]	Methylphenol (2-)
NPW	[EPA 625] [SM 6410 B]	Decane (n-)
NPW	[EPA 625] [SM 6410 B]	Hexachlorocyclopentadiene
NPW	[EPA 625]	N-Nitroso-di-n-butylamine
NPW	[EPA 625]	N-Nitrosodiethylamine
NPW	[EPA 625] [SM 6410 B]	N-Nitrosodimethylamine
NPW	[EPA 625] [SM 6410 B]	N-Nitrosodiphenylamine
NPW	[EPA 625]	N-Nitrosopyrrolidine
NPW	[EPA 625] [SM 6410 B]	Octadecane (n-)
NPW	[EPA 625] [SM 6410 B]	Pentachloroethane
NPW	[EPA 625] [SM 6410 B]	Pyridine
NPW	[EPA 625] [SM 6410 B]	Napthoquinone (1,4-)
NPW	[EPA 507]	Alachlor
NPW	[USER DEFINED EPA 1657]	Azinphos methyl
NPW	[EPA 1657 or 622]	Bolstar
NPW	[EPA 1657, 508, or 622]	Chloropyrifos
NPW	[EPA 622] [EPA 1657]	Coumaphos
NPW	[SM 6640 B]	D (2,4-)
NPW	[EPA 515.5, 515.2, 615, 1658 or 555]	DB (2,4-)
NPW	[SM 6640B]	Dalapon
NPW	[EPA 1658]	Dalapon

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Matrix	Approved Method	Parameter Description
NPW	[EPA 622] [USER DEFINED EPA 1657]	Demeton (o-)
NPW	[EPA 622] [USER DEFINED EPA 1657]	Demeton (s-)
NPW	[USER DEFINED EPA 1657]	Diazinon
NPW	[EPA 615] [USER DEFINED SM 6640B 18/19th ED]	Dicamba
NPW	[EPA 1658]	Dichlorprop
NPW	[EPA 1657, 507, or 622]	Dichlorvos
NPW	[EPA 622] [EPA 1657]	Dimethoate
NPW	[EPA 515.5, 515.2, 1658, or 615] [USER DEFINED SM 6640B]	Dinoseb
NPW	[USER DEFINED EPA 1657]	Disulfoton
NPW	[EPA 1657]	EPN
NPW	[EPA 507, 1657 or 622]	Ethoprop
NPW	[SM 6630 C]	Etridiazole
NPW	[EPA 1657 or 622]	Fensulfothion
NPW	[EPA 1657 or 622]	Fenthion
NPW	[USER DEFINED EPA 1657]	Malathion
NPW	[EPA 555, 1658, or 615]	MCPA
NPW	[EPA 555, 1658, or 615]	MCPP
NPW	[EPA 507, 1657, or 622]	Merphos
NPW	[EPA 507]	Metribuzin
NPW	[EPA 507, 1657, or 622]	Mevinphos
NPW	[EPA 1657 or 632]	Naled
NPW	[EPA 1657 or 614] [EPA 622]	Parathion
NPW	[EPA 622] [USER DEFINED EPA 1657]	Parathion methyl
NPW	[EPA 1657 or 622]	Phorate
NPW	[EPA 1657]	Ronnel
NPW	[EPA 622] [EPA 1657]	Stirofos
NPW	[EPA 622] [EPA 1657]	Sulfotepp
NPW	[SM 6640 B]	T (2,4,5-)
NPW	[EPA 622] [EPA 1657]	TEPP
NPW	[SM 6640 B]	TP (2,4,5-) (Silvex)
NPW	[EPA 622] [EPA 1657]	Tokuthion [Protothiofos]
NPW	[EPA 1657]	Trichloronate
NPW	[SM 6630 B]	Trifluralin
NPW	[EPA 2002.0]	Toxicity - acute, FW organism
NPW	[EPA 2000.0]	Toxicity - acute, FW organism
NPW	[EPA 1000.0]	Toxicity - chronic, FW organism
NPW	[EPA 1002.0]	Toxicity - chronic, FW organism

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Matrix	Approved Method	Parameter Description
NPW, SCM	[SM 9222D + EPA 625/R-92/013 Appendix F]	Fecal coliform
NPW, SCM	[SM 9260D + EPA 625/R-92/013 Appendix F]	Salmonella sp. Bacteria
NPW, SCM	[SW-846 1010A] [SW-846 1010] [USER DEFINED ASTM D93]	Ignitability
NPW, SCM	[SW-846 9040B] [SW-846 9040C]	Corrosivity - pH waste, >20% water
NPW, SCM	[SW-846 1110] [SW-846 1110A]	Corrosivity toward steel
NPW, SCM	[SW-846 1311]	Volatile organics
NPW, SCM	[SW-846 1311]	Semivolatile organics
NPW, SCM	[SW-846 1311]	Metals
NPW, SCM	[SW-846 1310B] [SW-846 1310A]	Metals - organics
NPW, SCM	[SW-846 1312]	Metals - organics
NPW, SCM	[SW-846 1320]	Metals - organics
NPW, SCM	[SW-846 9040B] [SW-846 9040C]	pH
NPW, SCM	[SW-846 6010C] [SW-846 6010B]	Aluminum
NPW, SCM	[SW-846 6010C] [SW-846 6010B]	Antimony
NPW, SCM	[SW-846 6020A] [SW-846 6020]	Antimony
NPW, SCM	[SW-846 6010C] [SW-846 6010B]	Arsenic
NPW, SCM	[SW-846 6020A] [SW-846 6020]	Arsenic
NPW, SCM	[SW-846 6010C] [SW-846 6010B]	Barium
NPW, SCM	[SW-846 6020A] [SW-846 6020]	Barium
NPW, SCM	[SW-846 6010C] [SW-846 6010B]	Beryllium
NPW, SCM	[SW-846 6010C] [SW-846 6010B]	Boron
NPW, SCM	[SW-846 6010C] [SW-846 6010B]	Cadmium
NPW, SCM	[SW-846 6020A] [SW-846 6020]	Cadmium
NPW, SCM	[SW-846 6010C] [SW-846 6010B]	Calcium
NPW, SCM	[SW-846 6010C] [SW-846 6010B]	Chromium
NPW, SCM	[SW-846 6020A] [SW-846 6020]	Chromium
NPW, SCM	[SW-846 7196A]	Chromium (VI)
NPW, SCM	[SW-846 6010C] [SW-846 6010B]	Cobalt
NPW, SCM	[SW-846 6010C] [SW-846 6010B]	Copper
NPW, SCM	[SW-846 6020A] [SW-846 6020]	Copper
NPW, SCM	[SW-846 6010C] [SW-846 6010B]	Iron
NPW, SCM	[SW-846 6010C] [SW-846 6010B]	Lead
NPW, SCM	[SW-846 6020A] [SW-846 6020]	Lead
NPW, SCM	[SW-846 6010C] [SW-846 6010B]	Lithium
NPW, SCM	[SW-846 6010C] [SW-846 6010B]	Magnesium
NPW, SCM	[SW-846 6010C] [SW-846 6010B]	Manganese
NPW, SCM	[SW-846 6020A] [SW-846 6020]	Manganese
NPW, SCM	[SW-846 7470A]	Mercury - liquid waste
NPW, SCM	[SW-846 6010C] [SW-846 6010B]	Molybdenum

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Matrix	Approved Method	Parameter Description
NPW, SCM	[SW-846 6020A] [SW-846 6020]	Molybdenum
NPW, SCM	[SW-846 6010C] [SW-846 6010B]	Nickel
NPW, SCM	[SW-846 6020A] [SW-846 6020]	Nickel
NPW, SCM	[SW-846 6010C] [SW-846 6010B]	Potassium
NPW, SCM	[SW-846 6010C] [SW-846 6010B]	Selenium
NPW, SCM	[SW-846 6020A] [SW-846 6020]	Selenium
NPW, SCM	[SW-846 6010C] [SW-846 6010B]	Silver
NPW, SCM	[SW-846 6020A] [SW-846 6020]	Silver
NPW, SCM	[SW-846 6010C] [SW-846 6010B]	Sodium
NPW, SCM	[SW-846 6010B] [SW-846 6010C]	Strontium
NPW, SCM	[SW-846 6010C] [SW-846 6010B]	Thallium
NPW, SCM	[SW-846 6020A] [SW-846 6020]	Thallium
NPW, SCM	[SW-846 6010C] [SW-846 6010B]	Tin
NPW, SCM	[SW-846 6020] [SW-846 6020A]	Tin
NPW, SCM	[SW-846 6010B] [SW-846 6010C]	Titanium
NPW, SCM	[SW-846 6010C] [SW-846 6010B]	Vanadium
NPW, SCM	[SW-846 6020A] [SW-846 6020]	Vanadium
NPW, SCM	[SW-846 6010C] [SW-846 6010B]	Zinc
NPW, SCM	[SW-846 6020A] [SW-846 6020]	Zinc
NPW, SCM	[SW-846 8011]	Dibromoethane (1,2-) (EDB)
NPW, SCM	[SW-846 8011]	Dibromo-3-chloropropane (1,2-)
NPW, SCM	[SW-846 8015D] [SW-846 8015B]	Methyl alcohol (Methanol)
NPW, SCM	[SW-846 8015D] [SW-846 8015B]	Ethyl alcohol
NPW, SCM	[USER DEFINED MA-DEP-VPH, WI GRO, NW TPH Gx] [SW-846 8015D] [SW-846 8015B]	Gasoline range organic
NPW, SCM	[USER DEFINED MA-DEP-EPH, TN-EPH, WI DRO, NW TPH Dx] [SW-846 8015D] [SW-846 8015B]	Diesel range organic
NPW, SCM	[OTHER FL - PRO] [USER DEFINED TX 1005, TX 1006, CT ETPH, NW TPH ID]	Petroleum Organics
NPW, SCM	[OTHER IA - OA-1]	Petroleum Organics
NPW, SCM	[OTHER IA - OA-2]	Petroleum Organics
NPW, SCM	[USER DEFINED CA LUFT - diesel]	Petroleum Organics
NPW, SCM	[OTHER NJ-OQA-QAM-025, Rev. 7]	Petroleum Organics
NPW, SCM	[SW-846 8021B]	Benzene
NPW, SCM	[SW-846 8021B]	Ethylbenzene
NPW, SCM	[SW-846 8021B]	Toluene
NPW, SCM	[SW-846 8021B]	Xylene (o-)
NPW, SCM	[SW-846 8021B]	Xylene (m-)
NPW, SCM	[SW-846 8021B]	Xylene (p-)

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Matrix	Approved Method	Parameter Description
NPW, SCM	[SW-846 8021B]	Xylenes (total)
NPW, SCM	[SW-846 8021B]	Methyl tert-butyl ether
NPW, SCM	[SW-846 8081B] [SW-846 8081A]	Alachlor
NPW, SCM	[SW-846 8081B] [SW-846 8081A]	Aldrin
NPW, SCM	[SW-846 8081B] [SW-846 8081A]	Alpha BHC
NPW, SCM	[SW-846 8081B] [SW-846 8081A]	Beta BHC
NPW, SCM	[SW-846 8081B] [SW-846 8081A]	Delta BHC
NPW, SCM	[SW-846 8081B] [SW-846 8081A]	Lindane (gamma BHC)
NPW, SCM	[SW-846 8081B] [SW-846 8081A]	Chlordane (technical)
NPW, SCM	[SW-846 8081B] [SW-846 8081A]	Chlordane (alpha)
NPW, SCM	[SW-846 8081B] [SW-846 8081A]	Chlordane (gamma)
NPW, SCM	[SW-846 8081B] [SW-846 8081A]	Chloroneb
NPW, SCM	[SW-846 8081B] [SW-846 8081A]	Chlorothalonil
NPW, SCM	[SW-846 8081B] [SW-846 8081A]	DDD (4,4'-)
NPW, SCM	[SW-846 8081B] [SW-846 8081A]	DDE (4,4'-)
NPW, SCM	[SW-846 8081B] [SW-846 8081A]	DDT (4,4'-)
NPW, SCM	[SW-846 8081B] [SW-846 8081A]	Dieldrin
NPW, SCM	[SW-846 8081B] [SW-846 8081A]	Endosulfan I
NPW, SCM	[SW-846 8081B] [SW-846 8081A]	Endosulfan II
NPW, SCM	[SW-846 8081B] [SW-846 8081A]	Endosulfan sulfate
NPW, SCM	[SW-846 8081B] [SW-846 8081A]	Endrin
NPW, SCM	[SW-846 8081B] [SW-846 8081A]	Endrin aldehyde
NPW, SCM	[SW-846 8081B] [SW-846 8081A]	Endrin ketone
NPW, SCM	[SW-846 8081B] [SW-846 8081A]	Etridiazole
NPW, SCM	[SW-846 8081B] [SW-846 8081A]	Heptachlor
NPW, SCM	[SW-846 8081B] [SW-846 8081A]	Heptachlor epoxide
NPW, SCM	[SW-846 8081B] [SW-846 8081A]	Hexachlorobenzene
NPW, SCM	[SW-846 8081B] [SW-846 8081A]	Hexachlorocyclopentadiene
NPW, SCM	[SW-846 8081B] [SW-846 8081A]	Methoxychlor
NPW, SCM	[SW-846 8081B] [SW-846 8081A]	Permethrin
NPW, SCM	[SW-846 8081B] [SW-846 8081A]	Propachlor
NPW, SCM	[SW-846 8081B] [SW-846 8081A]	Toxaphene
NPW, SCM	[SW-846 8081B] [SW-846 8081A]	Trifluralin
NPW, SCM	[SW-846 8082A] [SW-846 8082]	PCB 1016
NPW, SCM	[SW-846 8082A] [SW-846 8082]	PCB 1221
NPW, SCM	[SW-846 8082A] [SW-846 8082]	PCB 1232
NPW, SCM	[SW-846 8082A] [SW-846 8082]	PCB 1242
NPW, SCM	[SW-846 8082A] [SW-846 8082]	PCB 1248
NPW, SCM	[SW-846 8082A] [SW-846 8082]	PCB 1254
NPW, SCM	[SW-846 8082A] [SW-846 8082]	PCB 1260
NPW, SCM	[SW-846 8141B] [SW-846 8141A]	Azinphos methyl

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Matrix	Approved Method	Parameter Description
NPW, SCM	[SW-846 8141B] [SW-846 8141A]	Bolstar
NPW, SCM	[SW-846 8141B] [SW-846 8141A]	Chloropyrifos
NPW, SCM	[SW-846 8141B] [SW-846 8141A]	Coumaphos
NPW, SCM	[SW-846 8141B] [SW-846 8141A]	Demeton (o-)
NPW, SCM	[SW-846 8141B] [SW-846 8141A]	Demeton (s-)
NPW, SCM	[SW-846 8141B] [SW-846 8141A]	Diazinon
NPW, SCM	[SW-846 8141B] [SW-846 8141A]	Dichlorvos
NPW, SCM	[SW-846 8141B] [SW-846 8141A]	Dimethoate
NPW, SCM	[SW-846 8141B] [SW-846 8141A]	Disulfoton
NPW, SCM	[SW-846 8141B] [SW-846 8141A]	EPN
NPW, SCM	[SW-846 8141B] [SW-846 8141A]	Ethoprop
NPW, SCM	[SW-846 8141B] [SW-846 8141A]	Fensulfothion
NPW, SCM	[SW-846 8141B] [SW-846 8141A]	Fenthion
NPW, SCM	[SW-846 8141B] [SW-846 8141A]	Malathion
NPW, SCM	[SW-846 8141B] [SW-846 8141A]	Merphos
NPW, SCM	[SW-846 8141B] [SW-846 8141A]	Mevinphos
NPW, SCM	[SW-846 8141B] [SW-846 8141A]	Naled
NPW, SCM	[SW-846 8141B] [SW-846 8141A]	Parathion
NPW, SCM	[SW-846 8141B] [SW-846 8141A]	Parathion methyl
NPW, SCM	[SW-846 8141B] [SW-846 8141A]	Phorate
NPW, SCM	[SW-846 8141B] [SW-846 8141A]	Ronnel
NPW, SCM	[SW-846 8141B] [SW-846 8141A]	Stirofos
NPW, SCM	[SW-846 8141B] [SW-846 8141A]	Sulfotepp
NPW, SCM	[SW-846 8141B] [SW-846 8141A]	TEPP
NPW, SCM	[SW-846 8141B] [SW-846 8141A]	Tokuthion [Protothiofos]
NPW, SCM	[SW-846 8141B] [SW-846 8141A]	Trichloronate
NPW, SCM	[SW-846 8151A]	Dalapon
NPW, SCM	[SW-846 8151A]	Dicamba
NPW, SCM	[SW-846 8151A]	Dichlorprop
NPW, SCM	[SW-846 8151A]	Dinoseb
NPW, SCM	[SW-846 8151A]	D (2,4-)
NPW, SCM	[SW-846 8151A]	DB (2,4-)
NPW, SCM	[SW-846 8151A]	T (2,4,5-)
NPW, SCM	[SW-846 8151A]	TP (2,4,5-) (Silvex)
NPW, SCM	[SW-846 8151A]	MCPA
NPW, SCM	[SW-846 8151A]	MCPP
NPW, SCM	[SW-846 8310]	Acenaphthene
NPW, SCM	[SW-846 8310]	Acenaphthylene
NPW, SCM	[SW-846 8310]	Anthracene
NPW, SCM	[SW-846 8310]	Benzo(a)anthracene
NPW, SCM	[SW-846 8310]	Benzo(a)pyrene

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Matrix	Approved Method	Parameter Description
NPW, SCM	[SW-846 8310]	Benzo(b)fluoranthene
NPW, SCM	[SW-846 8310]	Benzo(ghi)perylene
NPW, SCM	[SW-846 8310]	Benzo(k)fluoranthene
NPW, SCM	[SW-846 8310]	Chrysene
NPW, SCM	[SW-846 8310]	Dibenzo(a,h)anthracene
NPW, SCM	[SW-846 8310]	Fluoranthene
NPW, SCM	[SW-846 8310]	Fluorene
NPW, SCM	[SW-846 8310]	Indeno(1,2,3-cd)pyrene
NPW, SCM	[SW-846 8310]	Naphthalene
NPW, SCM	[SW-846 8310]	Phenanthrene
NPW, SCM	[SW-846 8310]	Pyrene
NPW, SCM	[SW-846 8330A] [SW-846 8330]	HMX
NPW, SCM	[SW-846 8330A] [SW-846 8330]	RDX
NPW, SCM	[SW-846 8330A] [SW-846 8330]	Trinitrobenzene (1,3,5-)
NPW, SCM	[SW-846 8330A] [SW-846 8330]	Dinitrobenzene (1,3-)
NPW, SCM	[SW-846 8330A] [SW-846 8330]	PETN
NPW, SCM	[SW-846 8330A] [SW-846 8330]	Tetryl
NPW, SCM	[SW-846 8330A] [SW-846 8330]	Nitrobenzene
NPW, SCM	[SW-846 8330A] [SW-846 8330]	Trinitrotoluene (2,4,6-)
NPW, SCM	[SW-846 8330A] [SW-846 8330]	Dinitrotoluene (4-amino-2,6-)
NPW, SCM	[SW-846 8330A] [SW-846 8330]	Dinitrotoluene (2-amino-4,6-)
NPW, SCM	[SW-846 8330A] [SW-846 8330]	Dinitrotoluene (2,4-)
NPW, SCM	[SW-846 8330A] [SW-846 8330]	Dinitrotoluene (2,6-)
NPW, SCM	[SW-846 8330A] [SW-846 8330]	Nitrotoluene (2-)
NPW, SCM	[SW-846 8330A] [SW-846 8330]	Nitrotoluene (3-)
NPW, SCM	[SW-846 8330A] [SW-846 8330]	Nitrotoluene (4-)
NPW, SCM	[SW-846 8330] [SW-846 8330A]	Nitroglycerine
NPW, SCM	[SW-846 8260C] [SW-846 8260B] [USER DEFINED LUFT]	Benzene
NPW, SCM	[SW-846 8260C] [SW-846 8260B]	Bromobenzene
NPW, SCM	[SW-846 8260C] [SW-846 8260B]	Butyl benzene (n-)
NPW, SCM	[SW-846 8260C] [SW-846 8260B]	Sec-butylbenzene
NPW, SCM	[SW-846 8260C] [SW-846 8260B]	Tert-butylbenzene
NPW, SCM	[SW-846 8260C] [SW-846 8260B]	Chlorobenzene
NPW, SCM	[SW-846 8260C] [SW-846 8260B]	Chlorotoluene (2-)
NPW, SCM	[SW-846 8260C] [SW-846 8260B]	Chlorotoluene (4-)
NPW, SCM	[SW-846 8260C] [SW-846 8260B]	Dichlorobenzene (1,2-)
NPW, SCM	[SW-846 8260C] [SW-846 8260B]	Dichlorobenzene (1,3-)
NPW, SCM	[SW-846 8260C] [SW-846 8260B]	Dichlorobenzene (1,4-)
NPW, SCM	[SW-846 8260C] [SW-846 8260B] [USER DEFINED LUFT]	Ethylbenzene
NPW, SCM	[SW-846 8260C] [SW-846 8260B]	Isopropylbenzene

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Matrix	Approved Method	Parameter Description
NPW, SCM	[SW-846 8260C] [SW-846 8260B]	Propylbenzene (n-)
NPW, SCM	[SW-846 8260C] [SW-846 8260B] [USER DEFINED LUFT]	Toluene
NPW, SCM	[SW-846 8260C] [SW-846 8260B]	Isopropyltoluene (4-)
NPW, SCM	[SW-846 8260C] [SW-846 8260B]	Trichlorobenzene (1,2,3-)
NPW, SCM	[SW-846 8260C] [SW-846 8260B]	Trimethylbenzene (1,2,4-)
NPW, SCM	[SW-846 8260C] [SW-846 8260B]	Trimethylbenzene (1,3,5-)
NPW, SCM	[SW-846 8260C] [SW-846 8260B]	Trimethylbenzene (1,2,3-)
NPW, SCM	[SW-846 8260C] [SW-846 8260B] [USER DEFINED LUFT]	Xylenes (total)
NPW, SCM	[SW-846 8260C] [SW-846 8260B] [USER DEFINED LUFT]	Xylene (m-)
NPW, SCM	[SW-846 8260C] [SW-846 8260B] [USER DEFINED LUFT]	Xylene (o-)
NPW, SCM	[SW-846 8260C] [SW-846 8260B] [USER DEFINED LUFT]	Xylene (p-)
NPW, SCM	[SW-846 8260C] [SW-846 8260B]	tert-Amylmethyl ether [TAME]
NPW, SCM	[SW-846 8260C] [SW-846 8260B]	Allyl chloride
NPW, SCM	[SW-846 8260C] [SW-846 8260B]	Bromochloromethane
NPW, SCM	[SW-846 8260C] [SW-846 8260B]	Bromodichloromethane
NPW, SCM	[SW-846 8260C] [SW-846 8260B]	Bromoethane
NPW, SCM	[SW-846 8260C] [SW-846 8260B]	Bromoform
NPW, SCM	[SW-846 8260C] [SW-846 8260B]	Bromomethane
NPW, SCM	[SW-846 8260C] [SW-846 8260B]	Cyclohexane
NPW, SCM	[SW-846 8260C] [SW-846 8260B]	Cyclohexanone
NPW, SCM	[SW-846 8260C] [SW-846 8260B]	Butadiene (2-chloro-1,3-)
NPW, SCM	[SW-846 8260C] [SW-846 8260B]	Dichloro-2-butene (cis-1,4-)
NPW, SCM	[SW-846 8260C] [SW-846 8260B]	Carbon tetrachloride
NPW, SCM	[SW-846 8260C] [SW-846 8260B]	Chloroethane
NPW, SCM	[SW-846 8260C] [SW-846 8260B]	Chloroethyl vinyl ether (2-)
NPW, SCM	[SW-846 8260C] [SW-846 8260B]	Chloroform
NPW, SCM	[SW-846 8260C] [SW-846 8260B]	Chloromethane
NPW, SCM	[SW-846 8260C] [SW-846 8260B]	Diethyl ether (Ethyl ether)
NPW, SCM	[SW-846 8260C] [SW-846 8260B]	Dichloropropene (trans-1,3-)
NPW, SCM	[SW-846 8260C] [SW-846 8260B]	Dibromochloromethane
NPW, SCM	[SW-846 8260C] [SW-846 8260B]	Dibromoethane (1,2-) (EDB)
NPW, SCM	[SW-846 8260C] [SW-846 8260B]	Dibromomethane
NPW, SCM	[SW-846 8260C] [SW-846 8260B]	Dibromo-3-chloropropane (1,2-)
NPW, SCM	[SW-846 8260C] [SW-846 8260B]	Dichlorodifluoromethane
NPW, SCM	[SW-846 8260C] [SW-846 8260B]	Dichloroethane (1,1-)
NPW, SCM	[SW-846 8260C] [SW-846 8260B]	Dichloroethane (1,2-)
NPW, SCM	[SW-846 8260C] [SW-846 8260B]	Dichloroethene (1,1-)

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Matrix	Approved Method	Parameter Description
NPW, SCM	[SW-846 8260C] [SW-846 8260B]	Dichloroethene (trans-1,2-)
NPW, SCM	[SW-846 8260C] [SW-846 8260B]	Dichloroethene (cis-1,2-)
NPW, SCM	[SW-846 8260C] [SW-846 8260B]	Dichloropropane (1,2-)
NPW, SCM	[SW-846 8260C] [SW-846 8260B]	Dichloropropane (1,3-)
NPW, SCM	[SW-846 8260C] [SW-846 8260B]	Dichloropropane (2,2-)
NPW, SCM	[SW-846 8260C] [SW-846 8260B]	Dichloropropene (1,1-)
NPW, SCM	[SW-846 8260C] [SW-846 8260B]	Dichloropropene (cis-1,3-)
NPW, SCM	[SW-846 8260C] [SW-846 8260B]	Dichloro-2-butene (trans-1,4-)
NPW, SCM	[SW-846 8260C] [SW-846 8260B]	Diisopropyl Ether [DIPE]
NPW, SCM	[SW-846 8260C] [SW-846 8260B]	Butanol (1-)
NPW, SCM	[SW-846 8260C] [SW-846 8260B]	Ethanol
NPW, SCM	[SW-846 8260C] [SW-846 8260B]	Methylene chloride (Dichloromethane)
NPW, SCM	[SW-846 8260C] [SW-846 8260B]	Nitropropane (2-)
NPW, SCM	[SW-846 8260C] [SW-846 8260B]	Tetrachloroethane (1,1,2,2-)
NPW, SCM	[SW-846 8260C] [SW-846 8260B]	Tetrachloroethene
NPW, SCM	[SW-846 8260C] [SW-846 8260B]	Tetrahydrofuran
NPW, SCM	[SW-846 8260C] [SW-846 8260B]	Trichloroethane (1,1,1-)
NPW, SCM	[SW-846 8260C] [SW-846 8260B]	Trichloroethane (1,1,2-)
NPW, SCM	[SW-846 8260C] [SW-846 8260B]	Trichloroethene
NPW, SCM	[SW-846 8260C] [SW-846 8260B]	Trichlorofluoromethane
NPW, SCM	[SW-846 8260C] [SW-846 8260B]	Trichloro (1,1,2-) trifluoroethane (1,2,2-)
NPW, SCM	[SW-846 8260C] [SW-846 8260B]	Trichloropropane (1,2,3-)
NPW, SCM	[SW-846 8260C] [SW-846 8260B]	Vinyl acetate
NPW, SCM	[SW-846 8260C] [SW-846 8260B]	Vinyl chloride
NPW, SCM	[SW-846 8260C] [SW-846 8260B]	Acetone
NPW, SCM	[SW-846 8260C] [SW-846 8260B]	Carbon disulfide
NPW, SCM	[USER DEFINED SW846 8260B]	Butanol (3,3-Dimethyl-1-)
NPW, SCM	[SW-846 8260C] [SW-846 8260B]	Butanone (2-)
NPW, SCM	[SW-846 8260C] [SW-846 8260B]	Butyl formate (t-)
NPW, SCM	[SW-846 8260C] [SW-846 8260B]	Ethyl-tert-butyl Ether [ETBE]
NPW, SCM	[SW-846 8260C] [SW-846 8260B]	Ethyl acetate
NPW, SCM	[SW-846 8260C] [SW-846 8260B]	Ethyl methacrylate
NPW, SCM	[SW-846 8260C] [SW-846 8260B]	Hexanone (2-)
NPW, SCM	[SW-846 8260C] [SW-846 8260B]	Methacrylonitrile
NPW, SCM	[SW-846 8260C] [SW-846 8260B]	Methyl acrylate
NPW, SCM	[SW-846 8260C] [SW-846 8260B]	Methyl methacrylate
NPW, SCM	[SW-846 8260C] [SW-846 8260B]	Methyl acetate
NPW, SCM	[SW-846 8260C] [SW-846 8260B]	Methyl iodide
NPW, SCM	[SW-846 8260C] [SW-846 8260B]	Iso-butyl alcohol
NPW, SCM	[SW-846 8260C] [SW-846 8260B]	Isopropanol
NPW, SCM	[SW-846 8260C] [SW-846 8260B]	N-Nitroso-di-n-butylamine

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Matrix	Approved Method	Parameter Description
NPW, SCM	[SW-846 8260C] [SW-846 8260B]	Pentachloroethane
NPW, SCM	[SW-846 8260C] [SW-846 8260B]	Pentanone (4-methyl-2-)
NPW, SCM	[SW-846 8260C] [SW-846 8260B]	Pentanol (2-Methyl-2-)
NPW, SCM	[SW-846 8260C] [SW-846 8260B]	Propionitrile
NPW, SCM	[SW-846 8260C] [SW-846 8260B] [USER DEFINED LUFT]	Methyl tert-butyl ether
NPW, SCM	[SW-846 8260C] [SW-846 8260B]	Amyl alcohol (t-)
NPW, SCM	[SW-846 8260C] [SW-846 8260B]	Tert-butyl alcohol
NPW, SCM	[SW-846 8260C] [SW-846 8260B]	Acetonitrile
NPW, SCM	[SW-846 8260C] [SW-846 8260B]	Acrolein
NPW, SCM	[SW-846 8260C] [SW-846 8260B]	Acrylonitrile
NPW, SCM	[SW-846 8260C] [SW-846 8260B]	Hexachlorobutadiene (1,3-)
NPW, SCM	[SW-846 8260C] [SW-846 8260B]	Hexachloroethane
NPW, SCM	[SW-846 8260C] [SW-846 8260B]	Methylcyclohexane
NPW, SCM	[SW-846 8260C] [SW-846 8260B]	Naphthalene
NPW, SCM	[SW-846 8260C] [SW-846 8260B]	Octane (-n)
NPW, SCM	[SW-846 8260C] [SW-846 8260B]	Styrene
NPW, SCM	[SW-846 8260C] [SW-846 8260B]	Tetrachloroethane (1,1,1,2-)
NPW, SCM	[SW-846 8260C] [SW-846 8260B]	Trichlorobenzene (1,2,4-)
NPW, SCM	[SW-846 8260B]	Trimethylpentane (2,2,4-)
NPW, SCM	[SW-846 8260C] [SW-846 8260B]	Dioxane (1,4-)
NPW, SCM	[SW-846 8270D] [SW-846 8270C]	Acetophenone
NPW, SCM	[SW-846 8270D] [SW-846 8270C]	Acetylaminofluorene (2-)
NPW, SCM	[SW-846 8270D] [SW-846 8270C]	Aminobiphenyl (4-)
NPW, SCM	[SW-846 8270D] [SW-846 8270C]	Aramite
NPW, SCM	[SW-846 8270D] [SW-846 8270C]	Benzal chloride
NPW, SCM	[SW-846 8270D] [SW-846 8270C]	Benzo(j)fluoranthene
NPW, SCM	[SW-846 8270D] [SW-846 8270C]	Benzotrichloride
NPW, SCM	[SW-846 8270D] [SW-846 8270C]	Benzyl chloride
NPW, SCM	[SW-846 8270D] [SW-846 8270C]	Chlorobenzilate
NPW, SCM	[SW-846 8270D] [SW-846 8270C]	Chloronaphthalene (1-)
NPW, SCM	[SW-846 8270D] [SW-846 8270C]	Diallate (cis)
NPW, SCM	[SW-846 8270D] [SW-846 8270C]	Diallate (trans)
NPW, SCM	[SW-846 8270D] [SW-846 8270C]	Dibenzo(a,e)pyrene
NPW, SCM	[SW-846 8270D] [SW-846 8270C]	Dibenz(a,h)acridine
NPW, SCM	[SW-846 8270D] [SW-846 8270C]	Dibenzo(a,h)pyrene
NPW, SCM	[SW-846 8270D] [SW-846 8270C]	Dibenz(a,j)acridine
NPW, SCM	[SW-846 8270D] [SW-846 8270C]	Dibenzo(a,i)pyrene
NPW, SCM	[SW-846 8270D] [SW-846 8270C]	Dibenzo(c,g)carbazole (7H-)
NPW, SCM	[SW-846 8270D] [SW-846 8270C]	Dichlorophenol (2,6-)
NPW, SCM	[SW-846 8270D] [SW-846 8270C]	Dimethoate
NPW, SCM	[SW-846 8270D] [SW-846 8270C]	Dimethylaminoazobenzene

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Matrix	Approved Method	Parameter Description
NPW, SCM	[SW-846 8270D] [SW-846 8270C]	Dimethylbenz(a)anthracene (7,12-)
NPW, SCM	[SW-846 8270D] [SW-846 8270C]	Dimethyl benzidine (3,3-)
NPW, SCM	[SW-846 8270D] [SW-846 8270C]	Dinitrobenzene (1,3-)
NPW, SCM	[SW-846 8270D] [SW-846 8270C]	Dinoseb
NPW, SCM	[SW-846 8270D] [SW-846 8270C]	Disulfoton
NPW, SCM	[SW-846 8270D] [SW-846 8270C]	Famphur
NPW, SCM	[SW-846 8270D] [SW-846 8270C]	Hexachlorophene
NPW, SCM	[SW-846 8270D] [SW-846 8270C]	Isodrin
NPW, SCM	[SW-846 8270D] [SW-846 8270C]	Isosafrole (cis-)
NPW, SCM	[SW-846 8270D] [SW-846 8270C]	Isosafrole (trans-)
NPW, SCM	[SW-846 8270D] [SW-846 8270C]	Kepone
NPW, SCM	[SW-846 8270D] [SW-846 8270C]	Methanesulfonate (Ethyl-)
NPW, SCM	[SW-846 8270D] [SW-846 8270C]	Methanesulfonate (Methyl-)
NPW, SCM	[SW-846 8270D] [SW-846 8270C]	Methapyrilene
NPW, SCM	[SW-846 8270D] [SW-846 8270C]	Methylcholanthrene (3-)
NPW, SCM	[SW-846 8270D] [SW-846 8270C]	Napthoquinone (1,4-)
NPW, SCM	[SW-846 8270D] [SW-846 8270C]	Napththylamine (1-)
NPW, SCM	[SW-846 8270D] [SW-846 8270C]	Napththylamine (2-)
NPW, SCM	[SW-846 8270C]	Nitrodiphenylamine (2-)
NPW, SCM	[SW-846 8270D] [SW-846 8270C]	N-Nitroso-di-n-butylamine
NPW, SCM	[SW-846 8270D] [SW-846 8270C]	N-Nitrosomorpholine
NPW, SCM	[SW-846 8270D] [SW-846 8270C]	N-Nitrosopiperidine
NPW, SCM	[SW-846 8270D] [SW-846 8270C]	Parathion
NPW, SCM	[SW-846 8270D] [SW-846 8270C]	Parathion methyl
NPW, SCM	[SW-846 8270D] [SW-846 8270C]	Pentachlorobenzene
NPW, SCM	[SW-846 8270D] [SW-846 8270C]	Pentachloroethane
NPW, SCM	[SW-846 8270D] [SW-846 8270C]	Pentachloronitrobenzene
NPW, SCM	[SW-846 8270D] [SW-846 8270C]	Phenacetin
NPW, SCM	[SW-846 8270D] [SW-846 8270C]	Phenylenediamine (1,4-)
NPW, SCM	[SW-846 8270D] [SW-846 8270C]	Phenylethylamine (alpha, alpha-Dimethyl)
NPW, SCM	[SW-846 8270D] [SW-846 8270C]	Phorate
NPW, SCM	[SW-846 8270D] [SW-846 8270C]	Phosphorothioate (O,O,O-triethyl)
NPW, SCM	[SW-846 8270D] [SW-846 8270C]	Phosphorothioate (O,O-diethyl-O-2-pyrazinyl) [Thionazin]
NPW, SCM	[SW-846 8270D] [SW-846 8270C]	Picoline (2-)
NPW, SCM	[SW-846 8270D] [SW-846 8270C]	Pronamide
NPW, SCM	[SW-846 8270D] [SW-846 8270C]	Quinoline -1-Oxide (4-Nitro)
NPW, SCM	[SW-846 8270D] [SW-846 8270C]	Safrole
NPW, SCM	[SW-846 8270D] [SW-846 8270C]	Sulfotepp
NPW, SCM	[SW-846 8270D] [SW-846 8270C]	Tetrachlorobenzene (1,2,3,4-)
NPW, SCM	[SW-846 8270D] [SW-846 8270C]	Tetrachlorobenzene (1,2,3,5-)

The information listed is subject to change.
Always check with the laboratory for the most updated information.

Matrix	Approved Method	Parameter Description
NPW, SCM	[SW-846 8270D] [SW-846 8270C]	Tetrachlorobenzene (1,2,4,5-)
NPW, SCM	[SW-846 8270D] [SW-846 8270C]	Tetrachlorophenol (2,3,4,6-)
NPW, SCM	[SW-846 8270D] [SW-846 8270C]	Toluidine (2-) (2-Methylaniline)
NPW, SCM	[SW-846 8270D] [SW-846 8270C]	Toluidine (5-Nitro-2-)
NPW, SCM	[SW-846 8270D] [SW-846 8270C]	Trinitrobenzene (1,3,5-)
NPW, SCM	[SW-846 8270D] [SW-846 8270C]	N-Nitrosodiethylamine
NPW, SCM	[SW-846 8270D] [SW-846 8270C]	N-Nitrosodimethylamine
NPW, SCM	[SW-846 8270D] [SW-846 8270C]	N-Nitroso-di-n-propylamine
NPW, SCM	[SW-846 8270D] [SW-846 8270C]	N-Nitrosodiphenylamine
NPW, SCM	[SW-846 8270D] [SW-846 8270C]	N-Nitrosomethylethylamine
NPW, SCM	[SW-846 8270D] [SW-846 8270C]	N-Nitrosopyrrolidine
NPW, SCM	[SW-846 8270D] [SW-846 8270C]	Diphenylamine
NPW, SCM	[SW-846 8270D] [SW-846 8270C]	Carbazole
NPW, SCM	[SW-846 8270D] [SW-846 8270C]	Benzidine
NPW, SCM	[SW-846 8270D] [SW-846 8270C]	Dichlorobenzidine (3,3'-)
NPW, SCM	[SW-846 8270D] [SW-846 8270C]	Diphenylhydrazine (1,2-)
NPW, SCM	[SW-846 8270D] [SW-846 8270C]	Aniline
NPW, SCM	[SW-846 8270D] [SW-846 8270C]	Chloraniline (4-)
NPW, SCM	[SW-846 8270D] [SW-846 8270C]	Nitroaniline (2-)
NPW, SCM	[SW-846 8270D] [SW-846 8270C]	Nitroaniline (3-)
NPW, SCM	[SW-846 8270D] [SW-846 8270C]	Nitroaniline (4-)
NPW, SCM	[SW-846 8270D] [SW-846 8270C]	Chloronaphthalene (2-)
NPW, SCM	[SW-846 8270D] [SW-846 8270C]	Hexachlorobenzene
NPW, SCM	[SW-846 8270D] [SW-846 8270C]	Hexachlorobutadiene (1,3-)
NPW, SCM	[SW-846 8270D] [SW-846 8270C]	Hexachlorocyclopentadiene
NPW, SCM	[SW-846 8270D] [SW-846 8270C]	Hexachloroethane
NPW, SCM	[SW-846 8270D] [SW-846 8270C]	Hexachloropropene
NPW, SCM	[SW-846 8270D] [SW-846 8270C]	Trichlorobenzene (1,2,4-)
NPW, SCM	[SW-846 8270D] [SW-846 8270C]	Bis (2-chloroethoxy) methane
NPW, SCM	[SW-846 8270D] [SW-846 8270C]	Bis (2-chloroethyl) ether
NPW, SCM	[SW-846 8270D] [SW-846 8270C]	Bis (2-chloroisopropyl) ether
NPW, SCM	[SW-846 8270D] [SW-846 8270C]	Chlorophenyl-phenyl ether (4-)
NPW, SCM	[SW-846 8270D] [SW-846 8270C]	Bromophenyl-phenyl ether (4-)
NPW, SCM	[SW-846 8270D] [SW-846 8270C]	Dinitrotoluene (2,4-)
NPW, SCM	[SW-846 8270D] [SW-846 8270C]	Dinitrotoluene (2,6-)
NPW, SCM	[SW-846 8270D] [SW-846 8270C]	Isophorone
NPW, SCM	[SW-846 8270D] [SW-846 8270C]	Nitrobenzene
NPW, SCM	[SW-846 8270D] [SW-846 8270C]	Butyl benzyl phthalate
NPW, SCM	[SW-846 8270D] [SW-846 8270C]	Bis (2-ethylhexyl) phthalate
NPW, SCM	[SW-846 8270D] [SW-846 8270C]	Diethyl phthalate
NPW, SCM	[SW-846 8270D] [SW-846 8270C]	Dimethyl phthalate

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Matrix	Approved Method	Parameter Description
NPW, SCM	[SW-846 8270D] [SW-846 8270C]	Di-n-butyl phthalate
NPW, SCM	[SW-846 8270D] [SW-846 8270C]	Di-n-octyl phthalate
NPW, SCM	[SW-846 8270D] [SW-846 8270C]	Acenaphthene
NPW, SCM	[SW-846 8270D] [SW-846 8270C]	Anthracene
NPW, SCM	[SW-846 8270D] [SW-846 8270C]	Acenaphthylene
NPW, SCM	[SW-846 8270D] [SW-846 8270C]	Benzo(a)anthracene
NPW, SCM	[SW-846 8270D] [SW-846 8270C]	Benzo(a)pyrene
NPW, SCM	[SW-846 8270D] [SW-846 8270C]	Benzo(b)fluoranthene
NPW, SCM	[SW-846 8270D] [SW-846 8270C]	Benzo(ghi)perylene
NPW, SCM	[SW-846 8270D] [SW-846 8270C]	Benzo(k)fluoranthene
NPW, SCM	[SW-846 8270D] [SW-846 8270C]	Chrysene
NPW, SCM	[SW-846 8270D] [SW-846 8270C]	Dibenzo(a,h)anthracene
NPW, SCM	[SW-846 8270D] [SW-846 8270C]	Fluoranthene
NPW, SCM	[SW-846 8270D] [SW-846 8270C]	Fluorene
NPW, SCM	[SW-846 8270D] [SW-846 8270C]	Indeno(1,2,3-cd)pyrene
NPW, SCM	[SW-846 8270D] [SW-846 8270C]	Methylnaphthalene (2-)
NPW, SCM	[SW-846 8270D] [SW-846 8270C]	Naphthalene
NPW, SCM	[SW-846 8270D] [SW-846 8270C]	Phenanthrene
NPW, SCM	[SW-846 8270D] [SW-846 8270C]	Pyrene
NPW, SCM	[SW-846 8270D] [SW-846 8270C]	Methyl phenol (4-chloro-3-)
NPW, SCM	[SW-846 8270D] [SW-846 8270C]	Chlorophenol (2-)
NPW, SCM	[SW-846 8270D] [SW-846 8270C]	Dichlorophenol (2,4-)
NPW, SCM	[SW-846 8270D] [SW-846 8270C]	Dimethylphenol (2,4-)
NPW, SCM	[SW-846 8270D] [SW-846 8270C]	Dinitrophenol (2,4-)
NPW, SCM	[SW-846 8270D] [SW-846 8270C]	Dinitrophenol (2-methyl-4,6-)
NPW, SCM	[SW-846 8270D] [SW-846 8270C]	Methylphenol (2-)
NPW, SCM	[SW-846 8270D] [SW-846 8270C]	Methylphenol (4-)
NPW, SCM	[SW-846 8270D] [SW-846 8270C]	Nitrophenol (2-)
NPW, SCM	[SW-846 8270D] [SW-846 8270C]	Nitrophenol (4-)
NPW, SCM	[SW-846 8270D] [SW-846 8270C]	Pentachlorophenol
NPW, SCM	[SW-846 8270D] [SW-846 8270C]	Phenol
NPW, SCM	[SW-846 8270D] [SW-846 8270C]	Trichlorophenol (2,4,5-)
NPW, SCM	[SW-846 8270D] [SW-846 8270C]	Trichlorophenol (2,4,6-)
NPW, SCM	[SW-846 8270D] [SW-846 8270C]	Dibenzofuran
NPW, SCM	[SW-846 8270D] [SW-846 8270C]	Dichlorobenzene (1,2-)
NPW, SCM	[SW-846 8270D] [SW-846 8270C]	Dichlorobenzene (1,3-)
NPW, SCM	[SW-846 8270D] [SW-846 8270C]	Dichlorobenzene (1,4-)
NPW, SCM	[SW-846 8270D] [SW-846 8270C]	Benzoic acid
NPW, SCM	[SW-846 8270D] [SW-846 8270C]	Benzyl alcohol
NPW, SCM	[SW-846 8270D]	Decane (n-)
NPW, SCM	[SW-846 8270D]	Octadecane (n-)

The information listed is subject to change.
Always check with the laboratory for the most updated information.

Matrix	Approved Method	Parameter Description
NPW, SCM	[USER DEFINED CA LUFT - diesel]	Petroleum Organics
NPW, SCM	[SW-846 8270D] [SW-846 8270C]	Pyridine
NPW, SCM	[SW-846 8270D] [SW-846 8270C]	Benzo(a)anthracene
NPW, SCM	[SW-846 8270D] [SW-846 8270C]	Benzo(a)pyrene
NPW, SCM	[SW-846 8270D] [SW-846 8270C]	Benzo(b)fluoranthene
NPW, SCM	[SW-846 8270D] [SW-846 8270C]	Benzo(k)fluoranthene
NPW, SCM	[SW-846 8270D] [SW-846 8270C]	Dibenzo(a,h)anthracene
NPW, SCM	[SW-846 8270D] [SW-846 8270C]	Indeno(1,2,3-cd)pyrene
NPW, SCM	[SW-846 9010C] [USER DEFINED 9010B]	Cyanide
NPW, SCM	[SW-846 9010C] [USER DEFINED 9010B]	Cyanide - amenable to Cl2
NPW, SCM	[SW-846 9012B] [USER DEFINED 9012A]	Cyanide
NPW, SCM	[SW-846 9030B]	Sulfides, acid sol. & insol.
NPW, SCM	[SW-846 9034]	Sulfides, acid sol. & insol.
NPW, SCM	[SW-846 9056] [SW-846 9056A]	Sulfate
NPW, SCM	[SW-846 9040C]	pH - waste, >20% water
NPW, SCM	[SW-846 9060A] [SW-846 9060]	Total organic carbon (TOC)
NPW, SCM	[SW-846 9056] [SW-846 9056A]	Nitrite
NPW, SCM	[SW-846 9056] [SW-846 9056A]	Nitrate
NPW, SCM	[SW-846 9056] [SW-846 9056A]	Bromide
NPW, SCM	[SW-846 9056] [SW-846 9056A]	Chloride
NPW, SCM	[SW-846 9056] [SW-846 9056A]	Fluoride
NPW, SCM	[EPA 300.0]	Guanidine nitrate
NPW, SCM	[SW-846 8330]	Guanidine nitrate
NPW, SCM	[SM 2540 G]	Total, fixed, and volatile solids (SQAR)
SCM	[SW-846 1030]	Ignitability of solids
SCM	[SW-846 3031]	Metals
SCM	[SW-846 3040A]	Metals
SCM	[SW-846 3050B]	Metals
SCM	[SW-846 3051A] [SW-846 3051]	Metals
SCM	[SW-846 3052]	Metals
SCM	[SW-846 3060A]	Metals
SCM	[SW-846 7471B] [SW-846 7471A]	Mercury - solid waste
SCM	[SW-846 3540C]	Semivolatile organics
SCM	[SW-846 3550C] [SW-846 3550B]	Semivolatile organics
SCM	[SW-846 3546]	Semivolatile organics
SCM	[SW-846 3580A]	Organics
SCM	[SW-846 3585]	Organics
SCM	[SW-846 5035A] [SW-846 5035L]	Volatile organics - low conc.
SCM	[SW-846 5035A] [SW-846 5035H]	Volatile organics - high conc.

The information listed is subject to change.
Always check with the laboratory for the most updated information.

Matrix	Approved Method	Parameter Description
SCM	[SW-846 3610B]	Semivolatile organics
SCM	[SW-846 3611B]	Semivolatile organics
SCM	[SW-846 3620C] [SW-846 3620B]	Semivolatile organics
SCM	[SW-846 3630C]	Semivolatile organics
SCM	[SW-846 3660B]	Semivolatile organics
SCM	[SW-846 3665A]	Semivolatile organics
SCM	[SW-846 8440]	Total rec. petroleum hydrocarbons
SCM	[SW-846 9013] [USER DEFINED 9013A]	Cyanide
SCM	[SW-846 9023]	Extractable organic halides (EOX)
SCM	[SW-846 9045D] [SW-846 9045C]	pH - soil and waste
SCM	[SW-846 9071 B]	Oil & grease - sludge-hem
SCM	[SW-846 9071 B]	Oil & grease - sludge-hem-npm
SCM	[ASTM D5468 and D482]	% ash
SCM	[ASTM D240]	Heat of combustion (BTU)
SCM	[SW-846 9095] [USER DEFINED 9095A]	Free liquid
SCM	[SW-846 9056] [SW-846 9056A]	Orthophosphate

3.4 ABBREVIATIONS/ACRONYMS

The quality department is responsible for setting up and maintaining a list of abbreviations used in the quality manual.

ABBREVIATION	***DESCRIPTION***
A2LA	*AMERICAN ASSOCIATION FOR LABORATORY ACCREDITATION*
AIHA	*AMERICAN INDUSTRIAL HYGIENE ASSOCIATION*
BLANK	*See FIELD, TRIP, METHOD, EQUIPMENT*
CAL	*CALIBRATION*
CCB	*CONTINUING CALIBRATION BLANK*
CCV	*CONTINUING CALIBRATION VERIFICATION*
CDOC	*CONTINUING DEMONSTRATION OF CAPABILITY*
COC	*CHAIN OF CUSTODY*
CA	*CORRECTIVE ACTION*
DQO	*DATA QUALITY OBJECTIVES*
DUP	*DUPLICATE*
EB	*EQUIPMENT BLANK*
FB	*FIELD BLANK*
GC	*GAS CHROMATOGRAPHY*
GCMS	*GAS CHROMATOGRAPHY MASS SPECTROMETRY*
HPLC	*HIGH PRESSURE LIQUID CHROMATOGRAPHY*
IC	*ION CHROMATOGRAPHY*
ICP	*INDUCTIVELY COUPLED PLASMA*
ICPMS	*INDUCTIVELY COUPLED PLASMA MASS SPECTROMETRY*
ICS	*INTERFERENCE CHECK SAMPLE*
ICV – See SSCV	*INITIAL CALIBRATION VERIFICATION*
IDOC	*INITIAL DEMONSTRATION OF CAPABILITY (SEE ALSO CDOC)*
IDL	*INSTRUMENT DETECTION LIMIT*
IS	*INTERNAL STANDARD*
LCS	*LABORATORY CONTROL SAMPLE (Typically 2ND Source)*
LOD	*LIMIT OF DETECTION*
LDR	*LINEAR DYNAMIC RANGE*
MAT	*MATRIX*
MS	*MATRIX SPIKE*
MSD	*MATRIX SPIKE DUPLICATE*
MDL	*METHOD DETECTION LIMIT*
MB	*METHOD BLANK*
NC	*NEGATIVE CONTROL*
NELAP	*NATIONAL ENVIRONMENTAL LABORATORY ACCREDITATION*
% Rec	*PERCENT RECOVERY*

ABBREVIATION	***DESCRIPTION***
PC	*POSITIVE CONTROL*
PDL	*PRACTICAL DETECTION LIMIT*
PQL	*PRACTICAL QUANTITATION LIMIT also See Reporting Limit (RL)*
PT	*PROFICIENCY TEST SAMPLE*
QUAL	*QUALIFIER*
QA	*QUALITY ASSURANCE*
QAM	*QUALITY ASSURANCE MANUAL*
QAO	*QUALITY ASSURANCE OFFICER*
QC	*QUALITY CONTROL*
RL	*REPORTING LIMIT*
RPD	*RELATIVE PERCENT DIFFERENCE*
RF	*RESPONSE FACTOR*
SSCV	*SECONDARY SOURCE CALIBRATION VERIFICAION*
SOP	*STANDARD OPERATING PROCEDURE*
SRM	*STANDARD REFERENCE MATERIAL*
SURR	*SURROGATE*
UV	*ULTRAVIOLET*
VOC	*VOLATILE ORGANIC COMPOUND*

4.0 MANAGEMENT REQUIREMENTS

4.1 ORGANIZATION

4.1.1 Legal identity

The laboratory is authorized under Title 62 of the Tennessee Code Annotated and is identified as Environmental Science Corporation (d.b.a. ESC Lab Sciences) located at 12065 Lebanon Road, Mount Juliet, TN 37122

4.1.2 Organization

The laboratory is a public entity and is structured to provide environmental support services in compliance with numerous federal, state, and local regulations as well as to meet the analytical needs of the client.

4.1.3 Facilities Under Management System

The scope of the ESC management system is comprehensive and covers all technical and supporting work conducted at all facilities at the primary Lebanon Road location as well as customer support and shipping operations across the US.

4.1.4 Independence

ESC Lab Sciences is an independent analytical facility and therefore remains uninfluenced by external factors, such as financial or political considerations.

4.1.5 Management Responsibilities and Policies

The assignment of responsibilities, authorities, and interrelationships of the personnel who manage, perform, or verify work affecting analytical quality is documented in the job descriptions maintain by the Human Resources department. Management bears specific responsibility for maintenance of the Quality System. This includes defining roles and responsibilities of personnel, approving documents, providing required training, providing a procedure for confidential reporting of data and ensuring data integrity, along with periodically reviewing data, procedures, and documentation. Management ensures that audit findings and corrective actions are completed within required time frames. Alternates are appointed by management during the absence of the Laboratory Manager, Technical Director or the Quality Manager. The organizational structure indicated in this section is designed to minimize the potential for conflicting or undue stresses that might influence the technical judgment of analytical personnel. Additionally, it provides adequate management for consistent supervision of laboratory practices and procedures.

Operations Management is responsible for defining the minimal level of education, qualifications, experience, and skills necessary for all analytical positions in the laboratory and assuring that technical staff has demonstrated capabilities in their tasks. Training is kept up-to-date by periodic review of training records and through employee performance reviews.

4.1.5.1 ***Chief Executive Officer***

Peter Schulert, Bachelor of Science in Chemistry, is the laboratory's Chief Executive Officer (CEO). He joined ESC in 1987 after the completion of his service with the United States Naval Submarine Service. In his five years of nuclear submarine experience in the Navy, Mr. Schulert qualified as an officer. This qualification included supervision of nuclear reactors and power plant operations. His vision for automation and client services has been a key component of ESC's rise to the top ranks of the industry. Mr. Schulert is responsible for developing and executing ESC's strategic plan. Under his leadership, ESC has become a large single location laboratory, with a comprehensive national certification program and industry leading data management tools. In his absence, all operational responsibilities are delegated to the Chief Financial Officer, Laboratory Director, Director of Technical & Regulatory Affairs, and the Chief Information Officer.

4.1.5.2 ***Director of Technical & Regulatory Affairs***

Judith R. Morgan, Master of Science in Analytical Chemistry and Registered Environmental Manager, is the Laboratory Director of Technical & Regulatory Affairs and serves as the laboratory Quality Assurance Officer (QAO). She has been serving the environmental industry since 1986 and is a respected expert witness. The majority of her experience is specific to quality and regulatory matters; however, she does have previous experience as an analyst in both organic and inorganic methods. In matters of laboratory QA/QC, she reports directly to Peter Schulert, CEO, thus making her QAO functions separate from laboratory operations. Her primary responsibility is the oversight of administrative and technical operations of the laboratory. She specifies and/or approves all methodologies used in the laboratory and ensures continued accreditation of the laboratory. She is responsible for maintaining the laboratory QA manual, initiating and overseeing audits, activating corrective measures (when necessary), implementing numerous international quality standards and preparing internal QA/QC reports. Additionally, she oversees the Technical Specialist group, which includes personnel who are considered to be experts in one or more facets of the laboratory. The Technical group maintains specific regulatory information that impacts quality, client relations, and strategic marketing. Dixie Marlin assumes responsibility for all QA functions, in the absence of the director.

4.1.5.3 ***Laboratory Director***

Eric Johnson, B.S. in Chemistry, is the Laboratory Director and is responsible for the supervision of each laboratory division and the overall compliance of the laboratory to this Quality Manual. Mr. Johnson provides ESC with necessary experience for all aspects of sample handling from sample shipping and receiving through sample disposal. He has been involved in many aspects of environmental analyses since 1991. He coordinates all production areas and is responsible for operational scheduling, process specifications, and implementation of quality standards. He focuses his background and experience on the improvement of existing systems in order to maximize efficiency and improve quality. He reports directly to the CEO. In his absence, all operations responsibilities are delegated to Tom Mellette and then to individual department managers.

4.1.5.4 ***Quality Control Manager***

Dixie Marlin, B.S. in Biology, is the laboratory Quality Control Manager. She has more than 20 years of combined laboratory experience in research, regulatory, and production lab environments. This experience has spanned the environmental lab in both privately owned, university facilities, and Federal Superfund sectors, with additional experience gained in state regulatory agencies. Her primary function is to assist production chemists/technicians regarding quality assurance/control measures, ensure compliance with method requirements and procedures, and perform audits of internal laboratory functions. Where necessary, she identifies, develops, and implements improvement of the laboratory measurement capability to meet the requirements of governing authorities, department programs, and laboratory clients. She is responsible for the supervision of the laboratory QC group and technical specialists. Judith Morgan assumes responsibility for these functions in her absence.

4.1.5.5 ***Chief Information Officer***

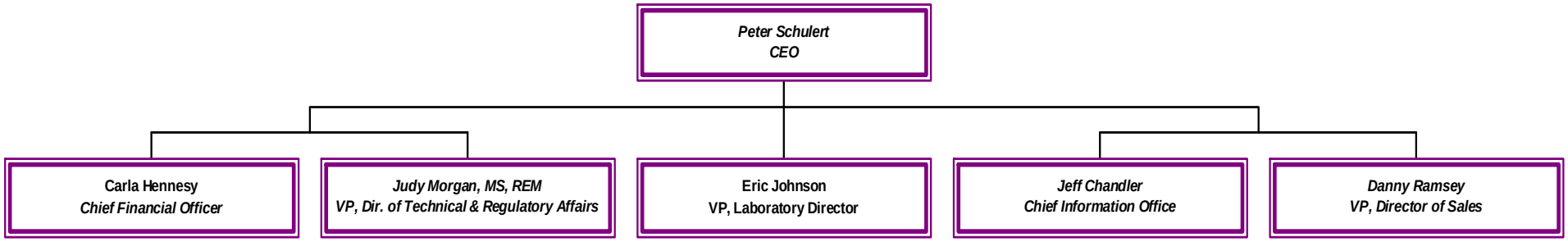
Jeff Chandler, B.S. in Computer Science, is the ESC CIO. His responsibilities include direction of laboratory computer systems, internal and external software development, database management, records management system and control of ESC's laboratory information management system. Prior to joining ESC, Mr. Chandler served as VP of eCommerce for a large internet retailer for seven years, preceded by three years in management within a major consulting company. He has over twenty-three years experience in information technology disciplines, including project management, software development, hardware infrastructure planning /deployment, and voice/data analysis. Tom White is responsible for the department in Mr. Chandler's absence.

4.1.6 Management System Effectiveness

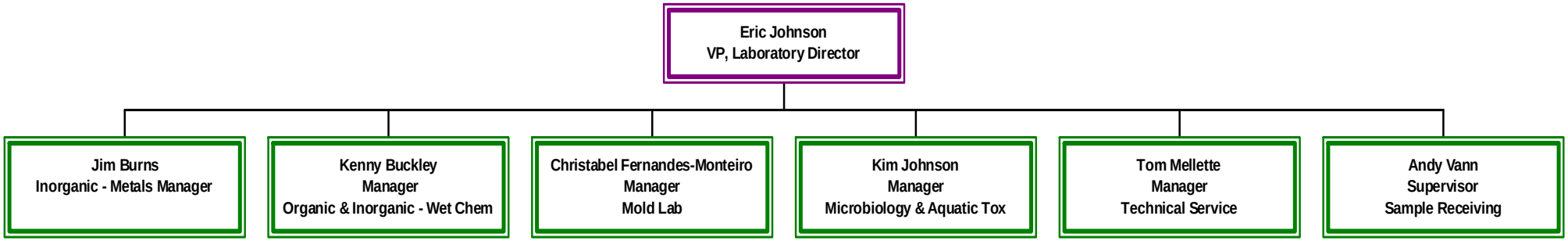
Senior management ensures that appropriate communication processes are established within the laboratory for implementation of the management system and that communication takes place regarding the effectiveness of the management system.

Figure 4.1 is the corporate organizational chart, which lists key individuals and relevant departmental structure.

Figure 4.1 Corporate Organizational Chart (Subject to change)



Laboratory Department Managers



4.2 MANAGEMENT SYSTEM

4.2.1 Management Documentation

Management system documentation consists of different levels:

- Documented statements of the quality policy (issued under the authority of the chief executive officer) and the quality objectives of this manual
- Documented procedures required by all applicable standards that detail the implementation of requirements and operation guidelines.
- Instructions: details of quality or inspection information and specific instructions for performance of individual tasks.
- Documents needed by the organization to ensure the effective planning, operation and management of its processes
- Records required by all applicable standards per the records procedure.

When the term “documented procedure” appears within this quality manual, the procedure is established, documented, implemented and maintained.

The laboratory maintains its documents in various formats including paper and various electronic formats.

4.2.2 Quality Management Policy

The management of ESC is committed to maintaining a quality assurance/quality control program that allows data generated by ESC, or any subcontractors under ESC's supervision, to meet both required and stated accuracy goals. The most important aspect of the program is to ensure that all activities whether involving sampling, analytical, or engineering activities, are congruent with EPA laboratory practices and regulatory guidelines. Issues relating to the quality program are reviewed during weekly operations meetings with upper management and in quarterly management reviews. ESC personnel who have direct responsibility for overseeing the quality assurance program report to ESC's president.

ESC has a diverse accreditation/certification program, which requires continuous monitoring of changes and modifications within a variety of state and federal organizations. The certification program represents greater than 48 separate state and national certifications. ISO 17025 is maintained as the minimum foundation to meet each program requirement. This requires an extreme dedication to the overall quality system and analytical testing.

4.2.3 Management Commitment

ESC management is committed to the development and implementation of the laboratory's management system as well as compliance with all statutory and regulatory requirements. These commitments, along with the importance of meeting client requirements, are continually communicated to all levels within the laboratory.

4.2.4 Commitment to the QAM and Related Procedures

Data Integrity is the result of the processes that work together to assure the production of data of known and documented quality.

The ESC Policy Manual requires a strict adherence to ethics and confidentiality. This policy covers all aspects of the laboratory function from client contact to sample analysis and analytical reporting, invoicing, and archive. Each staff member must maintain a professional attitude towards all colleagues, regulators, auditors, and laboratory clients while continuously striving to improve technical knowledge and professional competence.

ESC supports individual authority and provides the necessary resources for each staff member to carry out their duties. Each staff member is responsible for the identification of departures, from the quality system and/or established analytical procedures, within their area of concern, and for the initiation of actions to prevent or minimize such departures. In addition, ESC strives to ensure that its management and personnel are free from any undue internal and external commercial, financial, and other pressures and influences that may adversely affect the quality of their work.

All ESC personnel, including contract and temporary, are required to sign an "Attestation of Ethics and Confidentiality" at the time of employment and during annual refresher training. This document clearly identifies inappropriate and questionable behavior. Violations of this document result in serious consequences, including prosecution and termination, if necessary. The ESC Policy Manual addresses this subject in detail. See SOP# 010102, *Ethics, Data Integrity, and Confidentiality.*

4.2.4.1 Quality Manual (QAM)

ESC has established and maintains a quality manual that:

- Defines the structure of the management system.
- Makes reference to the quality policy, the supporting procedures (also technical) and instructions.
- Defines the roles and responsibilities of technical and quality staff

The management system documentation is communicated to each laboratory staff member. All employees sign a document, kept in their personnel file, which states that they have read and understood the *Quality Manual*, including the quality policy.

4.2.4.2 Commitment to the QAM and Related Procedures

This Quality Assurance Manual outlines the procedures that have been developed to implement laboratory policies and to fulfill the laboratory's commitment to the client. These procedures are further defined and integrated into ESC's standard operating procedures. The policies are stated such that this manual serves as a QA handbook of responsibilities for all laboratory personnel. The manual is reviewed and approved under the authority of the highest level of laboratory management. Where the *Quality Manual* documents laboratory requirements, a separate SOP or policy is not required. This document is also used as a supplement for project planning, client reference, and personnel training.

4.2.5 Procedure List

A list of the procedures, the instructions and the quality records, which are included in the management system, is maintained by the Quality Department and is available via the ESC intranet.

4.2.6 Management Commitment

4.2.6.1 Programs

The management of ESC is the main support of the quality program. Each manager is aware of the requirements of each external auditing agency and is responsible to ensure that their respective departments meet the requirements of each agency. ESC maintains full compliance and agreement with the following organizations/regulations: A2LA, ISO 17025, AIHA, EPA, GALP/GLP, NELAP, and individual states who carry primacy concerning certification and regulation.

4.2.6.2 ESC Policy Manual

ESC has policies and procedures, in the ESC Policy Manual, to insure that there is no employee involvement in any activities that would diminish confidence in their competence, impartiality, judgment or operational integrity.

All staff members employed by ESC are issued a Company Policy Manual that covers a wide array of topics and defines the expectations and policies of ESC. The Manual addresses both corporate and professional conduct, including confidentiality, professional ethics, and discipline. No deviations from the company policy are permitted without the approval of the CEO.

4.2.7 Management of System Changes

Top management ensures that the integrity of the management system is maintained when changes to the management system are planned and implemented.

4.3 DOCUMENT MANAGEMENT

This Section describes procedures for document management, which includes controlling, distributing, reviewing, and accepting modifications. The purpose of document management is to ensure that adequate instruction is readily available for laboratory employees and to preclude the use of invalid and/or obsolete documents.

The laboratory manages three types of documents: 1) controlled, 2) approved, and 3) obsolete.

A CONTROLLED DOCUMENT is one that is uniquely identified, issued, tracked, and kept current as part of the quality system. Controlled documents may be internal documents or external documents.

APPROVED means reviewed, and either signed and dated, or acknowledged in writing or secure electronic means by the issuing authority(ies).

OBSOLETE DOCUMENTS are documents that have been superseded by more recent versions.

4.3.1 Required Documents

Documents required by the management system, as well as analytical records are managed per the SOP #010103, *Document Control and Distribution Procedure*.

4.3.2 Document Control

The documentation management procedure is established to define the means needed to:

- Approve documents for adequacy prior to issue
- Review, update and re-approve existing documents as necessary
- Ensure that changes and the current revision status of documents are identified
- Ensure that relevant versions of applicable documents are available at points of use
- Ensure that documents remain legible and readily identifiable
- Ensure that documents of external origin are identified and their distribution managed using the documentation master list
- Prevent the unintended use of obsolete documents and to apply suitable identification to them if they are retained for any purpose.

4.3.2.1 Document Review and Approval

Documents are reviewed and approved for use by the individual department managers and QAO, or designee, prior to issue.

Documents are reviewed at least annually or sooner, as deemed necessary to ensure their contents are suitable, comply with the current quality systems requirements and accurately describe current operations.

Approved copies of documents are available at all locations where operations are essential to the effective functions of the laboratory.

4.3.2.2 Document Distribution

Controlled internal documents are uniquely identified with:

1) date of issue
2) revision identification
3) page number
4) total number of pages or a mark to indicate the end of the document
5) the signatures of the issuing authority (i.e. management).

A master list of controlled internal documents is maintained that includes distribution, location, and revision dates. A master list of controlled external documents is also maintained that includes title, version or copyright date, and and location. The controlled document list is maintained by the QA Department and is continually updated. All invalid or obsolete documents are removed from circulation and clearly marked to prevent use. Obsolete documents retained for legal use or historical knowledge preservation are appropriately marked and retained.

4.3.3 Changes to Controlled Documents

4.3.3.1 Review and Approval of Changes

Document changes are re-approved by the original approving authority.

4.3.3.2 Identification of New or Altered Text

Where practicable, the altered text or new text in the draft is identified during the revision or review process to provide for easy identification of the modifications. Pending changes in each revision are indicated in the ESC SOP/Minor Revision Form that is attached to the SOP. Historical changes are described in the SOP Attachment I, Revision History.

4.3.3.3 Procedure for Document Revision

Document revision is controlled under SOP# 010103, *Document Control*. Suggested revisions to electronic documents are presented to management for review and approval. Changes to electronic documents can only be made by the QAO, or designee. The document management process allows for "minor revisions" or amendments to documents where changes are not sufficient to cause a full procedure change. Minor revisions may take the form of handwritten notes on an approved SOP Minor Revision form. Document changes are approved with signature and date by management. The modified document is then copied and distributed, and obsolete documents are removed. Minor revisions to documents are incorporated into the next full revision as soon as practicable.

4.3.3.4 Changes in Electronic Documents

The QA Manual, SOPs, Safety Plan, and other controlled documents are maintained electronically on a protected directory. Access rights are restricted to QA personnel and the IT Director. Electronic copies of current and previous versions of all controlled documents are maintained on the computer network system. They are stored with the same security settings as the most recent version; however previous versions of documents are access controlled to prevent employee use of outdated material. The documents are archived to tape storage with regular back up of the entire network system

4.3.3.5 Standard Operating Procedures

Standard Operating Procedures (SOPs) are written procedures that describe in detail how to accurately and consistently reproduce laboratory processes or provide additional direction for laboratory personnel. Copies of all SOPs are accessible to all personnel. SOPs consist of three types:

- Technical SOPs, pertaining to a laboratory process which have specifically required details
- Administrative SOPs which document the more general organizational procedures.
- Quality SOPs that provide background and process for quality policy.

SOPs do not have to be formal documents with pre-defined section headings and contents. They can be less formal descriptions of procedures described in the *Quality Manual* or other documents.

4.3.3.5.1 Format

Each SOP indicates the effective date, the revision number, and the signature(s) of the QA Department and Department Manager/Laboratory Director. Department Manager approval is also required on technical procedures. Detailed information can be found in SOP# 010100, *Writing, Revising, and Maintaining Standard Operating Procedures*

All Standard Operating Procedures, QA Manuals, and Safety Plans are written in a format that incorporates the document name, date revised, pages included, and section.

Deviations from SOPs and Quality documents are not allowed without the permission of the QAO, or designee. In the event that a deviation is requested, the circumstance is considered and the procedure is evaluated for necessary change and allowance.

Determinative Method SOPs

The laboratory has SOPs for all analytical methods within its scope, which is listed in Table 3.1. Where equipment manuals or published methods accurately reflect laboratory procedures in detail, a separate SOP is not required. Any deviation from a method is documented in the method modifications section of the respective SOP, including both a description of the change made and a technical justification. The deviation is reported to the client. Each determinative method SOP includes or references (as applicable) the following:

- Scope and Application;
- Method Summary and Definitions;
- Health and Safety;
- Sample Preservation, Containers, Handling and Storage;
- Interferences;
- Equipment and Supplies;
- Reagents and Standards;
- Procedure;
- Data Analysis and Calculations;
- Quality Control and Method Performance;
- Data Validation and Corrective Action;
- Pollution Prevention and Waste Management;
- Method Modifications/Clarifications;
- References;
- Procedure Revision/Review History;

4.4 REVIEW OF REQUESTS, TENDERS, AND CONTRACTS

4.4.1 Procedure for Contract Review

When ESC enters into a contract to provide laboratory services, it follows SOP# 020303, *Contract Review*. On receipt of a request or invitation to tender, the clients' requirements are examined by the contract review personnel to establish that the necessary details are adequately outlined and that the laboratory is able and willing to meet them.

4.4.2 Records of Reviews

Records of reviews of requests, tenders and contracts (including significant changes) are maintained. Records are also maintained of pertinent discussions with the client relating to the client's requirements and the results of the work during the period of execution of the contract.

4.4.3 Subcontracted Work

Clients' requirements for custom analyses and for work subcontracted to other laboratories are reviewed by the appropriate technical staff for logistics and feasibility.

4.4.4 Deviations from the Contract

The client and the affected personnel are informed of any deviation from the contract.

4.4.5 Contract Amendments

If a contract requires amendment after work has commenced, the same contract review process is repeated and any amendments are communicated to all affected parties.

4.5 SUBCONTRACTING

A subcontract laboratory is defined as a laboratory external to ESC, or at a different location than the address indicated on the front cover of this manual, that performs analyses for this laboratory.

4.5.1 Subcontractor Competence

ESC only performs analytical techniques that are within its documented capability, when this is not possible, the laboratory follows SOP# 030209, *Subcontracting.* Subcontracting occurs in the special circumstances where technical, safety, or efficiency issues dictate need. When subcontracting analytical services, the laboratory assures work requiring specific accreditation is placed with an accredited laboratory or one that meets applicable statutory and regulatory requirements.

4.5.2 Client Notification

ESC notifies the client of the intent to subcontract the work in writing. The laboratory typically gains the approval of the client to subcontract their work prior to implementation, preferably in writing.

4.5.3 ESC Responsibility

ESC assumes responsibility for the qualifications of the subcontractor (except when the client or an authority specifies a subcontractor) and the client is advised.

All reports, which contain data from subcontracted laboratories, include a statement on the final report, which references the subcontractor laboratory/service. As part of the initial subcontractor approval process, a copy of the applicable certificates and scopes for subcontractor's accreditation/certifications is maintained as evidence of compliance.

4.5.4 Subcontractor List

ESC maintains a list of all approved subcontract laboratories.

4.6 PURCHASING SERVICES AND SUPPLIES

4.6.1 Purchasing Policies and Procedures

ESC maintains SOP# 030210, *Materials Procurement for Analytical Processes*, which describes the purchasing process, including vendor selection and acceptance criteria, for the purchase, storage, and evaluation of supplies and services. Where specifications of outside services and supplies are relevant to the measurement integrity of analyses, ESC uses services and supplies of adequate quality. The various department managers are responsible for ordering supplies/chemicals that meet the method stated requirements.

4.6.2 Quality of Purchased Items

Where assurance of the quality of outside support services or supplies is unavailable, the laboratory uses these items only after they have been inspected or otherwise verified for adequate quality. Records of inspections, verifications, and suppliers are maintained in the laboratory.

4.6.3 Purchasing Documents

Purchasing documents contain data clearly describing the product and/or services.

4.6.4 Approved Supplier List

An approved list of material/service suppliers is maintained where products/services purchased affect the quality of analyses produced by the laboratory.

4.7 SERVICE TO THE CLIENT

The ESC Technical Service Department provides specific project service through the use of Technical Service Representatives (TSRs). The TSR is responsible for all contract requirements and laboratory/client communication, including information concerning schedules, delays, and major deviations in the testing process.

4.7.1 Meeting Client Expectations

The TSR works closely with the client to clarify the client's requests and to monitor the laboratory's performance in relation to the work requested, while ensuring confidentiality to other clients. The laboratory confidentiality policy prohibits divulging or releasing any information to a third party without proper authorization. See SOP# 010102, *Ethics, Data Integrity, and Confidentiality*. All electronic data (storage or transmissions) are kept confidential, based on technology and laboratory limits, as required by client or regulation. All electronic transmissions contain a confidentiality notice that represents the following: *Notice: This communication and any attached files may contain privileged or other confidential information. If you have received this in error, please contact the sender immediately via reply email and immediately delete the message and any attachments without copying or disclosing the contents. Thank you.*

For additional information see SOP# 020301, *TSR (Project Management).*

4.7.2 Client Feedback

Service related feedback is obtained from clients by surveys. This feedback is used to improve the management system, quality system, testing and calibration activities and client services. The feedback is discussed in management reviews.

4.7.3 Client Access

ESC provides reasonable access, as needed by outside parties, to relevant areas of the lab for witnessing tests.

4.7.4 Client Project Information

Clients may be provided supplementary documents, as needed, to further strengthen the project information. This may include: preparation documents, packaging information, verification of calibrations, and certification information.

4.7.5 Communication with the Client

ESC's Technical Service Representatives maintain good communication with outside parties and are able to provide sound advice/guidance in technical matters and opinions/interpretations based on results. Communication with the client, especially in large assignments, is maintained throughout the work. The client is informed of any delays or deviations in the performance of the tests and/or calibrations.

4.8 COMPLAINTS

The purpose of this section is to ensure that customer complaints are addressed and corrected. This includes requests to verify results or analytical data. All client concerns are initially addressed by the Technical Service Representatives. If further resolution is required, the QAO (or designee) and other pertinent personnel, as deemed necessary by the depth of the problem, conduct needed investigations and provide client support. See SOP# 020302, *Client Complaint Resolution Procedure*.

4.8.1 Investigation of Complaints

In the event of a complaint, negative audit finding, or any other circumstance, which raises doubt concerning the laboratory's competence or compliance with required procedures, the laboratory ensures that those areas of activity are promptly investigated. A resolution of the situation is promptly sought and, where necessary, retesting is conducted.

4.8.2 Causes and Corrective Actions

The personnel in the quality department examine all documents and records associated with complaints and the department manager investigates audit findings and other circumstances. This investigation seeks to identify specific root causes and initiate any necessary corrective action.

4.8.3 Documentation

Records of events and the actions taken by the laboratory to resolve issues and to prevent future occurrences are maintained (see Section 4.11).

4.9 CONTROL OF NON-CONFORMING WORK

4.9.1 Policies and Procedures

A nonconformance is an event that does not meet the requirements of the governing documents. Nonconformances can include unacceptable quality control results (See SOP# 030208, *Corrective Action)* or departures from standard operating procedures or test methods. Requests for departures from laboratory procedures are approved by the QAO, or designee, and documented.

Types of non-conformances are:

- Deviations from written procedures that were not pre-approved by QA.
- Changes to an existing SOP that is not included in the current revision
- A single and/or continuous trend of inappropriate habits
- A single and/or continuous trend of bias in the QC results
- Unusual changes in detection limit
- Deficiencies identified during an internal/external audit
- Unacceptable results on performance testing samples
- Valid issues reported by clients, data reviewers, or auditors
- General activities that demonstrate the possibility of a negative impact to the quality of the data

A policy has been established to ensure the use of analytical techniques that do not conform to specified requirements are prevented. This control provides for identification, documentation, evaluation, segregation (when practical) and disposition of nonconforming tests/calibrations. The control also calls for notification to the appropriate laboratory divisions. Any non-conforming tests/calibrations are reported to the supervisor of the affected laboratory division who is responsible for corrective actions. Records are documented on corrective action requests.

4.9.2 Correcting Non-conforming Work

The correction action system is used to identify nonconforming tests and/or calibrations. See SOP 030208, *Corrective and Preventive Action.*

4.9.3 Review and Disposition of Nonconforming Tests/Calibrations

Since the laboratory has adopted a continuous improvement philosophy, it has established a procedure for reviewing and disposing of nonconforming tests/calibrations. This procedure includes:

- Reworking the test/calibration to meet the requirements
- Rejecting the test/calibration
- Informing the client (if necessary)

4.10 IMPROVEMENT

The laboratory continually improves the effectiveness of its management system through the use of the quality policy, quality objectives, audit results, analysis of data, corrective and preventive actions and management review.

4.11 CORRECTIVE ACTIONS

ESC strives for the continual improvement of its organization and its services. Corrective Action is the process used to eliminate the causes of an existing nonconformity, defect, or other undesirable situation in order to prevent recurrence.

ESC recognizes that the data supplied by the professional staff must be legally and technically defendable. The Regulatory Affairs personnel continually monitor the quality assurance program to ensure that this goal is achieved. Each analyst is responsible for initiating corrective actions in their areas of expertise. The QAO, or designee, and Department Managers administer corrective action approval. It is the Manager's responsibility to evaluate the Corrective Action, appoint the appropriate person within the department to be responsible for completion of the CAR and submit it to the QA Department for processing.

4.11.1 General

The initiation, management, tracking, and closure of corrective actions is described in SOP# 030208, *Corrective and Preventive Action.*

4.11.2 Investigation of Corrective Actions

Each lab division is encouraged to take any corrective action to determine and eliminate the causes of actual nonconformances to the degree appropriate to the magnitude of problems and commensurate with the risks encountered.

4.11.3 Selection and Implementation of Corrective Actions

In addition to SOP# 030208, *Corrective and Preventive Action*, more specific guidance can be found in each determinative method.

In general, the corrective action procedure includes:

- The effective handling of client complaints and reports of nonconformities
- Investigation of the root cause of nonconformities relating to process, service, and management systems, and recording of results
- Determination of the corrective action needed to eliminate the cause of nonconformities
- Application of controls to ensure that corrective action is taken and that it is effective.

4.11.4 Monitoring of Corrective Actions

The closure and follow-up activities of corrective actions are approved and documented in ESC's tracking system to ensure that the actions have been effective in addressing and correcting the problem.

4.11.5 Additional Audits

When the identification of non-conformities or the corrective action investigation casts doubt on compliance with policies and procedures or the management system, laboratory management ensures that appropriate areas of activity are audited in accordance with Section 4.14.1. The results of corrective action are submitted for laboratory management review.

4.11.6 Cessation and Restarting of Work

All technical personnel are capable of invoking a "stop work" order, in the event that a situation impacts data validity or safety. It is the responsibility of the following personnel to (1) evaluate a "stop work" order whenever a severe non-conformance warrants a cessation of analysis and (2) ensure that the cause of the stop work order has been satisfactorily resolved and approve the restarting of work:

- Laboratory Manager/Director
- QA Department
- Technical Director/Supervisor
- Technical Service Representative

Technical directors review corrective action reports and suggest improvements, alternative approaches, and amended/revised procedures, where needed. If the data reported are affected adversely by the nonconformance, the client is notified in writing. The discovery of a nonconformance for results that have already been reported to the client must be immediately evaluated for significance of the issue, its acceptability to the client, and determination of the appropriate corrective action.

4.11.7 Release of Non-conforming Work

The laboratory allows the release of nonconforming data only with approval on a case-by-case basis by the appropriate Technical Director, or their designee. Planned departures from procedures or policies do not require audits or investigations. Permitted departures for nonconformances, such as QC failures, are fully documented and include the reason for the deviation and the impact of the departure on the data.

4.11.7 Other Sources That May Initiate Corrective Action

Deficiencies cited in external assessments, internal quality audits, data reviews, complaints, or managerial reviews are documented and require corrective action. Corrective actions taken are appropriate for the magnitude of the problem and the degree of risk.

Appendix II lists the current federal and state agencies that perform audits of ESC. This table also lists the required performance evaluations that may initiate corrective actions. ESC implements any reasonable corrective action deemed necessary by the regulatory QA/Certification Officers. In addition, the following types of samples may also initiate corrective action: split samples sent to another qualified laboratory, monthly blind field duplicates, quarterly purchased round robin samples, client submitted QC samples and periodic internal blind samples.

4.11.8 Corrective Action Documents

In general, corrective action documents are maintained by the Regulatory Affairs Department. These documents include the following: corrective action resulting from both internal and external audits, corrective action resulting from performance evaluation testing, corrective action as deemed necessary by the QA Department.

Corrective action resulting from analytical failure is kept with the analytical data and is recorded on the bench sheet or raw data. The Department Manager is responsible for making sure that suitable measures have been taken to ensure that the problem is identified and corrected.

Corrective action involving sample receiving is recorded on a Nonconformance form and is then filed with the original Chain of Custody.

4.12 PREVENTIVE ACTIONS

Preventive Action, rather than corrective action, aims at minimizing or eliminating inferior data quality or other nonconformance through scheduled maintenance and review, before the actual nonconformance occurs.

4.12.1 Management of Preventive Actions

ESC Management encourages preventive action measures. Each staff member is empowered to make suggestions for improving or fool-proofing processes throughout ESC. Where process areas show potential for nonconformance, measures are taken to identify the problem and formulate a plan to implement the defined change needed. The QAO, or designee, reviews any recommended changes before implementation to ensure the effectiveness of the modification.

4.12.2 SOP# 030208, *Corrective and Preventive Action*, is also employed for preventive actions.

In general, the procedure for preventive action includes:

- The use of appropriate sources of information, such as processes and work operations, which affect product or service quality, concessions, audit results, quality records, service reports, and client complaints to detect, analyze, and eliminate potential causes of non-conformities.
- Determination of the steps needed to deal with any problems requiring preventive action
- Initiation of preventive action and application of controls to ensure that it is effective.

Preventive action includes, but is not limited to, review of QC data to identify quality trends, regularly scheduled staff quality meetings, annual budget reviews, annual managerial reviews, scheduled column trimming, running a new LIMS system in tandem with the old system to assure at least one working system, and other actions taken to prevent potential problems.

4.12.3 Trend Analysis

A trend analysis is an investigation that involves the collection of data in a manner that reveals deviations over time. Examples of laboratory processes that can be analyzed for trend analysis are:

- Sample receipt or chain of custody discrepancies
- Sample storage or preservation errors
- Holding time violations
- Instrument calibration
- Control Charts – Charts that are generated from historical data that plot percent recovery vs. time
- Method QC failures and problems

4.13 CONTROL OF RECORDS

Records are a subset of documents, usually data recordings that include annotations, such as daily refrigerator temperatures, posted to laboratory forms, lists, spreadsheets, or analyst notes on a chromatogram. Records may be on any form of media, including electronic and hardcopy. Records allow for the historical reconstruction of laboratory activities related to sample handling and analysis.

4.13.1 General

Technical and quality assurance records are established and maintained to provide evidence of conformity to requirements and of the effective operation of the quality system. Mechanisms are established for records to remain legible, readily identifiable and retrievable. The laboratory maintains a record system appropriate to its needs, records all laboratory activities, and complies with applicable standards or regulations as required.

The laboratory has defined the length of time various records, pertaining to the management system and examination results, are to be retained. Retention time is defined by the nature of examination or specifically for each record. The laboratory retains all original observations, calculations and derived data, calibration records, chain of custody and a copy of the test report for a minimum of ten years, unless otherwise required by regulatory authority.

A documented records procedure SOP# 010103, *Document Control and Distribution Procedure*, and SOP# 020304, *Protection and Transfer of Records*, is established to define the means needed for the identification, storage, protection, retrieval, retention time, transfer, and/or disposition of records.

4.13.2 Technical and Quality Records

NOTE: ALL records/data are stored for a minimum of 10 years, unless otherwise noted.

All hardcopy department logbooks, such as temperature, maintenance, and preparation logs are placed into storage boxes and archived via a unique numbering system, to the ESC storage facility. Additional information regarding reagents/standards can be found in the Standards Logger (Tree) digital archive system. This digital system is backed up according to the ESC IT backup procedure.

Archived information and access logs are protected against fire, theft, loss, environmental deterioration, vermin, and in the case of electronic records, electronic or magnetic sources.

Data Storage Criteria	
Data Type	**Storage Criteria**
Manual Data Wet Chemistry	All manually generated data are stored in specific laboratory analysis workbooks. Each individual analysis is located in a separate notebook which contains all data relating to the test including, calibration curves/data, QC charts/limits, SOP, and completed analysis sheets. These notebooks are centrally located and contain completed data that is filed by analysis and date analyzed. Monthly – Data is removed from the notebook and placed in a dedicated filing cabinet. Semi-annually – Data is removed from the filing cabinet, placed in storage boxes and archived, via a unique numbering system, in the ESC storage facility
Manual Data Prep Labs	All logbooks utilized in manually recording sample preparation information are placed into storage boxes and archived, via a unique numbering system, in the ESC storage facility. This includes organic prep, metals prep, and TCLP.
Manual Data Env. Micro, Mold	All manually generated data is stored in specific laboratory files and notebooks. These files are centrally located and contain completed data that is filed by analysis and date analyzed. Data is placed into storage boxes and (when full) archived, via a unique numbering system, in the ESC storage facility.
All Data Aquatic Toxicity	All manually generated data is stored in specific laboratory files and notebooks. These files are centrally located and contain completed data that is filed by analysis and date analyzed. Data is placed into storage boxes and (when full) archived, via a unique numbering system, in the ESC storage facility. Final reports and Reference Toxicant results are also scanned into ESC's electronic document management system. The data storage device on which this data resides is backed up daily. Data files are archived on to magnetic tape and retained per laboratory policy.
Computerized Data - Organic Dept.	Injection logs are printed and kept in a notebook with the instrument. The instrument data is printed to a secure server and remains in a format that cannot be changed after printed. Upon printing, the data in the original file is generated. This storage system is backed up nightly utilizing a seven-day rotation cycle. The data is immediately available for up to two years. After two years, raw instrument data files are archived onto a separate secure server and kept a minimum of ten years. Original raw data files cannot be edited.
Computerized Data – Inorganic Metals Dept.	All data produced by metals instrumentation is backed up to a secure drive, nightly, utilizing a seven-day rotation cycle. Hard copies are printed and filed by date and instrument. All data is archived on a network attached storage device and is immediately available for up to two years. After two years, raw instrument data files are archived on to a separate secure server and kept a minimum of ten years. Original raw data files cannot be edited.
Final Report Storage - LIMS	The LIMS facilitates access to any finished data and sample information by client code, sample number, and parameter run number. Furthermore, any data pertaining to a sample or client can be obtained. The LIMS also contains the information from the COC such as sample description, time and date collected, sampler ID, container type, preservative, sample receipt data, finished/approved analytical data, analyst, etc. The LIMS Oracle Database is backed up daily on tape. The back up tape is kept in secure storage. While all LIMS data are accessible, data older than six months is moved from the active production database and is available in an archive database.
Final Report Storage - PDF	Copies of all reports are stored according to client code in PDF format on a network attached storage device and are immediately available for up to ten years**.** After ten years data files are archived onto magnetic tape and kept an additional ten years. These reports include chain of custody forms, login confirmation reports, the final approved printed report, invoices and any other associated documents. Samples that require subcontract work also have a copy of the final report in the client file.
Misc. Data Storage	Company records that are not stored on a secure electronic device are placed in storage boxes and archived, via a unique numbering system, in the ESC storage facility. This includes quality records, such as audits, state certifications, PT results, internal audits, corrective actions, training files, logbooks, etc.

4.13.3 Records Disposal

Records that have exceeded the required storage requirement are disposed of through the use of professional records destruction firm. ESC retains the manifest of documents destroyed and files the verification receipt that is generated at the time of destruction.

4.13.4 Records Transfer

In the event that corporate ownership is transferred or that laboratory activities are terminated for any reason, all records become property of the transferee in accordance with ESC SOP# 020304, *Protection and Transfer of Laboratory Records*.

4.13.5 Legal Chain of Custody Records

Evidentiary Sample Data are used as legal evidence. Procedures for evidentiary samples are documented in a separate SOP.

4.14 AUDITS

4.14.1 Internal Audits

SOP# 010104, *Internal Audits*, addresses the implementation and maintenance procedure for a comprehensive system of internal audits at planned intervals to verify the on-going effectiveness of the management system.

4.14.1.1 The QA Department is responsible for administering the internal audit system per the documented procedures. The department develops a schedule for internal audits according to management system requirements and conducts unscheduled audits (internal and external) when reasons for such audits exist.

4.14.1.2 Audits are conducted utilizing documented checklists and/or audit plans. Audit results are documented in audit reports per established procedures. Copies of all audit reports including completed corrective action requests are forwarded to management of the audited area and maintained by the quality assurance department.

4.14.1.3 Audit plans are structured according to the following:

State/Certifying Agencies - Internal audits are conducted according to the various requirements set forth by the state and international agencies that accredit ESC. In addition, work procured from non-certifying states, also determine other requirements set forth by the state of origin. The audits are conducted to maintain compliance with the following Quality Standards: AIHA LQAP, A2LA, ANSI/ISO 17025, NELAC, and DOD QSM.

Method Specific Criteria – Technique, analytical method, standard operating procedures, and effectiveness are also reviewed during the internal audit. ESC maintains compliance with methods as listed in section 2.1.3.

Data Integrity and Analyst Ethics - In addition to established standard and method related criteria; the internal audit is designed to review the analytical data for integrity and defensibility. Any suspicion of ethics violations result in a confidential investigation involving only the QAO, or designee, Director of Technical & Regulatory Affairs, and any specialist personnel necessary to conduct a complete and thorough investigation. Investigations, of this type, are conducted in a timely manner and all details and supporting documentation are recorded and maintained for a period of at least 10 years. All investigations that result in findings of inappropriate activity are documented and include any disciplinary actions involved, corrective actions taken, and all appropriate notifications to clients. Clients are notified promptly when audit findings cast doubt on the validity of the data.

Support Systems – The internal audit process is also designed to assess support systems that are not a direct part of analytical activities. This includes, but is not limited to, the following:

- Contract Review
- Procurement and Vendor Approval
- Inventory Control
- Document Control
- Subcontracting
- Environmental, Safety, Security, and Health (ESSH)

4.14.1.4 Audit personnel are qualified per documented procedures and do not have direct responsibility for or control over the area being audited.

4.14.1.5 Management personnel responsible for the audited area determine and implement timely corrective actions for any reported nonconformance.

Follow-up audit activities include verification of the corrective actions taken and reporting of the results.

4.14.2 Performance Audits

Performance audits require evaluation of control and blind results. On a quarterly basis, documentation of results and corrective actions are evaluated as part of the management review process.

4.14.3 Proficiency Testing

The laboratory participates in various proficiency testing samples (PT) as required by each accreditation, and obtains test samples from approved providers. Corrective action procedures are initiated for all failed PT samples. All studies are conducted independently and no attempts are made to compare or obtain results from other labs or the provider. Proficiency Testing (PT) or Proficiency Evaluation (PE) samples are treated as typical samples in the normal production process where possible, including the same preparation, calibration, quality control and acceptance criteria, sequence of analytical steps, number of replicates, and sample log-in. PT samples are not analyzed multiple times unless routine environmental samples are analyzed multiple times.

- ***Annual Studies***

Study	***Frequency***	***Vendor***
WP (Water Pollution)	Semi-annually	Environmental Resource Associates
WS (Water Supply)	Semi-annually	Environmental Resource Associates
Matrix – Soil RCRA	Semi-annually	Environmental Resource Associates
Matrix – UST Soil/Water	Semi-annually	Environmental Resource Associates
Matrix – Air Canisters	Semi-annually	Environmental Resource Associates
DMRQA – Chemistry	Annually	Environmental Resource Associates
DMRQA – Aquatic Tox.	Annually	Environmental Resource Associates
ELLAP	Quarterly	AIHA
IHLAP	Quarterly	AIHA
EMLAP	Quarterly	AIHA
EMLAP – Direct Exam	Quarterly	AIHA
EMLAP – Fungal / Bacterial	Triannually	AIHA
Cryptosporidium / Giardia	Quarterly	US EPA
Aquatic Toxicity Performance Evaluation	Annually	North Carolina

- ***Blind Field Duplicates*** **–** ESC collects blind duplicates periodically to evaluate field collection and laboratory precision. ESC routinely receives unmarked field duplicates from clients to evaluate sample batches.

- ***Split Samples*** – ESC periodically participates in split samples with outside laboratories to confirm analytical results. This is performed on a project specific basis.

4.14.4 External Audits

ESC agrees to host on-site system audits from external organizations and currently participates in various system and performance audits. It is the laboratory's policy to cooperate and assist with all external audits, whether performed by clients or an accrediting authority. All external audits are fully documented and tracked to closure.

Management ensures that all areas of the laboratory are accessible to auditors as applicable and that appropriate personnel are available to assist in conducting the audit. Any findings related to an external audit follow corrective action procedures. Management ensures that corrective actions are carried out within the timeframe specified by the auditor(s).

SDWA

The ESC laboratory (EPA No. TN00003) is certified by the State of Tennessee under the Safe Drinking Water Act. The State of Tennessee routinely audits the ESC laboratory procedures, quality control and methods and has found the laboratory practices to be consistent with EPA requirements. ESC is also audited under the Safe Drinking Water Act by Arizona, Iowa, North Carolina, New Jersey - NELAP, and the A2LA. ESC maintains several other DW certifications, which have been granted in reciprocity. ESC participates in WS PE studies in support of drinking water certifications.

CWA/RCRA

ESC is certified for wastewater and solid waste through audits by the following states/organizations: A2LA, Arizona, Iowa, Minnesota, New Jersey (NELAP), North Carolina, OHIO VAP, West Virginia, Wisconsin, and USACE. In addition to Water Pollution or Non-Potable water studies, ESC is required to analyze additional blind samples for West Virginia. The laboratory is also periodically audited by the Metropolitan Government of Nashville and Davidson County and certified for wastewater sampling and analysis. ESC participates in WP Studies, DMR QA program, and Solid Matrix PE studies.

INDUSTRIAL HYGIENE

The American Industrial Hygiene Association routinely audits ESC to maintain certification for analytical support of organic chemical exposure monitoring, microbiological testing and metals exposure activities. ESC currently participates

in the required performance testing studies and maintains the quality systems to satisfy the requirements necessary for certification in the following: Environmental Lead (air, soil, paint and wipes), Industrial Hygiene (air filters, diffusive samplers, and sorbent tubes), Environmental Microbiology (fungal/bacterial testing and identification)

CLIENT AUDITS

Due to participation in a number of national contracts, ESC is audited by several clients; who are also ISO certified and are required to assess their suppliers.

ESC is subject to several external audits on an annual basis. The audits cover all disciplines, SDWA, CWA, CAA and RCRA/UST. In addition, the laboratory also participates in additional performance testing, where required by individual clients and for new method development purposes.

4.15 MANAGEMENT REVIEW

4.15.1 Items in Management Review

Regular management review meetings take place quarterly during the months of January, April, July and October and cover the events from the preceding quarter. The Quality Assurance Officer (QAO), the Laboratory Director, and all Department Managers are responsible for attending each meeting. Guidance, including agenda items, is given in ESC SOP# 010105, *Management Review*.

4.15.2 Records of Management Review

The Director of Technical & Regulatory Affairs and QA Department collects objective evidence on the effectiveness of the management system. This includes audit results, client feedback, contract performance data, nonconformance data, problem reports, changes affecting the management system and previous management review reports.

4.15.3 Evaluation

On the basis of this input, the management system is tested for its effectiveness, for its relevance, and for its implementation. In particular, quality objectives and the objectives set within the management system are examined. Adjustments are considered due to changes in the conduct or scope of business.

4.15.4 Improvement

Decisions are made regarding actions needed to improve the effectiveness of the quality management system.

4.15.5 Procedure

Details of this review, how it is be performed and recorded and the associated responsibilities can be found in the procedure for ESC SOP# 010105, *Management Review*.

5.0 TECHNICAL REQUIREMENTS

5.1 GENERAL

5.1.1 ESC recognizes that many factors determine the correctness and reliability of the analyses performed by a laboratory. These factors include contributions from: human factors (5.2), accommodations and environmental conditions (5.3), analytical/calibration methods and method validation (5.4), equipment (5.5), measurement traceability (5.6), and sample management - handling of test/calibration items (5.8).

5.1.2 The extent to which the factors contribute to the total uncertainty of measurement differs considerably between types of analyses. ESC takes into account these factors in developing analytical procedures, in the training and qualifications of personnel, and in the selection and calibration of the equipment utilized.

5.2 PERSONNEL

5.2.1 General Personnel Management

ESC management ensures the competency of all who operate specific equipment, who perform analyses, and who evaluate results and approve data reports. Approved signatories for support documents and final reports are kept by the Regulatory Affairs Department and, likewise, documents are maintained within each analytical department for the analysts. Personnel performing specific tasks are qualified on the basis of appropriate education, training, experience, and/or demonstrated skills, as required.

5.2.2 Training

All training and education requirements are outlined in SOP# 030205, *Technical Training and Personnel Qualifications*. Training requirements for safety and health are listed in the *Chemical Hygiene and Laboratory Safety Plan.* When staff members undergo training, adequate and appropriate supervision by fully trained analysts is provided.

5.2.2.1 Corporate Documents

All employees are required to read relevant corporate documents. At a minimum this includes:

- ESC Policy Manual
- ESC QA Manual
- Chemical Hygiene and Laboratory Safety Plan
- SOPs (As specified/required for work area)

Records of verification are required for each individual and are retained on file for a minimum of 10 years.

5.2.2.2 Specific Documents

Analysts are also required to undergo training specific to their position. This includes the following:

- Documented review & acknowledgement of Method Specific SOPs
- Documented review & acknowledgement of published methods related to the specific SOP
- Documented review & acknowledgement of other supporting methods related to the specific determinative SOP
- Certification Statement of acceptable performance of an Initial Demonstration of Capability (according to method criteria)
- Continuous acceptable performance on daily/batch control samples
- Performance Testing, where required, is reviewed as continued verification of analyst proficiency.
- Educational/training courses are provided where required by the position.
- Certification Statement of acceptable performance of a Continuing Demonstration of Capability (according to method criteria)

Records of verification are required for each individual and are retained on file for a minimum of 10 years.

5.2.2.3 Routine Training

Any routine training and re-training necessary for a person to perform a particular job effectively is specified in job descriptions, process procedures, maintenance procedures, etc., as appropriate.

5.2.2.4 Special Training

Special training required as a result of new technologies, contracts, expanding markets, company-wide improvement programs, new method development, etc. is conducted as the need arises.

5.2.2.5 Annual Training

An annual training plan is established by management and in conjunction with regulatory requirements. The plan is maintained by the Regulatory Affairs Department, which specifies details of the training to be carried out in each department to permit effective implementation of the management system. Managers ensure that the plan is implemented within their areas of responsibility.

5.2.3 General Responsibilities

See Organization Chart in Section 4.0 for more detailed information regarding company organizational structure.

Chemist/Analyst:

- Performs sample analyses
- Verifies detail and accuracy
- Records pertinent information in laboratory notebooks
- Stores all data (files and discs)
- Updates QC charts – where applicable
- Prepares and completes benchsheets/raw data for review

Laboratory Director:

The Laboratory Director is responsible for all operational laboratory activities. The Laboratory Director must approve the *Quality Manual*.

Laboratory Group Leader, Department Manager:

Day to day supervision of technical laboratory operations is the responsibility of these leaders who are full-time members of the staff and who assure reliable data through the following activities: monitoring quality control, corroborating the analysis performed, and approving demonstrations of capability. Additionally they certify that personnel with appropriate educational and/or technical background perform all analyses for which the laboratory is accredited. The laboratory group leader or supervisor oversees analytical raw data, ensures calculation/calibration correctness, and reviews instrument and sample preparation logs.

Laboratory QA Officer (Also called QA Manager)

The QAO has the authority and responsibility for ensuring that the quality system is implemented and followed. The QAO has direct access to the Laboratory Director and is independent of operations.

The QAO routinely reviews QA/QC policies for all analyses to ensure that the data is evaluated within method requirements. The QAO is also responsible for assessing data that is out of compliance and ensuring that necessary corrective action measures are taken and are effective.

***Laboratory QC Manager* (Also called QC Officer, QCO)**

The QCO shares the authority and responsibility for ensuring that the quality system is implemented and followed. The QCO has direct access to the Laboratory Director and is independent of operations.

The QCO routinely reviews QC policies for all analyses to ensure that the data is evaluated within method requirements. The QCO is also responsible for data review and is responsible for ensuring method/program compliance and that necessary corrective action measures are taken, completed, and remain effective.

QC Specialist (QCS)

Each ESC Analytical Division employs the use of a QC Specialist (QCS). This individual has analytical experience in their assigned area and reports to the QCO. Working knowledge of the instrumentation, printouts, and processes is key to successful approval of data being generated in that area. The QCS gives final approval of the initial raw data. The QCS is responsible for the review of data for method compliance. In addition, the application of qualifiers is verified and approved. If the QCS determines a result to be questionable, the data is given to the Department Manager to initiate appropriate action based on the severity of the problem.

Technical Specialist

Technical Specialists are a part of the Regulatory Affairs Department. These individuals have comprehensive experience in their areas of expertise. The Technical Specialist may be called upon for data interpretation, where compliance issues arise. In addition, these individuals often interface with the clients where questions arise concerning methods, data interpretation, and recommendations concerning alternate analyses.

Technical Service Representative (TSR)

The TSR is responsible for final report review. Once the data has completed the laboratory validation steps, the final report is generated. The TSR reviews the data for completeness and any outstanding anomalies. If an error is suspected, the report is delayed until the appropriate Department Managers can be contacted to resolve the question. Each TSR has laboratory experience in one or more departments.

LIMS Specialist

The LIMS Specialist tracks internal sample custody, computerizes data, and stores it in the LIMS system.

5.2.4 Job Descriptions

Employee qualification requirements are maintained by the Human Resources Department and are facilitated through the use of written job descriptions. Educational requirements and experience are included in the job description. The Department Manager determines specific education and experience requirements for individual positions based on the particular department need.

5.2.5 Training Records

Details of any employee training performed at ESC are recorded on training records. Procedural training records are maintained within each department, while policy records are maintained by Human Resources. Training on new or revised Standard Operating Procedures is maintained with the Master copy of the procedure by the Regulatory Affairs Department.

5.3 ACCOMMODATION & FACILITY DESIGN

5.3.1 Laboratory Facilities

The design of the laboratory supports good laboratory practices and does not adversely affect measurement integrity.

5.3.2 Environmental Conditions

All ESC laboratory facilities, analytical areas, energy sources, lighting, heating, and ventilation facilitate proper performance of calibrations and tests. The laboratory ensures that dust, electromagnetic interference, humidity, line voltage, temperature, sound and vibration levels are appropriately controlled for specific measurement results and do not adversely affect accuracy or increase the uncertainty of each measurement.

Environmental conditions are recorded on all data sheets, when monitoring is required. The laboratory documents deviations and corrective actions when environmental conditions are not within specified conditions.

Environmental conditions maintained by the laboratory, listed in Section 5.3.5 are within the limits recommended in **ANSI/AIHA Z9.5-2003**. Measurements are not made if environmental conditions deviate from those stated.

Laboratory staff ensures adequate conditions in the facility using the steps listed below:

- Verify that air conditioning, lighting, heating, and ventilation are controlled and monitored.
- Maintain good housekeeping practices to promote a clean, uncluttered laboratory.
- Have sufficient space to minimize the risk of injury to staff and/or damage to standards or equipment
- Maintain a convenient and efficient work environment with effective separation of incompatible activities.
- Limit the amount of paper products used or stored in sensitive and/or clean areas to prevent dust contamination.

5.3.3 Separation of Incompatible Activities

The ESC complex facilitates the physical separation of analytical activities to prevent possible contamination between departments.

Each laboratory structure is specifically designed for the type of analytical activity that it contains. The air handling systems, power supplies, and gas supplies are specific for each laboratory department.

The following areas are designated and maintained under proper conditions and security:

- Sample Receiving
- Sample/supply shipping
- Chemical Storage
- Waste storage/disposal
- Data Handling
- Data Archiving

Routinely, the departments are required to maintain cleanliness and exercise good housekeeping measures to further minimize potential for contamination that could adversely affect analytical processes.

5.3.4 Facilities Access Management

Entrance into any ESC building requires an electronic ID badge with appropriate assigned access. Access is controlled to each area depending on the required personnel, the sensitivity of the operations performed, and possible safety concerns. Chemical/receipt and storage is assigned to the purchasing department and is access controlled by an attendant who organizes and maintains the inventory.

5.3.5 Good Housekeeping

ESC ensures good housekeeping practices in all facilities to maintain a standard of cleanliness necessary for analytical integrity and personnel health and safety. Some areas are periodically monitored to detect and resolve specific contamination and/or safety issues**.**

5.4 TEST METHODS AND VALIDATION

Method Validation is the confirmation by examination and the provision of objective evidence that the particular requirements for a specific intended use are fulfilled.

5.4.1 General

5.4.1.1 ESC uses appropriate methods and procedures for all analyses within its scope. These include sampling, handling, transport, storage and preparation of items to be analyzed and/or calibrated, as well as statistical techniques for analysis of data and, where appropriate, an estimation of the associated measurement uncertainty.

5.4.1.2 ESC has instructions on the use and operation of all relevant equipment and on the handling and preparation of items for analysis, where the absence of such instructions could jeopardize the results. All instructions, standards, manuals and reference data relevant to the work of the laboratory are maintained current and be made readily available to personnel (see 4.3).

5.4.1.3 Deviation from methods occur only if the deviation has been documented, technically justified, authorized, and accepted by the client.

5.4.2 Selection of Methods

5.4.2.1 The laboratory uses analytical methods, including methods for sampling, which meet the needs of the client and are appropriate for the analyses performed. Methods utilized are preferably those published as international, regional, or national standards. The laboratory ensures that it uses the latest valid edition of a method unless it is not appropriate or possible to do so or unless regulatory requirements dictate specific revision use. Methods are supplemented with Standard Operating Procedures that list additional details to ensure consistent application.

Where mandated, only approved procedures are used. ESC utilizes a number of method sources to accomplish project requirements. See Section 2.1.3 for a list of method references.

5.4.2.2 When the client does not specify the method to be used or if a client selects an inappropriate or out of date method, the laboratory selects appropriate and approved methods that have been designated by the project regulatory program. The client is informed as to the method chosen and client confirmation is required.

5.4.3 Laboratory Developed Methods

5.4.3.1 Introduction of analytical methods developed by the laboratory for its own use is a planned activity and is assigned to qualified personnel equipped with adequate resources.

5.4.3.2 Plans are updated as development proceeds and effective communication is maintained with all personnel involved in the development process.

5.4.4 Non-Standard Methods

5.4.4.1 When it is necessary to employ methods not covered by approved industry standard methods, these are subject to agreement with the client and must include a clear specification of the client's requirements and the purpose of the analysis. The method developed must be validated appropriately before use.

5.4.4.2 For new analytical methods, procedures are developed prior to the analysis and contain at least the following information:

- appropriate identification
- scope
- description of the type of item to be analyzed
- parameters or quantities and ranges to be determined
- apparatus and equipment, including technical performance requirements
- reference standards and reference materials required
- environmental conditions required and any stabilization period needed
- description of the procedure, including:
  - affixing identification marks, handling, transporting, storing and preparing of items,
  - checks to be made before the work is started,
  - verifying equipment function and, where required, calibrating and/or adjusting the equipment before each use,
  - method of recording the observations and results
  - any safety measures to be observed;
- criteria and/or requirements for approval/rejection;
- data to be recorded and method of analysis and presentation;
- uncertainty or procedure for estimating uncertainty.

5.4.5 Validation of Methods – ESC SOP #030211, *Method Validation*

5.4.5.1 Validation Description

Validation is process of confirmation by examination and the provision of objective evidence that the stated requirements for a specific method/procedure are fulfilled.

5.4.5.2 Validation Summary

The laboratory validates all methods, including the following: EPA, NIOSH, OSHA, and program mandated methods, approved methods used outside their intended scope, non-standard methods and amplifications, and modifications of approved methods to confirm that the methods are fit for the intended use. The validation is as extensive as is necessary to meet the needs in the given application or field of application. The laboratory records the results obtained, the procedure used for the validation, and a statement as to whether the method is fit for the intended use.

5.4.5.3 Validation for Client Need

The range and accuracy of the values obtainable from validated methods (e.g. the uncertainty of the results, detection limit, selectivity of the method, linearity, limit of repeatability and/or reproducibility, robustness against external influences and/or cross sensitivity against interference from the matrix of the sample.) are assessed for the intended use as relevant to the clients' needs.

5.4.5.4 Limits

Descriptions of analytes, preparative and analytical methods, matrices, accuracy and precision targets, and MDLs and RLs are presented in the QAM Appendices.

Method Detection Limits (MDLs) – 40CFR, Part 136, Appendix B - SOP# 030206, *Method Detection Limits*

All detection limits are comparable to those established by the EPA and are not typically lower than recommended detection limits. To determine whether the EPA detection limit is being achieved, an MDL study is performed according to 40 CFR Part 136, Appendix B. The standard deviation of, at least,seven standards at or near the expected detection limit is calculated. MDLs are determined such that the risk of reporting a false positive is less than 1%. The method detection limit (MDL) is calculated as follows:

$$MDL = TS$$

where: S is the Standard Deviation of replicate measurements and
T is the value of Student’s T for n-1.

If the MDL is higher than the EPA-method-suggested MDL, the calculated value is used as a basis for establishing the reporting limit (RL) for reporting. MDLs are re-calculated on an annual basis or sooner if a material change in the instrumentation or method is enacted, or a change in the calibration response factor is noted. Additional studies may also be conducted to enhance the program.

Published MDLs may be set higher than experimentally determined MDLs to: 1) avoid observed positive interferences from matrix effects or common reagent contaminants or 2) for reporting convenience (i.e., to group common compounds with similar but slightly different experimentally determined MDLs).

Reporting Limits (RLs)

Reporting Limits (RLs) are typically set 3 - 10 times the standard deviation calculated in the MDL process listed above. Because reporting level checks are required, ease of preparation of commercial analytical mixes may dictate, to some extent, the reported RL. Generally, the RL is not set at less than 3 times the MDL. The final RL is determined based on the matrix, method, and analyst experience. RLs are verified daily using a calibration standard at a level equal to or less than the established RL.

ESC – Practical Detection Limit

Where necessary, ESC uses in-house protocol to determine a practical and real number for method detection. This is not a statistically derived number. It is a verified number that is validated using a 20% coefficient of variation. Signal to noise ratios and baseline behaviors are assessed and considered for each instrument type. Instrument performance is assessed based on the lowest possible detectable concentration that is 3X above the noise level. A series of samples are prepared at the determined level, using the method protocol. The samples must perform within a 20% coefficient of variation. The lowest concentration that meets the criteria is the Practical Detection Limit. This determination either confirms or replaces the MDL as determined using 40CFR Part 136.

5.4.5.5 Demonstration of Capability

Initial and Continuing Demonstration of Capability (IDOC & CDOC) (General Testing Other Than Environmental Lead)

NOTE: All IDOC & CDOC records are kept on file by the laboratory. Supporting data is filed with each demonstration. Completion is recorded on the form found in the NELAP Standard Appendix C. Records of verification are required for each individual and are retained for a minimum of 10 years.

General Requirements:

- A DOC is performed for each analyte whenever the method, analysts, analytes, or instrument type is changed.
- The Department Supervisor certifies that technical staff members in their area of expertise are trained and authorized to perform all analyses for which the laboratory is accredited by signing the DOC form. The QA department is the final approval of all IDOCs and CDOCs
- More specific information can be found in SOP# 030205: *Technical Training and Personnel Qualifications*

IDOC

An initial demonstration of capability (IDOC) must be made prior to using any analytical method, at any time there is a significant change in instrument or method, and when a new analyst is trained. An analyst can achieve the IDOC requirement for a specific method by using sample spike results. The following guide is a general outline of the IDOC requirements:

- A quality control sample is obtained from an outside source. If not available, the QC sample may be prepared by the laboratory using stock standards that are prepared independently from those used in instrument calibration.
- The analyte(s) is diluted in a volume of clean matrix sufficient to prepare four aliquots at the concentration specified, or if unspecified, to a concentration approximately 10 times the method stated or laboratory-calculated method detection limit.
- At least four aliquots are prepared and analyzed according to the method either concurrently or over a period of days.
- Using all of the results, calculate the mean recovery (x) in the appropriate reporting units (such as µg/L) and the standard deviations of the population sample (n-1) (in the same units) for each parameter of interest. When it is not possible to determine mean and standard deviations, such as for presence/absence values in micro and mold analyses, the laboratory must assess performance against established and documented criteria.

- Compare the information from above to the corresponding acceptance criteria for precision and accuracy in the published method. If no method criteria exist, the IDOC performance must be compared to in-house QC limits for laboratory control samples (LCS). Where appropriate, limits may be compared to the criteria listed in DOD QSM. If all parameters meet the acceptance criteria, the analysis of actual samples may begin. If any one of the parameters does not meet the acceptance criteria, the performance is unacceptable for that parameter. The analyst completes further training before attempting the IDOC process again.

CDOC

Continuing Demonstration of Capability (CDOC) are performed at least annually by documentation that technical personnel have read, understood and agreed to perform the most recent version of the analytical method (the approved method or standard operating procedure) and documentation of continued proficiency by at least one of the following once per year:

- Acceptable performance of a blind sample (single blind to the analyst);
- Another demonstration of capability using at least four consecutive laboratory control samples with acceptable levels of precision and accuracy
- Successful analysis of a blind performance study sample

Initial and Continuing Demonstration of Capability (IDOC & CDOC)
(Environmental Lead Only)

IDOC

Analysts/Technicians in training complete a minimum of four independent test runs of sample preparation and/or instrumental analysis. Independent runs are defined as analytical runs consisting of at least five known samples, one of which is a certified reference material or proficiency testing material, separated by a period of time sufficient to evaluate the testing material.

- Sample Preparation and Analytical Personnel - the recoveries of the associated reference materials or proficiency training samples for each run must be within ±10% of the certified value, 75% of the time.

NOTE: The reference/proficiency test samples utilized are: 1) similar to matrices the analyst encounters during routine sample analysis, 2) cover the sample mass range for which the analytical SOP has been designed and 3) cover the Lead (Pb) concentration for which the analytical SOP has been designed. In cases where there are several matrices of potential concern, four independent runs are not be sufficient to provide adequate demonstration of performance.

CDOC

Annual demonstrations are performed by Analysts/Technicians involved in Lead (Pb) analyses to showed continued ability to adequately analyze samples for Lead (Pb) based on standard reference materials (SRMs) or certified reference materials. This demonstration is done at a minimum of every six months and can be a part of the analysis of proficiency testing materials or quality control samples associated with routine sample runs.

5.4.6 Measurement Uncertainty - ESC SOP# 030221, *Measurement of Uncertainty*

5.4.6.1 Uncertainty Definition

Uncertainty is defined as a variable associated with the result of a measurement that characterizes the dispersion of the values that could reasonably be attributed to the measurement type. This definition of uncertainty focuses on the range of values that is relevant to the analytical technique being utilized for the analysis of field samples.

The uncertainty of testing results are calculated and documented in accordance with the requirements of ISO 17025 Clause 5.4.6. The Estimation of Uncertainty of Measurement Procedure is applied to all in-house analytical methods, where practical. The uncertainty of measurement determination is also required of all ESC subcontractors.

5.4.6.2 Uncertainty Procedure

The Estimation of Uncertainty of Measurement Procedure is applied for estimating uncertainty of measurement, except when the analytical methods preclude such rigorous calculations. In certain cases it is not possible to undertake metrologically and statistically valid estimations of uncertainty of measurement. In these cases the laboratory attempts to identify all the components of uncertainty and make the best possible estimation, and ensure that the form of reporting does not give an exaggerated impression of accuracy. Reasonable estimation is based on knowledge of the performance of the method and on the measurement scope, and makes use of previous experience and validation data.

The degree of rigor needed in an estimation of uncertainty of measurement depends on factors such as:

- Requirements of the method
- Requirements of the client
- The existence of narrow limits on which decisions on conformance are based

In practice the uncertainty of the result may arise from many possible sources, including an incomplete definition, sampling, matrix effects and interferences, environmental conditions, uncertainties of weights and volumetric equipment, reference values, approximations and assumptions incorporated in the measurement method and procedure, and random variation.

In cases where a well-recognized method specifies limits to the values of the major sources of uncertainty of measurement and specifies the form of presentation of calculated results, the laboratory is considered to have satisfied the estimation uncertainty of measurement by following the method and reporting instructions (see section 5.10).

5.4.6.3 Uncertainty Determination

Where possible, ESC utilizes data from Laboratory Control Samples (LCS) to determine the minimal uncertainty estimates in each matrix. LCSs are matrix dependent and are consistent representatives of the method effects on the particular matrix of choice. Uncertainty is determined per analytical technique, where applicable, and is performed using a population of 50 or more data points. Since the uncertainty is essentially constant, for each method, across a given matrix, ESC's method of choice is to determine uncertainty at the 95% confidence interval.

Procedure Summary:

- Select a group of representative data, from a single matrix. Data set must be 50 individual measurements or greater.
- Determine the relative standard deviation of recovery data
- Calculate the expanded uncertainty as two times the relative standard deviation

5.4.6.4 Uncertainty Results

ESC does not report uncertainty measurements on the final report. However, uncertainty determinations are available for review, when specifically requested for a project. The measurements are only applicable to the specific analytical procedure and matrix. No effects of sampling activities or related processes are considered in this determination.

5.4.7 Control of Data

5.4.7.1 Transfer Checks

Calculations and data transfers are subject to appropriate checks in a systematic manner.

5.4.7.2 Automated Acquisition

When computers or automated equipment are used for the acquisition, processing, recording, reporting, storage or retrieval of data, the laboratory ensures that:

- computer software developed by the user is documented in sufficient detail and suitably validated as being adequate for use
- procedures are established and implemented for protecting the data; such procedures includes, but not be limited to, integrity and confidentiality of data entry or collection, data storage, data transmission and data processing
- computers and automated equipment are maintained to ensure proper functioning and are provided with the environmental and operating conditions necessary to maintain the integrity of data.

5.4.7.3 Commercial Software

Commercial “off the shelf” software, e.g., word processing, database and statistical programs in general use within its designed application range may be considered sufficiently validated. However, laboratory software configuration/modifications are validated as in 5.4.7.2.

5.4.7.4 ESC Software Systems

Table 5.4.7.4a LIMS

System	Description
LIMS	The LIMS is a computerized database for data management. Access to the system is protected by coded password and access is granted based on user need.
Security	Level 1. Login, lookup sample status, generates worksheets. General access, every station has access. Level 2. Enter data, proofread and change data. The data entry person has access to this level. Level 3. Review and validate data, generate reports. Access is limited to the TSR, lab supervisors and QA. Once data is approved in the LIMS, it cannot be altered. Only the status of the sample may be changed to either "reported" or "invoiced."
Hardcopy Records	• Login summary - includes all information on sample and requested analyses • Lab preparation preview and benchsheets for digestions, extractions • Lab assignment/benchsheets to generate work assignments and record data • Data approval reports • Final reports for clients • QA summary
Hardcopy Records	All paper records are retained by ESC. As the pages become historical (prior to the current working range of log numbers), they are removed from the logbook, prep book, or workbook in sequential order and permanently bound for storage in banker's boxes. The Lab Support Supervisor maintains a log of numbered boxes and their contents. They are cross-referenced by sample log number, date and storage number.

Table 5.4.7.4a LIMS	
System	**Description**
Data Records	*Data* is available on electronic media. *Revisions* to the LIMS software are documented within the code. Each revision indicates the change in function, programmer's initials, and date of change. Programming has limited access and is accessible only by approved individuals through the use of passwords.
Manual Data Entry (verified by 4-step system)	• The section supervisor first approves raw data. • The data entry portion of the LIMS can only be accessed by authorized individuals, therefore allowing limited access to protect the integrity and maintain the confidentiality of the data. • The data entry person and a qualified laboratory analyst then proofread each group of entered data. • When all results for a sample are complete, a report is printed and examined by a Technical Service Representative for final approval.
Calculations	All calculations performed by the LIMS are approved and submitted by the Laboratory Supervisors. Each calculation is tested parallel to manual calculations to ensure proper function.
Automatic Data Transfer	Data is transferred electronically from instrumentation directly to the LIMS. Once the data has been transferred, it undergoes a screen review. The data is then printed and reviewed again. If data needs to be changed, a data entry specialist changes it and a hardcopy is printed of the final data.

Table 5.4.7.4b AUXILIARY SOFTWARE	
System	**Description**
Auxiliary	Auxiliary Computer and Software Used to Generate and Validate Data
General	Several instruments have their own dedicated single computer and manufacturer-designed software to run them. Instruction manuals and other documentation provided by each manufacturer are maintained. ESC receives updates as they become available from the manufacturer. All raw and filtered data is stored on media (with uniquely titled data files on floppy discs) and all associated printouts and paperwork is filed. The original raw data is not accessed again unless it is subjected to uncertainty.
Method Files	Creation of any method or analyte files, necessary to run the appropriate analyses is the responsibility of the group leader. The lab supervisor verifies that the compounds, wavelengths, retention time windows, calculation criteria, and other relevant parameters are correctly input into the specific method file. Analysts may only use the method files that have been specifically generated by the group leader.
Supplier Info	All purchased software that is used in conjunction with software specific instruments is guaranteed by the supplier to function as required. The supplier of the software performs all troubleshooting or software upgrades and revisions.
Validation	Computer software is validated for proper performance. The result of the validation is recorded, when in-house programming is the source of the calculation.

5.5 EQUIPMENT

5.5.1 Usability

Laboratory standards, equipment, and associated apparatus are suitable for the validation of acceptable performance of analyses and are maintained in accordance with this quality manual to include protection from dirt, dust, corrosion, and other causes of deterioration. Laboratory personnel investigate any equipment or standards, which are suspect in contributing to out-of-control conditions. Records of corrective actions for discrepancies are maintained in the laboratory (see Section 4.11).

5.5.2 Calibration of Equipment

5.5.2.1 To maintain the integrity of standards, all maintenance operations are performed according to documented procedures and the laboratory standards are:

- Selected for use according to the level of precision, accuracy, and uncertainty required
- Limited in access and use, to trained and authorized laboratory staff only
- Handled and safely stored according to method requirements

5.5.2.2 Primary standards, directly traceable to NIST standards, are obtained from a vendor approved by the A2LA or NELAP and all certificates of analysis are maintained on file in the laboratory.

5.5.2.3 Secondary standards are also obtained from a vendor approved by the A2LA or NELAP and all certificates of analysis are maintained on file in the laboratory. They are calibrated by comparison to primary standards. Calibration reports are maintained on file in the laboratory.

5.5.2.4. Working standards are prepared from certified stock standards. Standard preparation logs are maintained electronically via the Standards Logger in the ESC LIMS.

5.5.2.5 Support Equipment Calibration: Including, but is not limited to: balances, ovens, refrigerators, freezers, incubators, water baths, temperature measuring devices, volumetric dispensing devices, and thermal/pressure sample preparation devices. All support equipment is maintained in proper working order and records are kept of all repair and maintenance activities, including service calls.

5.5.2.6 Equipment used with nominal values and corrections is verified by calibration labs having ISO 17025, or other suitable, accreditation. A calibration interval is established for the equipment (i.e., environmental equipment, balances).

5.5.2.7 Calibration of equipment is conducted at a frequency to ensure that the equipment remains in tolerance during its use in the laboratory. Frequency of calibration is based on a review of calibration, maintenance, and repair history. Reviews are conducted by the Department Manager and records are maintained.

5.5.3 Equipment Operation and Maintenance – See Table 5.5.3.3 for General Information

5.5.3.1 ESC's preventative maintenance program provides guidelines to ensure that every effort is made to keep equipment well maintained and prepared for the next project. Most equipment is kept in duplicate and spare parts are kept in stock. Instrument/equipment manuals are kept in each department for quick reference to aid in problem diagnosis. ESC maintains service contracts on major laboratory equipment, so that in the event of failure, repairs can be made within a few days. The appropriate Department Manager is consulted if an instrument repair is required. If a solution to the problem is not found immediately, a call may be placed to the instrument manufacturer or maintenance support provider for assistance in diagnosing the problem, determining the extent of repair needed and a possible timeframe for repairs to be completed.

5.5.3.2 If analyses are scheduled and it appears that the equipment may be down for a longer period, ESC arranges for analyses to be performed by another qualified lab. This action is utilized if client required definite turnaround time or sample holding times would be exceeded.

5.5.3.3 General Equipment (All Labs)

If method calibration requirements for a particular procedure are more stringent than those listed here, they are followed when that procedure is performed.

Table 5.5.3.3a General Equipment Calibration			
Equipment	**Activity**	**Frequency ***	**Record Type**
Balances	Verified with Class I NIST traceable weights when used	Before use	Logbook – Located in each respective lab
Balances	• Clean • Check alignment • Service Contract Top-loading balances are allowed a tolerance of ±1%, while analytical balances are allowed a tolerance of ±0.1%.	Semi-annually under a service contract.	Certificates from contractor.
Weights – Class I	• Only use for the intended purpose • Use plastic forceps to handle • Keep in case • Store in desiccator • Re-calibrate	Checked for accuracy by an external source, annually, or sooner if necessary.	Certificates from contractor.
pH meters and probes	Calibration: • pH buffer aliquot are used only once • Buffers used for calibration bracket the pH of the media, reagent, or sample analyzed. • Check must perform within 0.05 pH units. Temperature correction is performed either automatically by the instrument or manually depending upon the instrument used.	Before use	Calibrations are recorded in a logbook.
Automatic pipettes	Verify for accuracy and precision using reagent water and analytical balance	In-house – Monthly Contract – Semi Annually Tolerance is set at 2.0%, (ASTM standard = 3%).	Monthly verifications are recorded in a logbook. Semi-annual cal. is verified by certificates from the cal. service.
Refrigerators, Freezers, Hot plates and BOD incubators	• Thermometers are immersed in liquid to the appropriate immersion line • The thermometers are graduated in increments of 1°C or less	Temperatures are recorded each day in use	Logbook
Ovens	• Thermometers are immersed in sand to provide even measurement • The thermometers are graduated in increments of 1°C or less	Temperatures are recorded each day in use	Logbook

Table 5.5.3.3a General Equipment Calibration			
Equipment	**Activity**	**Frequency ***	**Record Type**
Thermometers	ESC NIST certified thermometers	Calibrated annually by a NIST calibration service, accredited to ISO/IEC 17025 and ANSI/NCSL Z540-1.	Calibration certificates from the calibration service.
DO Meter	Calibrated according to manufacturer's specifications. Using the recorded temperature and barometric pressure the meter is calibrated to the air saturation of dissolved oxygen using a conversion chart provided by the manufacturer.	Before use	Calibration of each meter is recorded in a separate logbook.
Specific Conductivity Meter	The conductivity meter is calibrated according to manufacturer's specifications. Temperature correction is performed either automatically by the instrument or manually depending upon the instrument used. • Biomonitoring, potassium chloride with a conductivity value of 100 and 1000 umhos/cm is used as the calibration standard. • Wet Lab, potassium chloride with a value of 1413 umhos/cm is purchased from NSI for calibration purposes.	Before use	Calibration of each meter is recorded in separate daily logbooks.
Fume Hoods	Check semi-annually and must meet the OSHA minimum recommended face velocity of 60 – 100 fpm.		Recorded in Logbook

Table 5.5.3.3b Class 1 Weight Tolerance				
Value	**ASTM Class 1 Tolerance**	**Unit**	**ASTM Class 1 Tolerance**	**Unit**
1mg	0.01	mg	0.00001	g
2mg	0.01	mg	0.00001	g
3mg	0.01	mg	0.00001	g
5mg	0.01	mg	0.00001	g
10mg	0.01	mg	0.00001	g
20mg	0.01	mg	0.00001	g
30mg	0.01	mg	0.00001	g
50mg	0.01	mg	0.00001	g
100mg	0.01	mg	0.00001	g
200mg	0.01	mg	0.00001	g
300mg	0.01	mg	0.00001	g
500mg	0.01	mg	0.00001	g
1g	0.034	mg	0.000034	g
2g	0.034	mg	0.000034	g
3g	0.034	mg	0.000034	g
5g	0.034	mg	0.000034	g
10g	0.05	mg	0.00005	g
20g	0.074	mg	0.000074	g
30g	0.074	mg	0.000074	g

5.5.4 Identification of Equipment

Each item of equipment is uniquely labeled, marked or otherwise identified. Maintenance and calibration records for equipment and standards are maintained.

5.5.5 Records of Equipment

Equipment lists are department specific and are found in the associated appendices to the QA Manual.

5.5.6 Equipment Handling, Storage, Use, and Maintenance

All laboratory equipment is maintained, stored, and used in accordance with manufacturer's instructions. Operation manuals and instructions for proper maintenance of equipment are available to the staff and located in the laboratory.

Equipment is used or operated only when in a safe and reliable condition, by personnel who have been trained and are qualified. User instructions are available.

Table 5.5.6 - GENERAL PREVENTATIVE MAINTENANCE	
Type	***Description***
Glassware	Routine laboratory glassware is washed in a non-phosphate detergent and warm tap water. Before washing, all writing and large deposits of grease are removed with acetone. Glassware is then rinsed with: tap water, "No Chromix" solution, tap water, and deionized (DI) water. Glassware is stored in designated drawers or on shelves, inverted if possible. All organic glassware is rinsed with the required solvent, prior to use. Inorganic glassware is rinsed with DI water prior to use, which is a precaution against airborne cont.
Logbooks	Maintenance logs are kept on all major laboratory equipment. The logbook is updated and signed when maintenance is performed (i.e., new rings, column or septum change, etc.). Maintenance logbooks are located in the immediate area of the instrument. All preventive maintenance is noted either in the maintenance logbook or in the runlog notebook. At a minimum, all maintenance logs contain the following: • All entries in the maintenance logs must be initialed and dated by the person performing the maintenance. • All maintenance logs must be bound and paginated. • All pages of the maintenance logs must have “ESC” at the top of page. • The instrument ID number, or serial number. • Make and model of the instrument. • Date of installation, or the date the instrument was put in service (if available). • Condition of the instrument when installed (new or used) • A unique number for each notebook
Service Records	Maintenance that requires a service call from the vendor should contain the following: • Must state details when the problem began, and what the problem was. • When a service call was placed. • When the service engineer came to repair the instrument. • When the problem was solved. • How the problem was solved. To verify that the instrument is running properly after service has been performed, recalibrate and analyze QC samples before the service engineer leaves.
Additional Records – Misc. Monitoring	Additional records are kept, updated and signed when technicians are assigned to perform the following tasks: • Monitor laboratory devices such as air compressors, vacuum pumps, heaters, etc., to ensure that they are properly lubricated and in good working condition. • Monitor on a daily basis: general lab QC areas, such as BOD incubators, temperature, drying ovens, desiccators, deionized water, sample cooler temperature, etc., and record appropriate parameters in the assigned QC logbooks. • Monitor the supply and quality of purchased chemicals, reagents and glassware, and keep inventory at established levels. All chemicals are dated in relation to receipt and date opened.

5.5.7 Equipment Out of Service

When equipment is found to be in unacceptable condition or has been subjected to overloading or mishandling or if an instrument gives suspect results or has been shown by verification or otherwise to be defective, the equipment is clearly marked as out-of-service. Only the analyst responsible for the repair, or the Department Manager, can place equipment back in service. Once repaired and validated by calibration, verification, or other appropriate reviews, and found to perform satisfactorily, the equipment can be placed back in service. The laboratory examines the possible effect of defective equipment on any previous calibrations.

5.5.8 Status of Calibration

When appropriate, each item of equipment is labeled, marked, or otherwise identified to indicate its calibration status.

All equipment used with nominal values and corrections is labeled indicating the calibration status. Examples of this equipment include thermometers, calibration weights, and balances.

5.5.9 Equipment Returning to Service

When for any reason, equipment goes outside the direct control of the laboratory, the laboratory ensures that the function and calibration status of the equipment are checked and shown to be satisfactory before the equipment is returned to service.

5.5.10 Calibration Checks

Analytical instruments are calibrated per method requirements. Balances and temperature-indicating devices are verified semiannually. Records are maintained as quality assurance documents.

5.5.11 Calibration Factors

Where calibrations give rise to a set of correction factors, the laboratory has procedures to ensure that copies (e.g., in computer software) are correctly updated.

5.5.12 Safeguarding of Equipment Integrity

Analytical and supporting equipment is protected from inadvertent adjustments that could affect the integrity of the laboratory results. Instruments are located in access-protected areas. Software is tested and approved before use. Spreadsheets used in the calculation of analytical results are tested, approved, and locked before being placed into service.

5.6 MEASUREMENT TRACEABILITY

5.6.1 Policy (See SOP# 030202, *Receipt and Records of Stock, Intermediate, and Working Standards)*

5.6.1.1 Standards and equipment significantly affecting the measurement integrity of analyses conducted by the laboratory are monitored for stability as part of the measurement control program. Standards and equipment are calibrated and/or verified before use to ensure acceptable performance. Any standard or equipment that appears unreliable or has exceeded the calibration interval is evaluated and/or removed from service.

5.6.1.2 When standards, reagents, or other certified consumables are received, they are assigned a unique number. The number is recorded in the LIMS Standards Logger with other important information concerning receipt date, supplier, expiration date, description, and volume. The number is then placed on the item and the Certificate of Analysis. The Certificate of Analysis is maintained electronically. Each item is dated upon opening. Each laboratory appendix contains a list of standard sources, receipt, and preparation information. Field personnel obtain several field standards from the lab and the standards are NIST traceable.

5.6.2 Measurement Traceability

5.6.2.1 ESC has established a program of calibration and verification that is designed to ensure that the measurements made by the laboratory are documented and traceable to national standards.

5.6.2.2 To provide external evidence of traceability, the laboratory participates in measurement control programs, such as proficiency tests, and other interlaboratory and collaborative round robins, as required (See SOP# 030212, *PT Program*).

5.6.3 Calibration/Verification

5.6.3.1 Standards (Calibration)

5.6.3.1.1 Primary standards are calibrated to the standards set forth by the National Institute of Standards and Technology (NIST) or by an ISO 17025-accredited provider.

5.6.3.1.2 Primary standards are verified by secondary standards and are monitored through the measurement control programs established in the laboratory.

5.6.3.1.3 Standards are re-calibrated if there is damage to the standards or any significant change is observed in the measurement control program.

5.6.3.2 Standards (Verification)

5.6.3.2.1 Continuous verification of standards, through the measurement control program, ensures required measurement integrity of testing and includes:

- Statistical data from check standards and/or control charts (See SOP# 030207, *Quality Control Charting and Tracking)*
- Results from interlaboratory comparisons and/or proficiency tests (See SOP# 030212, *PT Program*).

5.6.3.2.2 Measurement assurance procedures for verification of standards are maintained in the laboratory, according to the individual method SOPs.

5.6.3.3 Measuring and Test Equipment

5.6.3.3.1 Equipment used with nominal values and corrections is calibrated by calibration labs having ISO 17025 accreditation, other suitable accreditation, or mutual recognition. A calibration interval is established for the equipment.

5.6.3.4 Standard/Reagent Sources, Records, & Preparation

Standard /Reagent Selection

Standards and reagents are selected according to the method requirements. A minimum of analytical reagent grade is used when not method stated. The Laboratory Director or designee(s) makes the actual determination concerning quality and manufacturer. The purchasing agent maintains a list of approved vendors that have been evaluated and approved as suppliers of critical consumables, supplies and services that may affect the quality of environmental testing and calibration. All supplies that are directly used for analysis are inspected and verified upon arrival at the Laboratory. ESC SOP# 030210, *Materials Procurement for Analytical Processes,* details the entire procedure.

Standard/Reagent Inventory

An inventory of consumables and reagents are stocked in the individual laboratory areas. Any overstock items are kept in a controlled area, maintained by the purchasing department. Items are taken from the inventory area to the laboratories upon request.

Standard/Reagent Preparation

When standards are prepared in-house, they are weighed on an analytical balance, calibrated against Class “I” weights, diluted in Class "A" glassware, and compared against an external reference standard. The standard is marked with concentration, then signed and dated by the analyst, and placed in the appropriate storage area.

All dilutions of stock standards are prepared in Class A volumetric glassware. Where dilutions are made to volume, TC (to contain) glassware is used. All volumetric pipettes are Class A and designated as TD (to deliver). If the intermediate or working standards are to be saved and used again, the standard container is marked with concentration, date, source standard, expiration, and the analyst's initials.

All purchased stock standards are kept in a designated area within the appropriate section. Each chemical is marked in relation to date received, date opened, and expiration date.

Standard/Reagent Logbooks

A standard log is kept with each analysis book, indicating date of preparation, which standard (by lot number, if applicable) used, the amount used to prepare the solution, when it was made and expiration date or the recommended holding time. Reagents are recorded in the same manner as standards. Reagents that are prepared on a daily basis are recorded directly onto the raw data sheet. The analyst preparing the reagent initials and dates the raw data sheet. Where appropriate, an electronic LIMS Standard Logger is used in lieu of handwritten logbooks.

5.7 SAMPLING

5.7.1 Sampling Plan

When the laboratory carries out sampling of substances, materials or products for subsequent testing or calibration, it has a sampling plan and procedure for sampling. The sampling plan as well as the sampling procedure are available at the location where sampling is undertaken. Sampling plans are, whenever reasonable, based on appropriate governing methods. The sampling process addresses the factors to be controlled to ensure the validity of the analytical results.

5.7.2 Client Requirements

ESC has no jurisdiction over client deviations from any sampling plan but clients are encouraged to maintain proper records and to include appropriate information in all documents and communications.

5.7.3 Sampling Records

See Appendix III for information regarding the records of relevant field data.

5.7.4 Field Sampling - General Summary

Sample Labels

All sample labels contain the following information: Client name, project name or ID, site ID, sampling point, time collected, and date collected. In addition the label includes information regarding preservation and method assignment. The project ID number is a unique ID number that can be associated with the client overseeing the project. Clients are designated in the ESC LIMS by a unique name referred to as a COCODE. The COCODE always precedes the project ID so that ESC personnel can easily relate a project ID to a particular client. As samples are logged in, they are assigned a unique sequential number. NO login number can be used twice. When the samples are logged in, all field label information is entered. All sample information can be accessed by entering the LIMS and viewing the sample login number. ESC has the capability to access all samples with the same project ID and print a summary of the samples. All field information can be reviewed in the field notebook by date and client.

Field Notebooks

Field notebooks are an essential part of the COC. Every detail concerning the sampling event must be documented. All documentation must be written with waterproof ink. All records are signed and dated by the individuals responsible for making the entry. Errors made during the documentation process are deleted by a single line with the initials of the person who corrected it and the date made.

Crucial information to be recorded in the field notebook includes:

- Site identification
- Sample location
- Date and time of sample collection.
- Names of individual(s) collecting and documenting each sample.
- Names of all individuals present at the time of collection.
- Pertinent field conditions, including weather, site, traffic, other events, problems, etc.
- A copy of the Shipping Batch Detail Report is included as an attachment to the COC with each kit prepared and shipped.
- Specific sampling equipment used for the collection of each individual sample or sample group (Unique equipment identification numbers can be used.)

- If field analyses are performed, calibrations and results are recorded in field workbooks.
- When sampling monitoring wells, the field notes (whether in notebooks or on standard forms) must also document:
  - *Well casing composition and diameter*
  - *Water table depth*
  - *Well depth*
  - *Calculations to determine the volume of water to be purged*
  - *The total volume of water purged and how accomplished*
  - *The date and time well was purged, beginning to end*
  - *Use of fuel-powered units, bailers, etc.*
- When collecting surface water samples, the field notes must include the depth at which the sample was taken and the type of sampling equipment used.
- When water samples are collected over a period of time, it is necessary to indicate the following information in the field notes:
  - Collection beginning and ending time and date
  - Specific equipment used (manual or automatic)
  - Abnormal conditions of the sampling location
  - Safety precautions taken.

Field Chain of Custody (COC)

All field records include the signature of the person(s) responsible for the collection of the samples.

COC forms are completed and returned with the samples collected by ESC personnel. COC forms are also made available to clients collecting their own samples. A copy of the COC is retained in pdf form along with a pdf copy of the final report in the LIMS. The original is returned to the client with the final report. The COC is signed by the sampling personnel in the space referred to as "Collected by:".

A sample label is affixed to the side of each sample container before or at the time of sample collection. Pertinent information on the label includes a unique field identification number, sample description, preservative, method requested, date and time the sample was collected.

5.7.5 Field Quality Control Checks

Blanks collected in the field are considered to be specific quality control for a set of samples. Analytical data that is consequential from the blanks is used to assess the integrity of field sampling and cleaning operations. This data can be used to confirm the use of contaminant-free sample containers and preservation reagents, and/or successful equipment cleaning. Potential on-site contamination, personnel sample collection technique accuracy, and problems that may occur in sample storage and transportation may also be revealed. Field blanks are treated in the same manner as regular samples: preserved with the same reagents, stored and transported in the same containers with samples, etc. For soil or solid samples, deionized water is used for blanks in similar containers.

5.7.5.1 Field/Equipment Blanks

The purpose of field blanks is to evaluate the purity of preservation or additive reagents. Field blanks also represent the collection techniques, general sample containers to be filled, and the effects of on-site environmental conditions and possible contaminants. Field blanks are prepared at sampling locations by filling sample containers with DI water, adding appropriate preservatives or additives, sealing the containers, and completing all paperwork required for the samples. Blanks are stored in the same shipping containers with the samples for transportation back to the lab.

Field blanks are generally collected at a rate of one blank per parameter group per day, or 5% of the samples in the parameter group, whichever is greater.

Equipment blanks help measure the effectiveness of pre-cleaning and field cleaning of equipment. They are used to evaluate sources of contamination that may also be found in a trip blank. Equipment blanks are collected according to the frequency shown in Table 5.7.5. Equipment blanks are prepared by rinsing the equipment with analyte-free water in the same manner as used for sample collection. The equipment blank is placed in the appropriate containers with required preservatives, if any. Blanks must be taken and preserved, where required, for each method group. The blanks are stored in the same shipping containers as samples for transportation back to the lab.

5.7.5.2 Trip Blanks

Trip blanks are used when sampling for volatile organic compounds to evaluate the cleanliness of the sample container, purity of the blank source water, and the exposure of the sample to contaminants during storage and/or transportation to and from the laboratory. The Laboratory supplies the trip blank with the sampling kit order, according to the following:

- The trip blanks are filled with analyte-free water plus any appropriate preservatives. (Matrix specific trip blanks are provided where necessary)
- The containers are sealed, labeled, and transported to the field in the same coolers or boxes with the sample containers to be used for sample collection.
- Trip blanks are not opened in the field.
- The trip blanks must be handled in the same manner as the samples being collected and are transferred (if required) with other samples for storage and transportation to the laboratory.
- If additional blanks (field and equipment) are necessary the same source water as the trip blanks are used.
- One trip blank per parameter group per cooler are used in the sampling event.
- The client is notified if the trip blank does not return with the sample set and a nonconformance is issued.

TABLE 5.7.5.2 EQUIPMENT BLANK COLLECTION PROCEDURE FOR EACH TYPE OF SAMPLING EQUIPMENT

No. of Samples	Precleaned Equipment Blank Per Parameter Group Prior to Sample Collection	Field-Cleaned Equipment Blanks Per Parameter Group
Less than 10	1 equipment blank if no field cleaning on site; OR	1 equipment blank for field-cleaned equipment
Greater than 10	1, or 5% of equipment sets, whichever is greater	1, or 5% of equipment sets cleaned, whichever is greater

NOTE: Equipment blanks must accompany samples in the same container used for transportation.

5.7.5.3 Field Duplicates

Field duplicates are collected for each analyte group and are required whenever five or more samples are being collected. If more than ten samples are to be collected, the field duplication rate is 10%.

5.7.5.4 Field QC Check Samples

All field instruments are calibrated at the beginning of each sampling day. Calibration is checked following every 10 samples or at maximum intervals of 4 hours. Calibration is verified at the end of the day. Recalibration is required if the QC check samples do not meet calibration criteria. The pH meter is evaluated after every ten samples using a buffer different than the ones used to calibrate the meter. The conductivity meter is evaluated by measuring the performance of the standard and the result must not vary by more than 5% from the true value after applying the cell constant.

5.7.5.5 Field Duplicate Analysis

All analyses run in the field have duplicates performed at a rate of 10% of the total samples.

5.8 SAMPLE MANAGEMENT

5.8.1 Sample Management Instructions

Clients supply environmental samples from various sources/programs for analysis. ESC utilizes method SOPs and contract requirements as the instructions to properly handle and process these samples.

5.8.1.1 Holding Time Verification

- The Login Technicians are trained to recognize analyses with immediate, 24-hour, and 48-hour holding times. When short-hold samples arrive at the laboratory, the Login procedure for those samples takes place immediately. All analysts are trained to assess incoming samples for holding time limitations.
- If a sample has a holding time limitation, the LIMS issues a due date on the bench sheet to ensure that the extraction or analysis is completed within time allowed.
- In the event that a holding time is exceeded, the TSR contacts the client, informs them of the situation, and requests further direction. If instructed by the client to proceed with the analysis, a qualifier is added to the benchsheet, which is then carried on to reporting. The final report bears the explanation in the form of a qualifier.

5.8.1.2 Sample container and Sub-Sampling

- Each container displays the following information once it has been released from sample login to the laboratory: the original sample container label and the sample login label showing the sample log number.
- If the sample requires special DOT labeling, the label remains with the sample through receiving and disposal. If the sampling personnel note any special handling or precautions due to the nature of the sample, it is recorded on the sample label. The login person, at that time, makes a note in the LIMS to ensure that all departments have the information.
- The importance of sample label review is stressed to all chemists/analysts and sample handling personnel.
- When a sample is obtained for analysis the chemist records in the appropriate prep book or benchsheet the log number, the date removed, his initials, and the volume or mass of sample removed.
- Samples are mixed prior to taking sub-samples for analysis, with the exception of VOC analyses. Sub-sampling within the laboratory is performed according to SOP# 030220, *Sample Homogenization and Sub-Sampling.*

5.8.1.3 Sample Preparation

The LIMS keeps track of samples and their corresponding log numbers to be analyzed. The analysts responsible for sample preparation maintain preparatory documentation, whether organic or inorganic. The analyst asks the LIMS to generate a prep sheet for a specific prep code. The LIMS provides all samples assigned to that prep code and prints a worksheet to record the required information.

- ESC currently maintains the following prep information: wet chemistry, metal digestions, organic extractions (by method), and GC and GC/MS injection logs.
- The chemist preparing the samples, dates and initials the entry, records any non-standard procedure (e.g., an aliquot for metal digestion other than 100mL for a water sample) or unusual observation, and which samples are spiked or duplicated.
- The organic extraction prep book contains all details concerning the sample extraction procedure.
- When a preparation is complete, the chemist assigned to perform the analysis is notified and the prepped sample is placed in the appropriate holding area.
- Each extract/digestate/distillate is labeled to provide the following information: date prepped, amount prepared (volume/weight), dilutions, etc.
- The various prep books, workbooks, and injection logs document every manipulation of the sample through receipt, preparation, and analysis.

5.8.1.4 Analysis & Analysts

- Each chemist has been assigned primary analytical procedures.
- Before beginning analysis they request a Laboratory Run Preview sheet from the LIMS and receive a printed page for the specific analysis in the form of a benchsheet. This Run Preview sheet lists all sample log numbers, sample type, and due dates relating to the samples that are ready for analysis. At that time the analyst can then select "all" or choose certain samples. Once the samples have been selected they are assigned to a unique run number and are then printed to a run benchsheet.
- The benchsheet provides all necessary information to complete the analysis such as: date and initials, flask numbers (where applicable), standards ID, instrument readings, response factors, aliquots, dilutions, final results, and all QC spike and duplicate information.
- When all data is recorded and the calculations are complete, a second chemist, a QC Specialist, performs a second analytical review. If all calculations and other performance objectives pass method criteria, the second reviewer dates and initials the data and then releases the data for final reporting.
- For data that cannot be transferred electronically, a Data Entry Specialist enters the results into the LIMS. The entered results are reviewed for transcription errors against the original worksheet by a chemist. If the lab supervisor or senior chemist rejects the work, he discusses the corrective action measures with the analyst.

5.8.1.5 Laboratory Documentation

- Laboratory notebooks and related documentation are an essential part of the analytical procedure. Every detail concerning the sample analysis must be documented.
- All documentation must be written with permanent/waterproof ink. All records are signed and dated by the individuals responsible for making the entry.
- Errors made during the documentation process are deleted by a single line, with the date and initials of the person making the change. The correct result is clearly recorded adjacent to the incorrect result.

5.8.1.6 Sample Storage and Transportation

- When a Chemist completes the preparation or analysis of a sample, he returns the sample container to the Sample Custodian.
- Samples transported under the responsibility of the laboratory are done so safely and according to storage conditions.
- Specific safety operations are addressed outside of this document.

5.8.1.7 Final Reporting

- When all analyses on a sample number have been completed, the LIMS prints the final report.
- The TSR reviews the final report for discrepancies. If discrepancies are found, re-analysis may be requested.
- The TSR gives the final approval on the report and indicates approval by signature.
- Routinely, data reports are transmitted to the client through email as a PDF file. Reports are sent as PDFs to prevent alteration of the document. The hardcopy report can be mailed to the client, when necessary. Reports may also be sent to the client by fax, or via secure access through the ESC website.
- Reports that are sent electronically are protected using the latest technology available to protect the confidentiality of the results and the client.

5.8.1.8 Sample Retention and Disposal

- Samples and related extracts/digestates are retained for 45 days.
- Non-hazardous samples containing preservative are neutralized and disposed through the conventional municipal waste system.
- Non-hazardous solids are heated at 400 degrees Fahrenheit for two minutes and disposed of in a commercial waste container.
- All other waste is disposed of according to Section 6.

5.8.1.9 Sample Subcontracting

- When samples are transferred to subcontracted facility, a COC accompanies the samples. The COC contains the following required information: collection date and time, ESC login ID number, quantity and type of container, date of sample collection, and the requested analysis.
- A copy of the COC and the sub-contract lab report is filed for permanent record.
- A subcontracted analysis log records date sent, where sent, log number, analysis requested, price, date report received, and date invoice received.

5.8.2 Sample Information and Labeling

A unique sample identification number is generated for each sample and is used throughout the analytical and disposal cycle. A record of all client-supplied samples is established and maintained. The samples are stored according to published method requirements and determinative SOP.. While in storage, the client samples are stored by sample ID and analyses required.

- When samples are logged in, the information entered into the LIMS includes sample description, date and time collected, collector ID, field ID, project ID, date and time received, receiver's ID, analysis requested, specific QC requirements, type of container and preservative, sample type, due date, and remarks.
- Each sample is assigned a unique and consecutive log number. After a sample is entered into the LIMS database and assigned a specific number identifier, the LIMS login screen automatically presents the next consecutive number for logging in the subsequent sample. Log numbers are not available for reuse and cannot be altered, although descriptive information, as well as sample specific comments can be modified until the final report is issued.
- A sample label with the log number is printed by the LIMS and affixed to the sample. Each label contains a unique container ID, represents the sample ID number, and is clearly marked with preservative and requested analysis.
- Duplicate samples, collected in the field, are logged with a separate laboratory ID. Laboratory personnel are typically unaware of field duplication.
- Replicate samples with multiple analyses and containers have the same login ID number.
- The login person records the sample numbers assigned onto the COC. The LIMS provides documentation on the person authorized to enter sample log information.

5.8.3 Sample Inspection and Receipt

Any sample supplied by the client is verified upon receipt as meeting its description and being free from damage. In the event of a client sample being lost, damaged or otherwise unsuitable for use, full details of the incident are recorded and reported to the client by the Technical Service Representative via a nonconformance form, prior to any analytical action being taken. Any further action taken is at the direction of the client.

The Login Technician is responsible for sample login and assessing sample container integrity, documentation, and identification. Samples are inspected and noted for temperature, pH using narrow-range pH paper, headspace, proper container type, container integrity (broken or leaking), and volume levels. Samples requiring preservation at 4°C must arrive at the laboratory above freezing but ≤6°C. If the samples are not appropriately preserved, the problem is noted on a sample nonconformance form, the sampler is notified, and, if the lab is instructed to proceed, proper preservation is performed. The sample nonconformance sheet becomes a permanent part of the COC. Samples, which require refrigeration, are placed in a laboratory cooler immediately after login. If extractions are necessary, the laboratory supervisor is notified, via daily management reports, to ensure that holding times are not exceeded for samples, extracts, or digestates.

5.8.3.1 Sample Objectives

ESC receives samples for analysis for a variety of reasons, such as planning, estimating, process control, treatability as well as permit compliance reporting, site investigation, and remediation. When general screening is the goal of the client/project, analysis of improperly preserved or collected samples may proceed provided that the client is notified. In this instance, the chemist is notified and the proper documentation is placed onto the final report.

5.8.3.2 Sample Rejection Criteria

Where the analytical results are to be used for regulatory or compliance purposes, samples are rejected under the following conditions:

- If there is insufficient sample volume
- If the preservation and container requirements were not followed correctly
- If there is headspace in a sample collected for volatiles analysis
- If the COC is missing, incomplete, or filled out in pencil
- If the holding time for the desired analysis has expired
- If the integrity of the sample container or custody seal has been violated, if samples are broken or leaking, or if apparent contamination has occurred.
- If the temperature is outside of the method stated requirement
- If the samples are known to contain high levels of chemicals that present a health/safety risk (i.e. dioxins, radioactivity above background, etc.)

5.8.3.3 Nonconformance Issues

- If there are problems with the samples, the event details are documented on the sample nonconformance form/COC; then, the sampler and/or client is notified.
- If the client insists on proceeding with analyses, even though he has full knowledge of the possible invalidity of the sample, a qualifier detailing the problem is added in the LIMS and it is also noted on the nonconformance form.
- The TSR, affected chemists, and reporting personnel are also notified.

5.8.3.4 Login Confirmation

- On a daily basis, login confirmations are printed and auto-emailed to the client. A pdf copy is maintained in the ESC LIMS.
- A dual check is performed by Login and the Technical Service Group to insure proper analytical login from the COC.
- The original COC is forwarded to the reporting personnel to be reviewed and included with the final report.

5.8.4 Sample Storage and Handling

Client samples remain in their original packaging until analysis. Any samples that need to be dispensed or removed from their original packaging are stored in conditions that provide the same degree of protection.

Sample/Extract Storage:

- Samples, extracts, distillates and digestates have specific storage locations arranged in log number order unless rush analysis is required.
- Access to these areas is limited to authorized personnel.
- Samples are stored either in the cooler or in ambient-temperature storage, according to method preservation requirements

- Extracts, digestates, and standards are stored separately from calibration and other QC Standards in dedicated areas as follows:
  - Organic extractions for pesticides and PCBs are stored in glass vials in a designated refrigerator in the SVOC GC lab.
  - Organic extractions for SVOCs are stored in glass vials in a designated refrigerator in the semivolatile GC/MS lab.
  - TCLP extracts for metals only and metal digestates are stored in the metals lab.
  - TCLP extracts for SVOCs, pesticide, and herbicide analysis are stored on designated sample shelves in the cooler. After the extraction, the extract is stored in a designated refrigerator in the semivolatile GC/MS lab.
  - Zero headspace extracts and samples for volatiles are stored in VOC vials and segregated in a designated cooler. Where necessary, samples collected by Method 5035 are frozen.
  - Volatile standards are stored in a designated freezer in the VOC lab.
  - Pesticide and PCB standards are stored in a designated refrigerator in the SVOC GC lab.
  - SVOC standards are stored in a designated freezer in the SVOC GC/MS lab.

5.8.5 Special Requirements

The following entities mandate any required needs for special handling, storage, packaging, preservation, shipping, and marking provisions:

- EPA Approved Methods
- 40CFR Part 136.3
- 29 CFR (OSHA)
- 49 CFR (DOT)
- IATA (Dangerous Goods)

5.8.6 Sample Transportation

When a sample is received by the laboratory, the method of transportation is recorded on the COC. ESC routinely uses FED-EX, UPS, USPS, Velocity Express and various air carriers. Locally collected samples are sometimes carried in by the client collection personnel or by ESC courier. When ESC is involved in the actual sample collection, the samples are packed with ice on site and transported by ESC field personnel utilizing proper COC protocol.

5.8.7 Sample Custody

Chain of Custody

An important part of any sampling/analytical plan is ensuring sample integrity from collection to data reporting. Figure 5.8.7a is a flow diagram that represents the sample custody process. All records and documentation required to track a sample from point of origin through disposal must be available. The documentation of the life of the sample is referred to as "chain of custody." Formal chain of custody (COC) starts when the sample containers are requested. Such documentation includes container/shipping sheets, COC forms, field notebooks, field sample labels and custody seals, laboratory sample log sheets, sample extraction and digestion prep books, analytical workbooks and instrument logs, QC data associated with the sample set, and the final report. Examples of these documents are presented in Figures 5.8.7b through 5.8.7k.

Legal Chain of Custody

Legal COC involves all of the above, but actually begins in the laboratory with container preparation. All sample containers for collection purposes are purchased from the vender as certified clean per EPA protocols. When a kit is prepared for delivery to the field a Shipping Batch Detail Report is filled out stating the number and type of bottles, required preservatives, date prepared, date sent, and person preparing kit. A copy of the Shipping Batch Detail Report is generally kept beyond the estimated time of receipt of the kit back into the laboratory. The Shipping Batch Detail Report is sent with the kit for sampling guidance. The COC/Shipping BDR also represents the number of bottles sent to the client and the person preparing the kit. The containers are sent to the field in a portable cooler that is sealed with the COC/Shipping BDR inside by the person involved with preparation and remains sealed until the recipient opens the kit. The individual receiving the containers for field use, signs the COC at the time the kit and containers are released for shipment to the laboratory. COC forms and sample container labels identify the analyses, dates, times, and individuals who remove samples.

The COC represents all persons who have the sample in their custody at a given time. The client designates common carriers on the COC when the sample is shipped back to the laboratory.

FIGURE 5.8.7a
CHAIN OF CUSTODY PROCESS

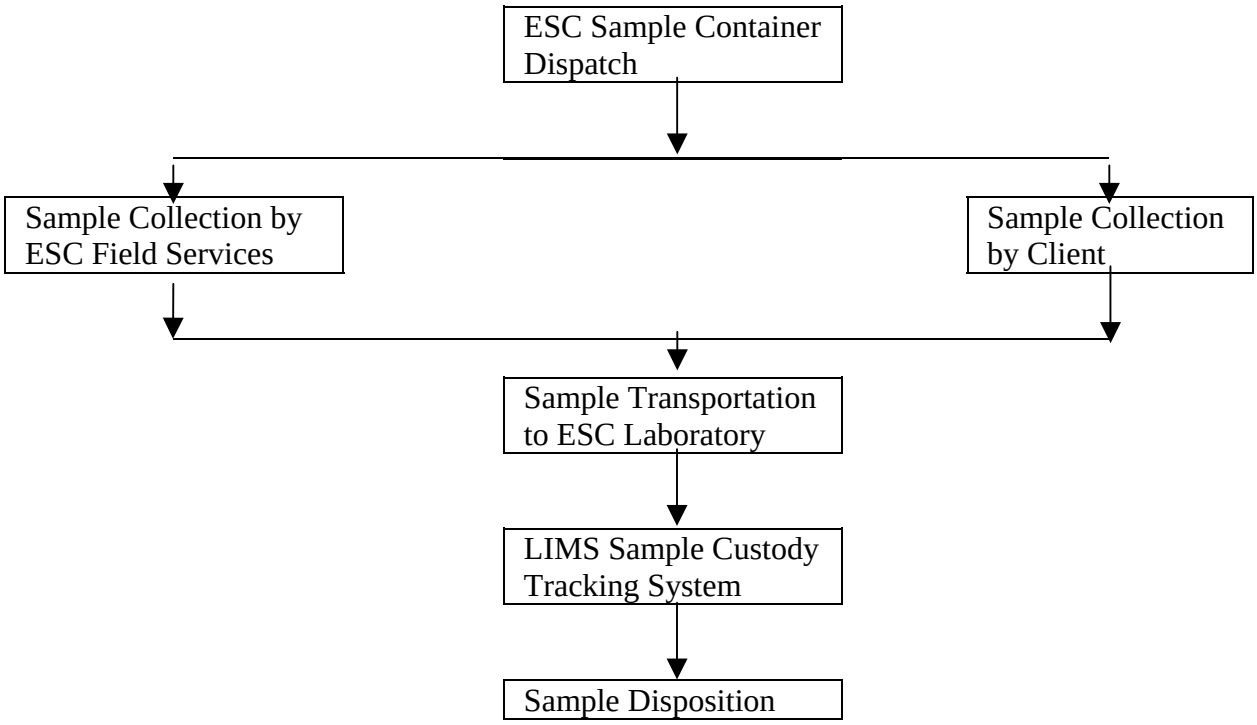
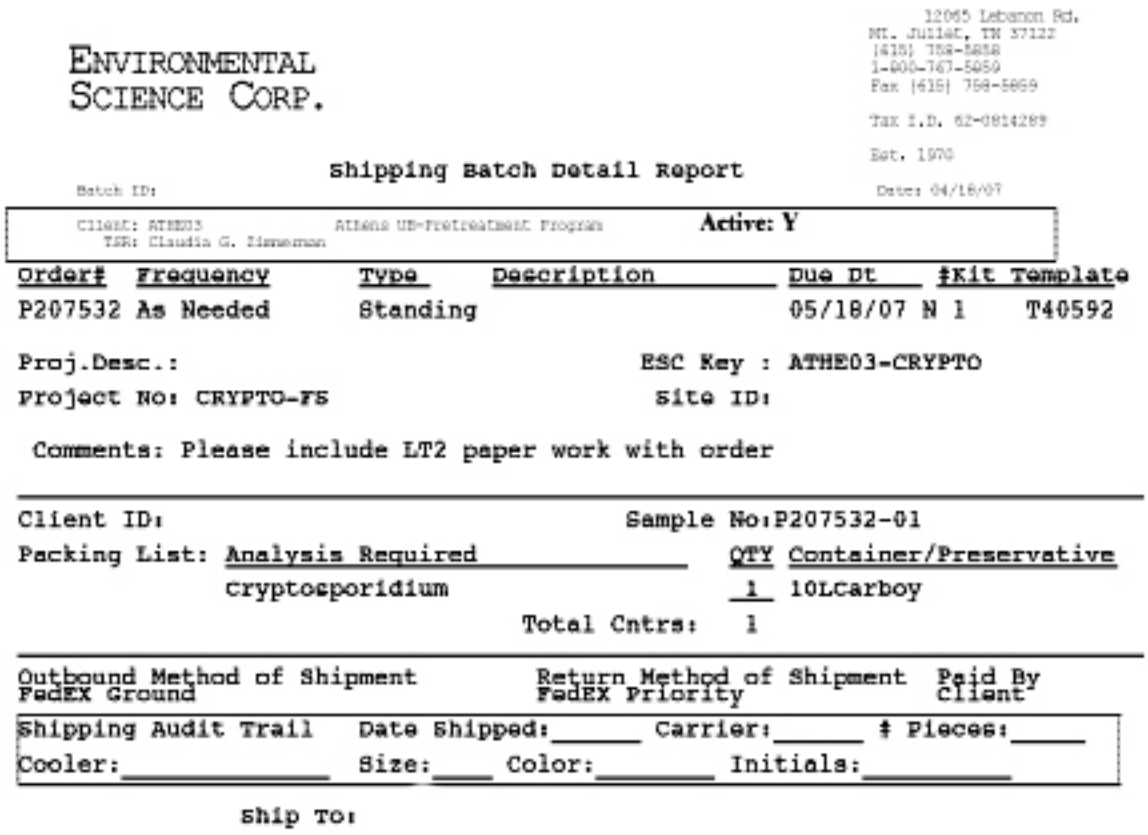


FIGURE 5.8.7b
INDIVIDUAL CONTAINER LOG
EXAMPLE
(Contents varies depending on client kit requirements)



ENVIRONMENTAL SCIENCE CORP.

12065 Lebanon Rd.
Mt. Juliet, TN 37122
(615) 758-5858
1-800-767-5859
Fax (615) 758-5859

Tax I.D. 62-0814289

Est. 1970

shipping Batch Detail Report

Batch ID:

Date: 04/18/07

Client: ATHE03 Athens UD-Pretreatment Program **Active: Y**
TSR: Claudia G. Zimmerman

Order#	Frequency	Type	Description	Due Dt	#Kit	Template
P207532	As Needed	Standing		05/18/07	N 1	T40592

Proj.Desc.: ESC Key : ATHE03-CRYPTO
Project No: CRYPTO-F5 Site ID:

Comments: Please include LT2 paper work with order

Client ID: Sample No:P207532-01

Packing List: Analysis Required	QTY	Container/Preservative
Cryptosporidium	1	10LCarboy
Total Cntrs:	1	

Outbound Method of Shipment	Return Method of Shipment	Paid By
FedEX Ground	FedEX Priority	Client

Shipping Audit Trail Date Shipped:_____ Carrier:_____ # Pieces:_____
Cooler:_____ Size:____ Color:_____ Initials:_____

Ship To:

Page 1 of 1

FIGURE 5.8.7c
CHAIN OF CUSTODY
GENERAL EXAMPLE
(Required Analysis is printed by ESC or Client)

Emerald Manufacturing
12065 Lebanon Road
Mount Juliet,TN 37122

Alternate billing information:

Analysis/Container/Preservative

Chain of Custody
Page ___ of ___

Prepared by:
ENVIRONMENTAL SCIENCE CORP.
12065 Lebanon Road
Mt. Juliet, TN 37122
Phone (800) 767-5859
FAX (615) 758-5859

Report to: Mr. Tom White
Email: twhite@envsci.com
Project Description: Demo report
City/State Collected
Phone: (615) 758-5858
FAX: (615) 758-5859
Client Project #: UPPB
Lab Project #: EMERALD-UPPB
Collected by (print):
Site/Facility ID#: 12345
P.O.#: 99999999999
Collected by (signature):
Packed on Ice N ___ Y ___
Rush? (Lab MUST Be Notified)
___Same Day 200%
___ Next Day 100%
___ Two Day 50%
Date Results Needed
Email? ___No X Yes
FAX? ___No ___Yes

Acctnum: EMERALD (lab use only)
Template/Prelogin: T33311/ P158345
Cooler #:
Shipped Via: FedEX Ground

Sample ID	Comp/Grab	Matrix*	Depth	Date	Time	No. of Cntrs	Metals 250mlHDPE-HNO3	SV8081 250ml Amb-No Pres	SV8270 1L-Amb-NoPres	SVOCS 1L-Amb NoPres SV8310	V8260 40ml Amb-HCl	SV8141 1L - AMB	SV8151 1L - AMB	Remarks/Contaminant	Sample # (lab only)
MW-1		GW				6	X	X	X	X	X	X	X		L219795-01
MW-2		GW				6	X	X	X	X	X	X	X		-02
MW-3		GW				6	X	X	X	X	X	X	X		-03
MW-4		GW				6	X	X	X	X	X	X	X		-04

*Matrix: SS - Soil GW - Groundwater WW - WasteWater DW - Drinking Water OT - Other_____

Remarks:

pH _____ Temp _____
Flow _____ Other _____

Relinquished by: (Signature)	Date:	Time:	Received by: (Signature)	Samples returned via: ☐UPS ☒FedEx ☐Courier ☐	Condition:	(lab use only)
Relinquished by: (Signature)	Date:	Time:	Received by: (Signature)	Temp: 4°C Bottles Received:		
Relinquished by: (Signature)	Date:	Time:	Received for lab by: (Signature)	Date: 12/25 Time:	pH Checked:	NCF:

FIGURE 5.8.7d

SAMPLE CONTAINER LABEL

ABC WASTEWATER PLANT

Prepared by Environmental Science Corp.

Project: Annual Sludge - SOUR/Class "B" Fecal

Proj #: 57243

Sample Location/ID: Sludge Digester

Analysis Req'd: Class "B" Fecal Coliform

NaThio Preservative Included

Date:_____ Time:_____

FIGURE 5.8.7e

SAMPLE CONTAINER CUSTODY SEAL

CUSTODY SEAL

Date:_____ **I-CHEM**

Chemists In The Container Business™

Signature: _____

FIGURE 5.8.7f

SAMPLE LOGIN LABEL

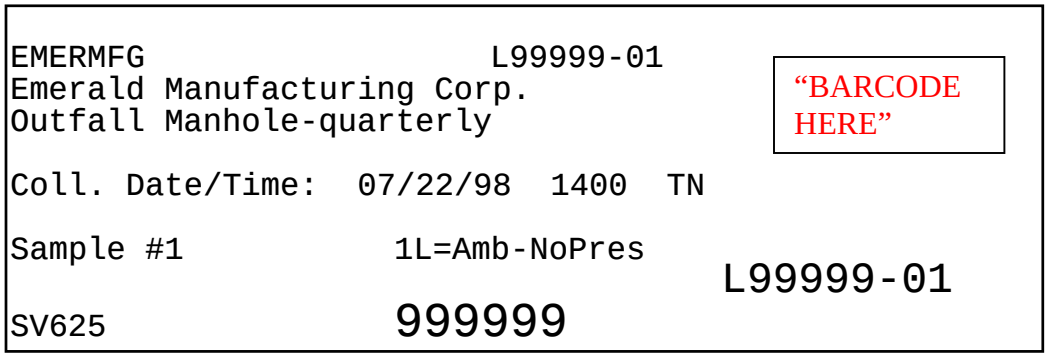


FIGURE 5.8.7g

EXAMPLE LAB PREPARATION SHEET

ENVIRONMENTAL SCIENCE CORP.

Laboratory Sample Prep Sheet

Date Created: 4/13/2007
Analyst: 196
Method: Hg
Matrix: Solid

Mercury by CVAA

Workgroup: WG295556

Samples

Account	Sample Name	Method	Weight(g)	Volume(mL)	Sample Description
[redacted]	L288458-01	7471A	0.58	30	Brown sludge
[redacted]	L288518-01	7471A	0.58	30	Brown clay
[redacted]	L288519-01	7471A	0.60	30	Brown clay
[redacted]	L288868-10	7471A	0.58	30	dark-brown clay
[redacted]	L288920-03	7471A	0.55	30	Purple paint
[redacted]	L288936-01	7471A	0.63	30	Brown clay
[redacted]	L288936-02	7471A	0.63	30	Brown clay
[redacted]	L288936-03	7471A	0.57	30	Brown clay
[redacted]	L288936-04	7471A	0.63	30	Brown clay
[redacted]	L288936-05	7471A	0.62	30	Brown clay
[redacted]	L288968-01	7471A	0.59	30	Black sludge
[redacted]	L288996-05	7471A	0.58	30	Brown sediment, rocks
[redacted]	L288997-20	7471A	0.57	30	Black sediment, rocks
[redacted]	L288997-21	7471A	0.57	30	Dark-brown sediment, rocks
[redacted]	L289003-01	7471A	0.59	30	Brown sludge
[redacted]	L289030-01	7471A	0.61	30	Grey clay
[redacted]	L289030-03	7471A	0.59	30	Grey clay
[redacted]	L289076-01	7471A	0.60	30	Brown sand, rocks
[redacted]	L289076-02	7471A	0.60	30	sand, rocks
[redacted]	L289095-01	7471A	0.58	30	Multicolored, rocks

QC Samples

Blank	BLKWG295556	7471A	0.60	30	Brown sand
LCS	LCSWG295556	7471A	0.10	30	Brown soil
DUP	L289076-01DUP	7471A	0.60	30	Brown sand, rocks
MS	L289076-02MS	7471A	0.60	30	Brown sand, rocks
MSD	L289076-02MSD	7471A	0.60	30	Brown sand, rocks

4/13/2007 11:05:50 PM 1 of 1

FIGURE 5.8.7h

EXAMPLE LAB ASSIGNMENT/WORKSHEETS

ENVIRONMENTAL SCIENCE CORP.

Laboratory Bench Sheet

TOTAL PHENOL BY 4AAP

Date Created: 4/2/2007
Analyst: 156
Method: 4AAP
Matrix: Water

Instrument: Lachat5

Workgroup: WG293700
Calibration Date: 03/15/07
Calib. Corr.: 0.999990
Units: mg/L

Prep Date: 4/2/2007

PrepStart: 11:20 PM
PrepEnd: 1:00 PM

Reagents

Reagent Name	Standard Number	Expiration Date
4 AAP	7D02049	04/03/07
PHENOL BUFFER	7C28009	04/04/07

Samples

Sample Name	Workgroup	Results	Dilution	Report Value	Qualifiers
L285470-02	WG293700	0.076	1	0.076**	
L286297-02	WG293700	-0.0208	1	<0.04	
L286321-02	WG293700	-0.0079	1	<0.04	
L286335-02	WG293700	0.0274	1	<0.04	
L286401-02	WG293700	0.0076	1	<0.04	
L286618-02	WG293700	0.0125	1	<0.04	
L286703-01	WG293700	0.042	1	0.042**	
L286703-02	WG293700	0.006	1	<0.04	
L286788-01	WG293700	0.0066	1	<0.04	
L286788-02	WG293700	0.0087	1	<0.04	
L286788-03	WG293700	0.0128	1	<0.04	
L286807-01	WG293700	0.162	1	0.162**	
L286807-02	WG293700	1.16	1	1.16**	
L286807-03	WG293700	0.069	1	0.069**	
L286807-04	WG293700	0.149	1	0.149**	

FIGURE 5.8.7i
EXAMPLE SAMPLE CONFIRMATION REPORT

Environmental Science Corp.
Login Confirmation Report
Apr 17 2007, 06:17 pm
Login Number: L3547 Template Number: N/A
Account: EMERALD Emerald Manufacturing

Report To: Tom White
: 12065 Lebanon Road
:
: Mount Juliet, TN, 37122
Telephone #: 615-758-5858
Fax #: -758-5859
Email: twhite@envsci.com;tomwhite@comcast.net
Project/Account Comments:

Client Project #: APP1
Project Description:
PO#: 1234
PO# Required: N
Lab Project #: EMERALD-123
Client Design: DEFAULT

TSR:151
Payment Terms: Net 30
Regulatory State: TN
Fax Report: N
Quote#:
Report Design:

Lab Sample #	Test	Sample ID	Desc.	Collect Date & Time	Collected By	Site	Receive Date	PR	Est.DueDate(1)	Method		Unit Price
L3547-01		MW-1		01-Nov-04, 12:00	Tom White	TN56383752	02-NOV-04	QR	09-NOV-04			
GW	P AP1		Appendix I List								$	250.00
GW	C AGICP		Silver		4625010	250mlHDPE-HNO3	DEFAULT			6010B	$	0.00
GW	C BAICP		Barium		4625010	250mlHDPE-HNO3	DEFAULT			6010B	$	0.00
GW	C BEICP		Beryllium		4625010	250mlHDPE-HNO3	DEFAULT			6010B	$	0.00
GW	C CDICP		Cadmium		4625010	250mlHDPE-HNO3	DEFAULT			6010B	$	0.00
GW	C COICP		Cobalt		4625010	250mlHDPE-HNO3	DEFAULT			6010B	$	0.00
GW	C CRICP		Chromium		4625010	250mlHDPE-HNO3	DEFAULT			6010B	$	0.00
GW	C CUICP		Copper		4625010	250mlHDPE-HNO3	DEFAULT			6010B	$	0.00
GW	C HG		Mercury		4625010	250mlHDPE-HNO3	DEFAULT			7470A	$	0.00
GW	C NIICP		Nickel		4625010	250mlHDPE-HNO3	DEFAULT			6010B	$	0.00
GW	C PBICP		Lead		4625010	250mlHDPE-HNO3	DEFAULT			6010B	$	0.00
GW	C SEICP		Selenium		4625010	250mlHDPE-HNO3	DEFAULT			6010B	$	0.00
GW	C SV8011		EDB/DBCP		4625011	40mlAmb-HCl	DEFAULT			8011	$	0.00
GW	C TLG		Thallium by ICPMS		4625010	250mlHDPE-HNO3	DEFAULT			6020	$	0.00
GW	C V8260AP1		App I Volatiles		4625011	40mlAmb-HCl	DEFAULT			8260B	$	0.00
GW	C ZNICP		Zinc		4625010	250mlHDPE-HNO3	DEFAULT			6010B	$	0.00
L3547-02		MW-2		01-Nov-04, 12:00	Tom White	TN56383752	02-NOV-04	QR	09-NOV-04			
Groundwater project												
GW	P AP1		Appendix I List								$	250.00
GW	C AGICP		Silver				DEFAULT		1 Bottles	6010B	$	0.00
GW	C BAICP		Barium				DEFAULT		1 Bottles	6010B	$	0.00
GW	C BEICP		Beryllium				DEFAULT		1 Bottles	6010B	$	0.00
GW	C CDICP		Cadmium				DEFAULT		1 Bottles	6010B	$	0.00
GW	C COICP		Cobalt				DEFAULT		1 Bottles	6010B	$	0.00
GW	C CRICP		Chromium				DEFAULT		1 Bottles	6010B	$	0.00
GW	C CUICP		Copper				DEFAULT		1 Bottles	6010B	$	0.00
GW	C HG		Mercury				DEFAULT		1 Bottles	7470A	$	0.00
GW	C NIICP		Nickel				DEFAULT		1 Bottles	6010B	$	0.00
GW	C PBICP		Lead				DEFAULT		1 Bottles	6010B	$	0.00
GW	C SEICP		Selenium				DEFAULT		1 Bottles	6010B	$	0.00
GW	C SV8011		EDB/DBCP				DEFAULT		2 Bottles	8011	$	0.00
GW	C TLG		Thallium by ICPMS				DEFAULT		1 Bottles	6020	$	0.00
GW	C V8260AP1		App I Volatiles				DEFAULT		2 Bottles	8260B	$	0.00
GW	C ZNICP		Zinc				DEFAULT		1 Bottles	6010B	$	0.00
L3547-03		MW-3		01-Nov-04, 12:00	Tom White	TN56383752	02-NOV-04	QR	09-NOV-04			

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Entered 28-JUN-02 By SEEDPAK

(1) Due Date listed is an estimate based on average workloads. Please communicate required due dates to your TSR.

5.9 QUALITY CONTROL

5.9.1 Quality Control Procedures

ESC has established quality control procedures for monitoring the validity of stated analytical methods. The resulting data are recorded in such a way that trends are detectable.

5.9.2 Quality Control Activities

Monitoring of quality may include the following:

- regular use of certified reference materials and/or internal quality control using secondary reference materials;
- participation in interlaboratory comparison or proficiency testing programs;
- replicate analyses
- re-testing or re-calibration
- logic check or correlation of results from related analyses

5.9.2.1 Quality control data are analyzed using statistical techniques and, where they are found to be outside pre-defined criteria, planned action is taken to correct the problem and to prevent incorrect results from being reported.

5.9.2.2 Laboratory Checks

See Section 3 for a description of QC samples and related definitions.

Table 5.9.2.2 BASIC LABORATORY QC CHECKS

QC Check Sample	Source	Prep Required
Method/reagent blanks - One blank is carried through each step of the analytical procedure for each batch of samples. Blanks are prepared for each preparation method and matrix (i.e., solids assay, dissolved metals, TCLP extraction, etc.). Blanks are used to confirm the absence of contaminants within the preparation and/or analytical system prior to and during the analysis of field samples.	Lab DI	Yes
Initial Calibration Verification (ICV) – An independently prepared standard used to verify the accuracy of the initial calibration (for ongoing calibration)	Primary or Secondary	No *
Laboratory Control Sample (LCS) – A known clean matrix is spiked with known amounts of the analyte(s) of interest used to verify the efficiency of the analytical system without interference from the field sample matrix. The LCS provides the best estimate of analytical system performance and may also be used to verify the validity of the on-going calibration.	Secondary	Yes
Continuing reference standard checks – Metals and Organics; *Also called SSCV (Secondary Source Calibration Verification) – An independently prepared standard used to verify the accuracy of the existing calibration.	Secondary	No
Continuing Calibration Verification (CCV) - A standard, usually near the mid-point of the calibration curve, made from the primary or same standard stock used for the calibration curve. The CCV is used to represent the ongoing calibration stability of the instrument and must perform within method stated criteria.	Primary	No *
Sample matrix spikes and spike duplicates (MS/MSD) –Prepared field samples spiked with known quantities of target analyte and carried through the entire preparation and analytical process concurrently with unspiked field samples to assess the effect of the sample matrix on the target analytes present and to provide an estimate of analytical precision. For analyses where field sample type does not allow for MS/MSD preparation (i.e. lead wipes, air samples on charcoal tubes, etc.) an LCS/LCSD pair may be substituted.	Primary or Secondary	Yes
Post Digestion Spike – (used in metals analysis) A standard prepared from a previously analyzed spiked sample digestate that yielded reduced recovery for the target analyte due to a suspected matrix interferent.	Primary	No
Sample duplicates – Second aliquots of field samples carried through the entire preparation and analytical process that used as an indication of sample precision or consistency in the field sample matrix.	Client Sample	Yes
Surrogate standards – Analytes not expected to occur naturally in field samples that are spiked by preparation/analytical personnel to assess sample preparation and analytical efficiency in each individual field sample.	NA	Yes
Internal standards– Analytes not expected to occur naturally in field samples that are spiked to provide a consistent basis for comparison with target analyte concentrations. ISTDs are used in internal calibration models.	NA	No

***** Preparation requirements can vary depending on method. Requirements are listed in each individual determinative SOP.

5.9.2.3 Batch QC Criteria

5.9.2.3.1 Environmental Samples

Sample Batch - Defined as a set of 20 or fewer samples of a similar matrix prepared and/or analyzed concurrently. The maximum number of samples possible per batch is dependent on the determinative method allowance.

Required Instrument QC per batch:

- Calibration Blank (CB or CCB)
- Initial Calibration Verification (ICV)
- (1) Continuing Calibration Verification (CCV) every 10-20 samples where and as required.
- (1) CCV at end of run where required.
- (1) Post-Digestion Spike – Metals analysis
- (1) Serial Dilution – Metals analysis

NOTE: The CCV is typically a mid-point concentration. In addition to the mid-point, where required, the CCV is run at a concentration that varies from the mid-point by +/-25% during each analytical run. The varied CCV must meet the same acceptance criteria as the mid-point.

Required Method QC per batch (Must include internal standards and surrogates, where required by the method):

- (1)Method/prep Blank
- (1) Laboratory Control Sample Duplicate Pair, LCS/LCSD must be analyzed for analytes where spiking procedures are not practical, such as total suspended solids, total dissolved solids, total volatile solids, total solids, pH, color, odor, oil& grease, temperature, dissolved oxygen or turbidity
- Matrix Spike/Spike Duplicate (MS/MSD) Pair, MS/MSD must be analyzed except for analytes where spiking procedures are not practical, such as total suspended solids, total dissolved solids, total volatile solids, total solids, pH, color, odor, oil& grease, temperature, dissolved oxygen or turbidity
- (1) Sample Duplicate (where sufficient field sample is available and where required by determinative method)

5.9.2.3.2 Industrial Hygiene Analyses, Including Environmental Lead

Sample Batch - Defined as a set of 20 or fewer samples of a similar matrix prepared and/or analyzed concurrently.

Required Instrument QC per batch:

- Calibration Blank (CB or CCB)
- Initial Calibration Verification (ICV)
- (1) Continuing Calibration Verification (CCV) every 10 samples
- (1) CCV at end of run.
- (1) Post-Digestion Spike – Metals analysis

NOTE: The CCV is typically a mid-point concentration. In addition to the mid-point, the CCV is run at a concentration that varies from the mid-point by +/-25% during each analytical run. The varied CCV must meet the same acceptance criteria as the mid-point.

Required Method QC per batch:

- (1) method/prep blank
- (1)Laboratory Control Sample/Laboratory Control Sample Duplicate Pair, LCS/LCSD
- Matrix Spike/Spike Duplicate (MS/MSD) pair, where matrix permits
- (1) Sample Duplicate (where sufficient sample is available)

5.9.2.3.3 Batch QC Protocols

If more stringent QC protocols are required than those outlined above for any method or project, then the more stringent method protocols are followed.

5.9.2.4 Inter-Laboratory Quality Control

- Reference samples are ordered from Environmental Resource Associates or similar provider. Samples are purchased to evaluate the following method types: Air, Water Supply, Water Pollution, and Solid Waste.
- Blind QC check samples are purchased at least semi-annually from Environmental Resource Associates or similar provider as an external source for performance evaluation samples. These samples are supplied to ESC without the true concentration values. For specific state water pollution programs, two levels are analyzed. The laboratory may perform additional studies as required by contract, regulatory agency or accreditation. ESC reviews the results as an overall check on internal QC procedures. If blind QC check sample results are unacceptable and such information impacts certification the laboratory immediately initiates corrective action and orders another check sample to ensure ongoing proficiency of that analyte.
- Blind field duplicates are collected at least annually to evaluate field collection and laboratory precision. Client field duplicates are collected based on project requirements. The field duplicates are logged in as regular samples and laboratory personnel are unaware of sample origin.
- Split samples are periodically sent to outside laboratories to confirm analytical results.

5.9.2.5 Procedures for Assessing Data Precision, Accuracy and Completeness

The following procedures apply to all analytes measured, unless more stringent QC has been specified. All field measurements must meet the same QC criteria as those run in the lab.

5.9.2.6 Use and Preparation of QC Samples

Certified standards, generated from reference materials, are used to check calibration throughout the analytical run. The standards are obtained from suppliers who are NIST recognized and ISO compliant. A Certificate of Analysis or other documentation verifying purity accompanies the standards.

Sample matrix spikes are prepared using actual samples prior to digestion, extraction, etc. Separate matrix spike limits are calculated for each type of sample (i.e., water, solid, TCLP extract, personnel filter, etc.). Sample duplicate analyses are also initiated prior to digestion, extraction, etc. Duplicate spikes and duplicate laboratory control samples are used to generate precision data.

Table 5.9.2 lists methods used to generate precision and accuracy targets.

TABLE 5.9.2.6 METHODS USED TO GENERATE PRECISION AND ACCURACY TARGETS		
Method	**Purpose**	**Method References**
Reference Standards (Laboratory Control Sample - LCS)	Accuracy	All analyses
Reference Standards (Dup. Laboratory Control Sample – LCSD)	Precision and Accuracy	All analyses
Matrix Spikes	Accuracy	All quantitative Wet Chemistry analyses. All Metals and Organics.
Duplicate Matrix Spikes	Precision and Accuracy	All quantitative Wet Chemistry analyses. All Metals and Organics.
Sample Duplicates	Precision	All analyses

5.9.2.7 QC Charts

When an analyst completes a reference standard check, a duplicate, or a matrix spike, the result is calculated and compared to the appropriate QC chart and evaluated against the established limits. A rough x-bar or duplicate QC graph, with mean, warning and control limits, is available. If the results are out of control limits, the analyst notes this problem for appropriate corrective action. Corrective action is taken, based on an established list of identified corrective action procedures.

Outliers

Control limits are calculated at least annually, where required. The data are evaluated using ± 4 times the standard deviation or 4σ criteria for outliers. Data that falls outside of ± 4 times the standard deviation are eliminated from the calculation. Data points are not eliminated otherwise, unless an obvious system failure has occurred and the error can be documented and identified.

Control Data Entry

For non-data transfer results, the data entry specialist gathers data directly from the benchsheet and enters it into the computer LIMS or Excel, depending on the origin of the data. For instrumentation with data transfer, the data is obtained directly from LIMS. The data is then brought into a spreadsheet and the charts can be plotted and evaluated by the computer software.

5.9.2.8 Accuracy

Laboratory Control Standards (LCS)

- Laboratory Control Standards are run with every analytical batch.
- X-bar control charts are generated using a minimum of the last 20 data points, based upon percent recovery.
- Warning limits are set at the 95% confidence interval and are plus/minus two standard deviations from the arithmetic mean.
- Control limits are set at the 99% confidence interval and are plus/minus three standard deviations.
- LCS limits are calculated at least annually where necessary. See the individual laboratory appendices for the list of established limits. Method stated limits override in-house calculated limits.

Percent Recovery:

$$Percent\ \ Recovery\ = \frac{Observed\ \ Concentrat\ ion}{True\ \ Concentrat\ \ ion} \text{ X } 100$$

Standard Deviation for Percent Recovery:

$$Sp = \sqrt{\frac{(P_1^2 + P_2^2 + P_3^2 + ...) \frac{(P_1 + P_2 + P_3 + ...)^2}{num\ of\ \ entries}}{num\ of\ \ entries - 1}}$$

Where: Sp = Standard deviation for percent recovery

$P_{1,2,3,..}$ = Percent recovery results

Matrix Spiked Samples

Spiked samples are typically ten percent of all samples, where matrix and sampling permits. Spiked samples are entered onto similar QC charts with the percent recovery. The target spike concentration routinely used is one to five times the initial concentration of the unspiked sample. This basis for the spike target provides analyte concentrations that do not exceed the range of the analysis and are not too small to be significantly affected by normal data variability. One exception for higher ratios is if an MS is spiked at one to five times the client sample concentration based on historical data but the client sample concentration turns out to be much lower or non-detect, the MS/MSD recovery results would still be usable.

- Matrix spiked samples are run with every analytical batch of samples.
- X-bar control charts are generated using a minimum of the last 20 data points, based upon percent recovery.
- Warning limits are set at the 95% confidence interval and are plus/minus two standard deviations from the arithmetic mean.
- Control limits are set at the 99% confidence interval and are plus/minus three standard deviations.
- MS limits are calculated at least annually or sooner where necessary. See the individual laboratory appendices for the list of established limits.
- Method stated limits supercede in-house calculated limits.

MS/MSD Percent Recovery:

$$\%\ Spike\ Recovery = \frac{Spiked\ sample\ value - initial\ sample\ value}{Concentration\ of\ spike}\ X\ 100$$

Standard Deviation for Percent Recovery:

Calculate using the same formula provided in the previous LCS section.

5.9.2.9 Precision

Precision is assessed through the use of duplicate client and/or QC samples, which constitute approximately 10% of all samples run. The relative percent difference (RPD) is calculated as follows:

$$RPD = \frac{\left|Duplicate\,1 - Duplicate\,2\right|}{\left[\frac{(Duplicate\,1 + Duplicate\,2)}{2}\right]} X\ 100$$

- Duplicates are analyzed with every analytical batch.
- X-bar control charts are generated using a minimum of the last 20 data points, based upon percent recovery.
- Warning limits (WL) are set at the 95% confidence interval using

$$WL = \text{Mean Value} + (2.456 \bullet \text{SD})$$

- Control limits are set at the 99% confidence interval and are plus three standard deviations.

$$CL = \text{Mean Value} + (3.268 \bullet \text{SD})$$

- Limits are calculated at least annually or sooner where necessary. See the individual laboratory Appendices for the list of established limits.
- For Laboratory Control Samples and Matrix Spikes - Calculate RPD using the actual analytical result.
- For Sample Duplicates – Calculate RPD using the actual analytical result.
- Calculate the standard deviation, separately for LCS, MS and Sample Duplicates by matrix, where appropriate.
- Method stated limits override in-house calculated limits.

5.9.2.10 Marginal Excedence Limits

Due to the large number of compounds analyzed using some analytical methods, it is statistically likely that accuracy and precision failures occur. Failures that occur on a random basis are deemed as marginal excedences and must meet the criteria below. Not all regulatory programs allow for the use of marginal excedence limits. In addition, not all analytical methods meet the requirements for the use of ME limits. Refer to the specific determinative SOP for more guidance regarding use and limitations.

Marginal excedences must be random events. If failures can demonstrate a pattern or occur with the same target analyte in a trend, the failure is actionable and not considered to be marginally exceeding the method requirements.

In addition, ME limits are utilized for methods with large numbers of target analytes being analyzed concurrently, as in the 8270/625 determinative method.

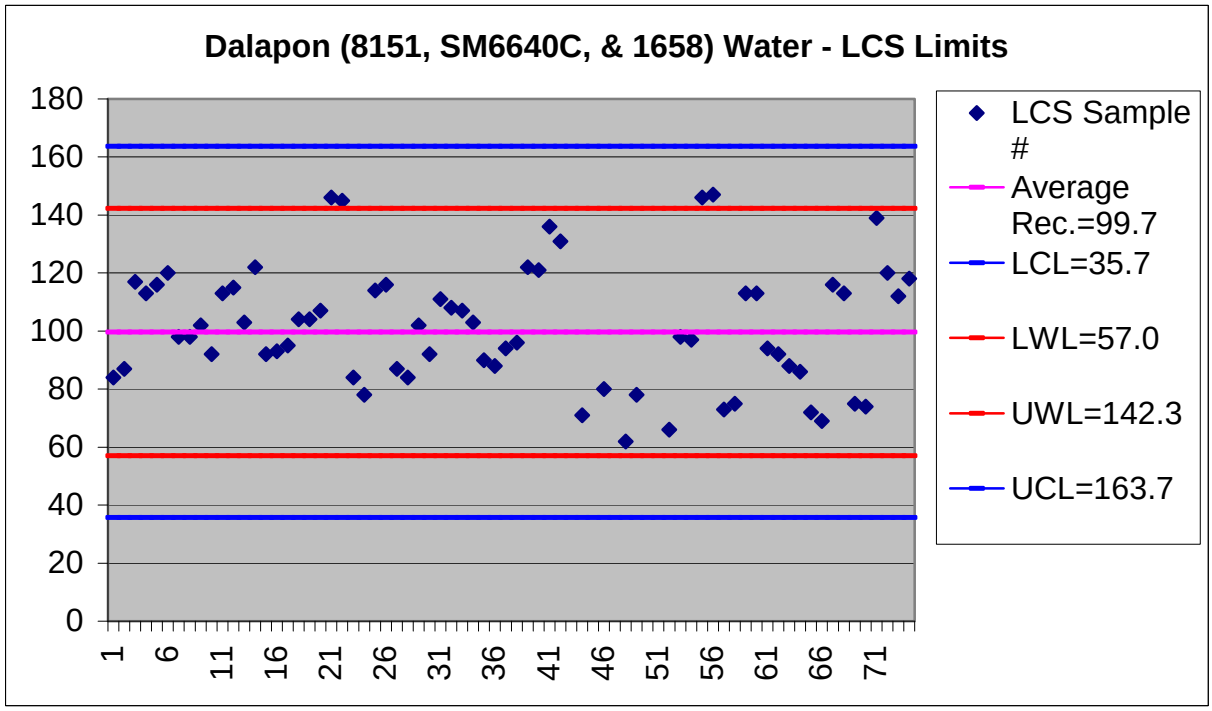
For example, the normal compound list for 8270/625 typically contains 90+ analytes; therefore, per the criteria listed below, only 5 analytes can be considered as marginally exceeding the acceptance criteria. If more than 5 failures occur or if the failures demonstrate a pattern that is causing the outliers, the entire sample batch with associated QC must be re-extracted and re-analyzed.

Upper and lower marginal excedence (ME) limits are established by +/- four times the standard deviation of historical accuracy data and the number of marginal excedences allowed is based on the number of analytes spiked in the LCS.

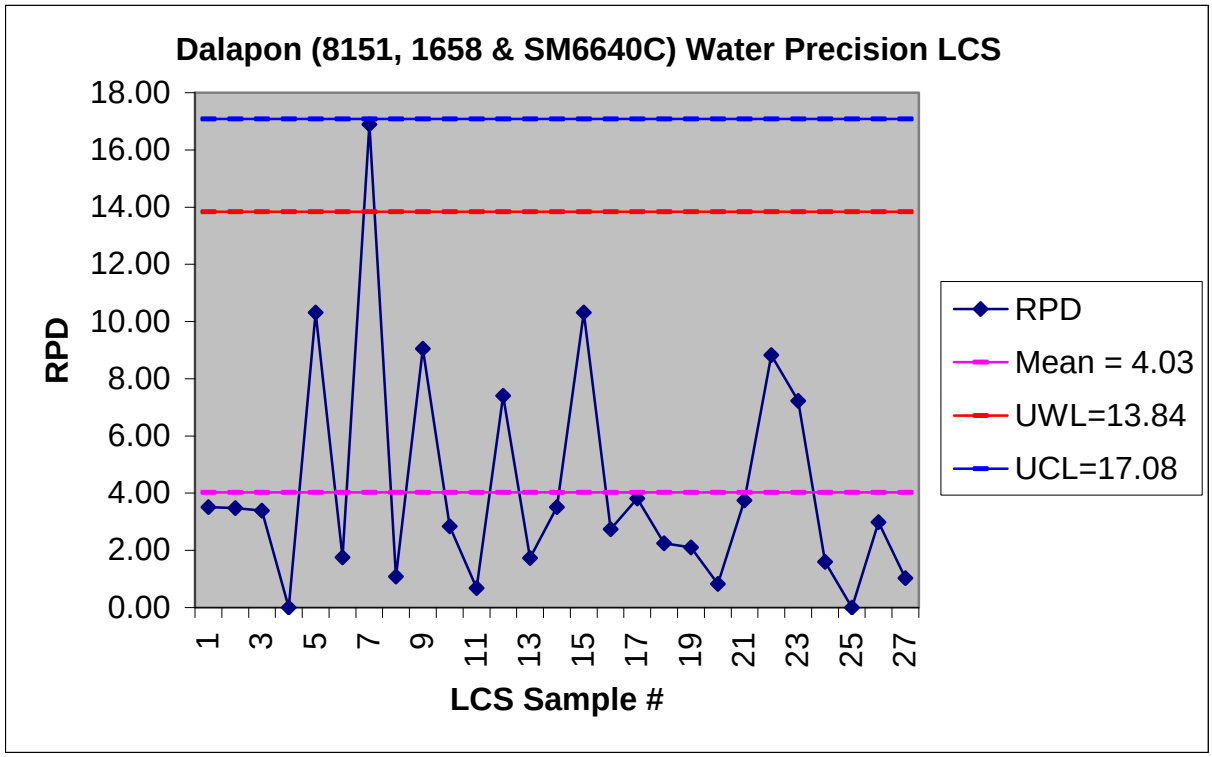
Number of Target Analytes	**Allowable Marginal Excedence Outliers**
90+	5 analytes allowed in the ME limit
71-90	4 analytes allowed in the ME limit
51-70	3 analytes allowed in the ME limit
31-50	2 analytes allowed in the ME limit
11-30	1 analytes allowed in the ME limit
<10	0 analytes allowed in the ME limit

FIGURE 5.9.2.10 PRECISION AND ACCURACY CHARTS

Dalapon LCS Duplicate Accuracy - Example



Dalapon LCS Duplicate Precision - Example



5.10 FINAL REPORTS/CERTIFICATES

5.10.1 General

The results of each analysis carried out by the laboratory are reported accurately, clearly, unambiguously, objectively, and in accordance with any specific instructions in the regulatory documents or standard operating procedures. The results are normally reported as a final client report and include all the information requested by the client and necessary for the interpretation of the analytical method results and all information required by the method of analysis.

5.10.2 Test Reports

In the case of a written agreement with the client, the results may be reported in a non-standard way and may not require the formalized information, but all associated analytical data is readily available and kept permanently on file for a minimum of 10 years. Specific programs or projects may require a longer data archive period.

Laboratory reports issued to the client for regulatory work, includes, at a minimum, the following information:

- Title – "Report of Analysis"
- Laboratory name, address and phone number
- Client name, address, and contact
- Client name and/or site name
- Client or field identification number
- Collection personnel
- Analyte Name
- Method number for each sample analyses
- Analytical result for each analysis with applicable Data Qualifier as outlined in Table 5.14
- Dilution factor (where applicable)
- Method Detection Limit (when requested)
- Practical Quantitation Limit – designated on final report as RDL
- Date of sample preparation (when requested)
- Time of sample preparation if the holding time is <48 hours (when requested)
- Date of sample analysis
- Temperature at which pH measurements are made
- Date and time of sample collection from the Chain of Custody form
- Units of measurement
- Wet/Dry weight ID – Dry weight includes total solids value
- Identification of all laboratories providing analytical results in the report, including the appropriate laboratory certification numbers from all certifying agencies. The "S" qualifier is used when analyses have been subcontracted.

- Individual report statements: "The reported analytical results relate only to the sample submitted." and "This report shall not be reproduced, except in full, without written approval from ESC".
- Approval Signature
- Sequential page numbering with total pages identified.
- Date/Time Printed
- Revision date – if any
- Laboratory certification numbers as assigned by each certifying agency.
- In conjunction with Ohio VAP projects, a signed affidavit is also required.

An example of a final client report is presented in below.

Figure 5.10.2 Example Final Client Report

ENVIRONMENTAL
SCIENCE CORP.

12065 Lebanon Rd.
Mt. Juliet, TN 37122
(615) 758-5858
1-800-767-5859
Fax (615) 758-5859

Tax I.D. 62-0814289

Est. 1970

REPORT OF ANALYSIS

Mr. John Jones
XYZ Consulting
000 Directors Drive
Anytown, US 00000

March 26, 2007

ESC Sample # : L99999-01

Site ID : 987-654

Project # : Low key

Date Received : March 16, 2007
Description : H2O

Sample ID : INFLUENT

Collected By : Jane Doe
Collection Date : 03/12/07 13:00

Parameter	Result	Det. Limit	Units	Method	Date	Dil.
TPH (GC/FID) Low Fraction	BDL	0.10	mg/l	8015GRO	03/20/07	1
Surrogate Recovery (70-130)						
a,a,a-Trifluorotoluene(FID)	87.8		% Rec.	8015GRO	03/20/07	1
Benzene	BDL	0.0010	mg/l	8260B	03/24/07	1
Toluene	BDL	0.0050	mg/l	8260B	03/24/07	1
Ethylbenzene	BDL	0.0010	mg/l	8260B	03/24/07	1
Total Xylenes	BDL	0.0030	mg/l	8260B	03/24/07	1
Methyl tert-butyl ether	BDL	0.0010	mg/l	8260B	03/24/07	1
Naphthalene	BDL	0.0050	mg/l	8260B	03/24/07	1
Surrogate Recovery						
Toluene-d8	95.7		% Rec.	8260B	03/24/07	1
Dibromofluoromethane	98.1		% Rec.	8260B	03/24/07	1
4-Bromofluorobenzene	99.7		% Rec.	8260B	03/24/07	1
Extractable Petroleum Hydrocarb	0.45	0.10	mg/l	EPH	03/20/07	1
Surrogate Recovery						
o-Terphenyl	82.8		% Rec.	EPH	03/20/07	1

BDL - Below Detection Limit
Det. Limit - Practical Quantitation Limit(PQL)
Note:
The reported analytical results relate only to the sample submitted.
This report shall not be reproduced, except in full, without the written approval from ESC.
.
Reported: 03/26/07 16:29 Printed: 03/26/07 20:01

ENVIRONMENTAL SCIENCE CORP.

12065 Lebanon Rd.
Mt. Juliet, TN 37122
(615) 758-5858
1-800-767-5859
Fax (615) 758-5859

Tax I.D. 62-0814289

Est. 1970

REPORT OF ANALYSIS

Ms. Alita Fire
ABC Consultants
123 Anywhere Street
Somewhere, US 00000

December 15, 2006

Date Received : December 07, 2006
Description : Place #1

Sample ID : Influent

Collected By : John Doe
Collection Date : 12/06/06 11:30

ESC Sample # : L00000-01

Site ID :

Project # : 123-456

Parameter	Result	MDL	RDL	Units	Q	Method	Date	Dil.
BOD	8.6	1.6	5.0	mg/l	J4	SM5210B	12/07/06	1
COD	27.	2.7	20.	mg/l		410.4	12/12/06	1
Ammonia Nitrogen	0.049	0.034	0.10	mg/l	J	350.1	12/11/06	1
Suspended Solids	15.	0.33	1.0	mg/l		160.2	12/11/06	1
Copper	U	0.0035	0.020	mg/l		6010B	12/08/06	1
Lead	U	0.0024	0.0050	mg/l		6010B	12/08/06	1

U = ND (Not Detected)
MDL = Minimum Detection Limit = LOD = SQL(TRRP)
RDL = Reported Detection Limit = LOQ = PQL = EQL = MQL(TRRP)
Note:
The reported analytical results relate only to the sample submitted.
This report shall not be reproduced, except in full, without the written approval from ESC.
.
Reported: 12/14/06 16:33 Revised: 12/15/06 09:10

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The following qualifier codes are used when reporting data values that either meet the specified description outlined below or do not meet the quality control criteria of the laboratory:

(This table provided for example and is subject to revision without notice. For a list current qualifiers, contact the laboratory)

Table 5.10.2 ESC Qualifiers and Descriptions (Updated 7/15/09)

QUAL	DESCRIPTION
A	ALC(EPA)-Aldol Condensation: Labels a suspected Aldol Condensation product for TICs.
B	(EPA) - The indicated compound was found in the associated method blank as well as the laboratory sample.
B1	(ESC) - The blank depletion was greater than the recommended maximum depletion of 0.2mg/L.
B2	(ESC) - The detection limit has been elevated due to blank contamination.
B3	(ESC) - The indicated compound was found in the associated method blank, but all reported samples were non-detect.
B4	(ESC) - The indicated compound was found in the associated instrument blank, but all reported samples were non-detect.
B5	(ESC) - The indicated compound was found in the associated instrument blank as well as the laboratory sample.
C	CBC(EPA)-Cannot be calculated: The analytical result cannot be calculated because the internal standard was not found.
D	Less than lower calibration limit. Actual value is known to be less than the lower calibration range due to dilution.
E	GTL (EPA) - Greater than upper calibration limit: Actual value is known to be greater than the upper calibration range.
F	SRN (EPA) - Diluted: The original sample was diluted due to high amounts of one or more target analytes. All associated method analytes will be subject to an elevated detection limit relative to the dilution factor.
G	SRS(EPA)-Secondary Dilution: The indicated analysis results were generated from a secondary dilution of the same sample. The sample had to undergo serial dilution.
H	RIN(EPA)-Re-Analyzed: The indicated analytical results were generated from a reinjection of the same sample extract or aliquot.
I1	(ESC) Not analyzed due to interference. (Sample reacted with method reagent or could not be analyzed due to interferences that could not be corrected)
J	(EPA) - Estimated value below the lowest calibration point. Confidence correlates with concentration.
J+	The associated batch QC was outside the upper control limits; associated data has a potential positive bias
J-	The associated batch QC was outside the lower control limits; associated data has a potential negative bias
J1	Surrogate recovery limits have been exceeded; values are outside upper control limits
J2	Surrogate recovery limits have been exceeded; values are outside lower control limits
J3	The associated batch QC was outside the established quality control range for precision.
J4	The associated batch QC was outside the established quality control range for accuracy.
J5	The sample matrix interfered with the ability to make any accurate determination; spike value is high
J6	The sample matrix interfered with the ability to make any accurate determination; spike value is low
J7	Surrogate recovery limits cannot be evaluated; surrogates were diluted out
J8	The internal standard associated with this data responded abnormally low. The data is likely to show a high bias concerning the result.
J9	The internal standard associated with this data responded abnormally high. The data is likely to show a low bias concerning the result.
K	REX(EPA)- Re-prepared: The indicated analytical results were generated from a re-extraction or preparation of the sample.
L	(ESC)Sample Pretreatment: The sample reaction impaired the ability to analyze the sample using normal analytical determination. Treatment outside of method protocol was required to determine the analytical result.
L1	(ESC) The associated batch LCS exceeded the upper control limit, which indicates a high bias; The sample analyte was "not detected" and is therefore unaffected.
L2	(ESC) The associated surrogate compound falls below 10%. The data should be used with caution. A re-extraction was not possible due to limited sample volume.

Table 5.10.2 ESC Qualifiers and Descriptions (Updated 7/15/09)

QUAL	DESCRIPTION
L3	(ESC) Sample reanalysis could not be performed due to lack of additional volume.
M	AVE(EPA)-Average Value: Used to report a range of values; e.g., relative response factors
N	PRE (EPA) - Presumptive evidence of material.
N8	PRE (EPA) - Presumptive evidence. The component has been tentatively identified based on mass spectral data.
N9	PRE (EPA) - Presumptive evidence. There is indication that the analyte is present, but QC requirements for confirmation were not met
O	(ESC) Sample diluted due to matrix interferences that impaired the ability to make an accurate analytical determination. The detection limit is elevated in order to reflect the necessary dilution.
O1	(ESC) The analyte failed both the method required serial dilution test and subsequent post-spike criteria. These failures indicate matrix interference.
P	NRP(EPA)-Non-Reproducible: Results of two or more injections are not comparable
P1	RPD value not applicable for sample concentrations less than 5 times the reporting limit.
Q	(ESC) Sample held beyond the accepted holding time.
R	REJ(EPA)-Rejected: Results have been rejected by the lab and should not be used
S	Subcontracted (ESC) - This analysis was performed by a subcontractor chosen to meet the project requirements.
T	(ESC) - Additional method/sample information: Sample collected using improper field protocol
T1	(ESC) - Additional method/sample information: Sample(s) received at greater than 4 degrees C.
T2	(ESC) - Additional method/sample information: The laboratory analysis was from an unpreserved or improperly preserved sample.
T3	(ESC) - Additional method/sample information: TOX analysis. Greater than 10% Breakthrough
T4	(ESC) - Additional method/sample information: QNS - Quantity Not Sufficient
T5	(ESC) - Additional method/sample information: QNS - Quantity not sufficient for reanalysis or replication as required by method.
T6	(ESC) - Additional method/sample information: Method used is an alternative to current approved methodology
T7	(ESC) - Additional method/sample information: Method 1664 (Total Oil & Grease), performed without silica gel
T8	(ESC) - Additional method/sample information: Sample(s) received past/too close to holding time expiration.
T9	(ESC) - Additional method/sample information: The sample result represents blank correction
U	BDL (EPA) - Below Detectable Limits: Indicates that the compound was analyzed but not detected.
V	(ESC) - Additional QC Info: The sample concentration is too high to evaluate accurate spike recoveries.
V1	(ESC) - Additional QC Info: Estimated concentration: due to inability to achieve ending QC standard as a result of sample matrix interference.
V2	(ESC) - Additional QC Info: The Total Cyanide value was below the reporting limit. Amenable Cyanide is assumed not to be present.
V3	(ESC) - Additional QC Info: The internal standard exhibited poor recovery due to sample matrix interference. The analytical results will be biased high. BDL results will be unaffected.
V4	(ESC) - Additional QC Info: Cont. Calibration Verification exhibited a response outside of the QC criteria, but within a 5% window. The associated analytical results are biased high. Non-detect results are unaffected.
V5	(ESC) - Additional QC Info: The Laboratory Control Sample exhibited a response outside of the QC criteria, but within a 5% window. The associated analytical results are biased high. Non-detect results are unaffected.
V6	(ESC) - Additional QC Info: The ICV responded above the recovery range for one of the following: Al, Ca, K, Fe, Na, Zn. The associated analytical results are biased high.
V7	(ESC) - Additional QC Info: This compound is not a 524.2 compound and was therefore evaluated using 8260B QC Criteria.
V8	(ESC) - Additional QC Info: The Interference Check Standard responded above the acceptable recovery range. The associated analytical result may be biased high for this element.

Table 5.10.2 ESC Qualifiers and Descriptions (Updated 7/15/09)

QUAL	DESCRIPTION
V9	(ESC) - Additional QC Info: Please refer to the Case Narrative provided with the report.
W	(ESC)-The laboratory analysis was from a sample collected in an improper container
W1	(ESC) - The laboratory analysis was from a sample collected in containers provided by the client.
W2	(ESC) - Insufficient sample amount to perform method as required. Sample amount approved per client instruction.
W3	(ESC) - BOD cannot be determined due to apparent toxicity exhibited by the sample.
X	(ESC)-Holding time exceeded due to National Emergency
X1	(ESC)-National Emergency: Temperature requirement has been exceeded due to delayed transportation.
Y	This sample most closely matches the laboratory standard for Kerosene
Y0	Significant peaks were detected outside of the hydrocarbon range defined by the method.
Y1	This sample most closely matches the laboratory standard for Diesel
Y2	This sample most closely matches the laboratory standard for #6 Fuel Oil
Y3	This sample most closely matches the laboratory standard for Hydraulic Fluid
Y4	This sample most closely matches the laboratory standard for Motor Oil
Y5	This sample has responded in the Diesel range, however it does not appear to be a hydrocarbon product
Y6	This sample has responded in the Oil range, however it does not appear to be a hydrocarbon product
Y7	This sample most closely matches the laboratory standard for Gasoline
Y8	This sample has responded in the Gasoline range, however it does not appear to be a hydrocarbon product
Y9	Sample has one or more single components in the gasoline range but the chromatographic trace is not characteristic of gasoline.
Z	(ESC)-Too many colonies were present(TNTC), the numeric value represents the filtration volume.

QUALIFIER REPORT INFORMATION:

ESC recognizes and utilizes sample and result qualifiers as set forth by the EPA Contract Laboratory Program. ESC firmly believes that relevant information pertaining to sample analysis be made available to the ESC client. In addition to the EPA qualifiers adopted by ESC, the laboratory has implemented ESC qualifiers to provide more information pertaining to analytical results. Each qualifier is designated in the qualifier explanation as either EPA or ESC. Definitions used in this table can be found in Section 3.

5.10.3 Optional Test Report Items

Where necessary, the final report contains a statement on the estimated uncertainty of measurement.

5.10.4 Calibration Certificates

ESC does not perform calibration activities for clients and therefore does not issue calibration certificates.

5.10.5 Opinions and Interpretations

Opinions and interpretations are allowed in final reports, in the form of qualifiers, provided that it is clear that the qualifiers are present to provide additional analytical information. In the event that a report must be issued with a revision, the original report remains unaltered and the revision is clearly identified. See SOP #030223, *Report Revision.*

5.10.6 Results from Subcontractors

ESC receives analytical reports from subcontracted laboratories. Results from subcontracted laboratories are clearly identified on the ESC client report.

5.10.7 Electronic Transmission of Results

Data packages are provided when requested by the client. They range from QC summaries to "CLP-like" packages with raw data. When a data package is requested at the beginning of a project, the level of package is identified, and it is then logged into the LIMS using the appropriate product code.

The analyst performing the analysis or a QC Specialist generates the QC documentation. The package is generated using the following process:

- Data and Supporting documentation is gathered by the QC Specialist (QCS)
- The package is formatted to the client request and submitted for review:
- Section Supervisor or Senior analyst
- Technical Specialist, Department Manager, Lab Director or designee.
- Once the reviews are complete, the package is logged, copied/scanned/burned to CD, and shipped. The ESC preferred means of delivery is via ESC's secure web site (PDF format) in recognition of the paperwork reduction act.
- See Table 10.8 for typical data package information.

5.10.8 Format of Reports

ESC client reports are designed to represent the analytical results unambiguously. Each client also has the option of using our web site to design a "custom" electronic report that will present results, historical data, and show trends in a format that is downloadable to a client database.

Client reports include the following information:

Table 5.10.8 Data Package Contents

Level I Level II	**Standard QC Data Package Provided Upon Request**
	Final Analytical Report with qualifiers where necessary
	Sub-Contract Final Report if applicable
	Chain of Custody (COC) Form
	Method Blank
	Matrix Spike/Spike Duplicate Summary (MS/MSD) - with Control Limits
	Laboratory Control Sample Summary (LCS) - with Control Limits
	Reporting Limits listed on all reports
	Surrogate Recoveries for GC and GC/MS analyses (on final report)
	Case Narrative upon request
Level III	**Data Package Provided Upon Request**
	All QC Data Included in Levels I and II plus:
	MS/MSD analysis performed on specific sample upon request
	Initial and Continuing Calibration Information
	Instrument blank performance
Level III - Mod	**Data Package Provided Upon Request**
	All QC Data Included in Levels I, II and III plus:
	Chromatograms, including Batch QC, and Samples
Level III - Mod	**Data Package Provided Upon Request**
	Quantitation Reports
	Analysis Log
	Extraction Logs
Level IV	**Data Package Provided Upon Request**
	("CLP-Like" Validation Package)
	All QC Data Included in Levels I, II, III and III mod plus:
	Multiple Sample Dilutions Reported
	Before/After reports when manual integration is necessary (where requested)
	Initial and Continuing Calibration Chromatograms and Quantitation
	Surrogate, Tune, Internal Std & Method Blank summary forms
	Standard Preparation Logs

5.10.9 Amendments to Reports

Reports that are amended after issue to the client, the amended report is clearly identified as such and a reference to the original report is made. The process is described in SOP 030223, *Report Revision*.

5.11 LABORATORY DATA REDUCTION *(SOP 030201 Data Handling & Reporting)*

The primary analyst completes the majority of data reduction using the following:

- Manual calculation, as represented on the bench sheet.
- Input of raw data for computer processing.
- Direct acquisition of raw data by computer.

5.11.1 Manual Calculations

If data requires manual calculation, the analyst has the responsibility of recording all steps involved directly on the bench sheet. Each bench sheet must be completed in a manner so that during review the person checking the raw data can easily reproduce the calculations. All pertinent information is included such as: response factors, dilution factors, and calibration constants. The analyst signs and dates each page of calculations in ink. A secondary review is required for all data. The second reviewer also initials and dates the worksheet. The worksheets are bound in chronological order in a laboratory workbook designated for each analysis.

5.11.2 Computer Processing

If data is input and processed using a computer, a hard copy of the input and output is reviewed to ensure that no discrepancies exist. The person entering the data and reviewing the data sign the data. The samples analyzed are evident. The data is identified by date analyzed or sample log number; in addition, a disc or tape backup is archived. Data files are uniquely identified by log number/parameter or date analyzed.

5.11.3 Data Acquisition

If data is directly acquired from instrumentation and processed, the analyst reviews the following for accuracy: sample log numbers, calibration constants, response factors, reporting units, and established numerical values used for detection limits (if a value is reported as less than the MDL). The analyst signs and dates the resulting output.

Data that is produced by instrumentation such as calibration curves, absorbance responses, chromatograms, etc. are identified with the following information:

- Date of analysis and initials of analyst
- Initials of review analyst
- Instrument Identification
- Type of analysis

Instrument run logs can be cross-referenced by date to access information on instrument conditions.

5.11.4 Analytical Data Records

Manual data entries are done with indelible ink. All errors are corrected with a single line strikethrough followed by initials and date. The corrected entry appears adjacent to the incorrect entry.

***Manual Data**:*

All manual analytical data represents the following:

- Lab Sample ID
- Analysis Type and Method Number
- Date of analysis
- Prep Date/time
- Time of analysis (if holding time <72 hours)
- Instrument ID
- Calibration Date
- Analyst Initials
- Required QC
- Calculations
- Matrix
- Sample volume/amount
- Dilutions (if any)
- Units of measure
- Correlation coefficient
- Reagent ID – cross reference to preparation date/origin
- Standard ID – cross reference to preparation date/origin
- Calculations where required (manual)
- Qualifiers
- Comments where necessary
- Reviewer initials

Instrument Data:

The instrument printout and supporting data represents the following:

- Instrument ID – cross reference to maintenance log and instrument conditions
- Date/time of analysis
- Injection log/Sample run log
- Operator ID
- Instrument Responses
- Chromatograms/printouts (including manual integrations)
- Units of measure
- Sample amount/volume
- Dilutions
- Sample ID
- QC Samples
- Calibration Date
- Filename
- Comments
- Analyst Initials
- Review Initials
- Standard ID – cross reference to preparation date/origin
- Software version
- Method ID

5.12 DATA VALIDATION PROCESS

5.12.1 Chain of Custody Review

One of the first steps in the validation process is review of the chain of custody (COC). The COC is reviewed first when the sample arrives. It is checked for completeness as well as time accountability. If the COC is complete and accurate, it is then processed through the system. If any irregularity is found, a non-conformance sheet is filled out, with the TSR sign-off, etc. The samples are released for analysis upon approval of the COC.

5.12.2 Field Data

Field data must meet all calibration and continuing calibration requirements. All field data is reviewed for accuracy and completeness. The field data must be approved before it can be entered onto a report. The Environmental Monitoring Manager reviews recorded field data. Field QC criteria are explained in detail in Section 5.7 and in Appendix III.

5.12.3 Laboratory Analysis, QC, and Data Review

Lab Analyst

- After the COC has been reviewed and the sample has been logged in, the laboratory performs all required analyses. The Lab Supervisors are responsible for ensuring that all samples are run within holding time.
- At the beginning of each analysis or sample preparation, the analyst is responsible for making sure that all laboratory ID numbers on the sample bottles match those listed on the benchsheet or logbook.
- Sample transfer from bottle to container is periodically spot checked by a qualified senior analyst.
- Upon completion of the analysis the analyst verifies that analytical information and results are correct and complete, the appropriate SOP has been followed, manual integrations (where applicable) have been correctly interpreted, QC samples are within established limits, and supporting documentation is complete.
- The benchsheet is then given to a QC Specialist who reviews the same information and ensures all portions of the benchsheet are complete.
- The review person then initials and dates the benchsheet.

Extraction/Sample Prep

- The Department supervisor's are responsible for reviewing all extraction/ preparation logs. The review verifies completeness regarding method, sample amount, reagent amount, times, temperatures, etc.
- The extraction/prep logs are reviewed for sample prep method as well as sample extraction date versus holding time.

Final Data Responsibility

- The Department supervisor for each section of the laboratory is responsible for reviewing instrument run logs and benchsheets to ensure that the samples are being prepared and analyzed within holding times.
- The QC Specialist performs a final review before the data is approved for input into the computer.
- This review includes performance of the various blanks, precision QC and accuracy QC to determine if the set is within quality control criteria. Data reviews are conducted according to the SOP #030227, *Data Review*, that provides more detail regarding specific steps taken in the review process. In some cases, specific regulatory guidelines on the data review process include additional requirements (i.e. Ohio VAP's data review checklist use) that are also included in the SOP.
- If the data is not approved during the final review process, it is given a pending status and returned to the laboratory.
- Pending data is reviewed for corrective action and may require only re-calculation or may result in re-analysis.

Final Report Review

- For manual data, the reviewed data is entered in the LIMS; the input is reviewed against the raw data by a second person for accuracy.
- Data transfer is reviewed and approved by a QC Specialist.
- The client reports are then prepared for review by the assigned Technical Service Representative (TSR). The report is reviewed for correlation between related parameters as well as possible trends. The TSR reviews related supporting documentation such as chain of custody records, field documents, sample receipt information, compliance with client/project specific requirements, etc.
- All field documents are reviewed and approved before the final review. Field data that does not pass established criteria is not processed through the final report review.
- The Environmental Monitoring Manager is responsible for any corrective actions necessary concerning field results.
- Laboratory result values that appear anomalous are sent back to the laboratory for a second review of the raw data.
- If there is no apparent reason for the anomaly the sample is re-analyzed.
- If the sample holding time has expired, the sample is re-analyzed and flagged.
- If the client desires, a new sample can be collected and evaluated.
- The chain of custody is also reviewed for a final time to ensure that all project objectives have been met.
- The LIMS footnotes any parameters that may exceed established limits as provided by the client.
- When the LIMS notes that a limit has been exceeded, the Technical Service Representative is notified and the client is contacted.

Table 5.12.3 DATA REDUCTION AND VALIDATION FLOW		
Primary Activity	**Supporting Activity**	**Responsibility**
Review of COC	Login Confirmation to Client	Initially by Login Personnel and again by Technical Service Representative
Data Production and Reduction	Supporting documentation	Primary Analyst/Chemist
Review of Laboratory QC	Review of Data Completion and QC Limit Verification	Primary Analyst/Chemist
Approval of Laboratory QC	Review of Data Completion and QC Limit Verification	QC Specialist/Senior Chemist
Approval of ESC Field QC and Data	Review of Field Records	Environmental Monitoring Manager
Data Entry to LIMS	Data Transfer	Analyst followed by QC Specialist
Data Entry to LIMS	Data Transfer - Application of Qualifiers	Data Entry Specialist followed by QC Specialist Verification
Data Entry to LIMS	Manual Entry of Data and Qualifiers	Data Entry Specialist followed by QC Specialist Verification
Draft Final Report Generation	Report printed and given to TSR for Approval	Data Entry Specialist or Administrative Assistant
Final Report Review and Approval	TSR Approval/Signature	Technical Service Representative (TSR)

6.0 WASTE MINIMIZATION/DISPOSAL AND REAGENT STORAGE

ESC's sample disposal policy is founded on RCRA [40 CFR Part 261.4 (d)] and CWA [40 CFR Part 403 (Pretreatment)]. Part 261.4 (Figure 6.1) excludes a sample of waste while it is a sample; however, once no longer fitting the description of a sample, it becomes waste again. The policy is further strengthened by information found in "Less is Better" published by the ACS and developed by the ACS Task Force on RCRA.

The EPA requires that laboratory waste management practice to be conducted consistent with all applicable rules and regulations. Excess reagents, samples and method process wastes must be characterized and disposed of in an acceptable manner. Refer to ESC SOP #030309, Waste Management Plan for detailed information.

6.1 QUARANTINED SOIL SAMPLES

ESC maintains a permit to receive and analyze soils from foreign or quarantined areas. All non-hazardous soil samples are disposed of as originating from a quarantined area. All unconsumed soil samples and containers are sterilized in accordance with the current USDA regulations found in 7 CFR 301.81. Both container and contents are dry-heated at 450°F for two minutes, then crushed prior to disposal into a sanitary landfill.

6.2 MOLD/BIOHAZARD SAMPLE DISPOSAL

The laboratory has contracted a local licensed medical waste removal and disposal firm to remove all biohazard and medical waste generated by the laboratory. All waste arriving at the treatment facility is incinerated or steam sterilized complying with all Federal, State, County and local rules, regulations and ordinances. The medical waste containers are picked up at least weekly and confirmation records are available in the laboratory.

All wastes classified as non-biohazard are disposed of via the sanitary sewer following treatment with a disinfectant such as Chlorox (hypochlorite) until the disinfectant and waste liquid is one part disinfectant and five parts waste liquid. Waste disposal records indicating the disposal method are available in the laboratory.

6.3 REAGENTS, STORAGE AND WASTE DISPOSAL

6.3.1 Reagents:

- All chemicals are at least ACS reagent-grade or better.
- All reagents and chemicals are checked for quality, purity and acceptability upon arrival in the laboratory.
- Each chemical container displays the following information: date opened and the expiration date.
- All reagent solutions prepared in-house contain the following information: date prepared, analyst initials, expiration date, and reagent name. In house reagents are recorded with the same information in a reagent prep book assigned to that method.
- Purchased reagent solutions are labeled when received and opened and with the expiration date.

6.3.2 Storage:

- Reagents requiring refrigeration are stored in the area of use in a suitable refrigerated storage that is separate from sample storage.
- Reagents and standards used for volatile organic analysis are stored in a separate refrigerator and are not stored with samples.
- See the following table for more information regarding reagent storage.

Item	Reagent Storage
Acids	Designated acid storage cabinets, in original container.
Organic Reagents - Flammables	Stored in flammables cabinet on separate air system from volatiles analysis.
Liquid Bases	Stored in designated cabinet, away from acids.
Solid Reagents	General cabinet storage.
Refrigerated Aqueous Reagents/Standards	Stored in walk-in cooler on designated shelves, away from samples.
Stable Standard Solutions	Storage cabinet designated in each laboratory for standards.
Dehydrated Media	Dehydrated media is stored at an even temperature in a cool dry place away from direct sunlight. Media is discarded if it begins to cake, discolor, or show signs of deterioration. If the manufacturer establishes an expiration date, the media is discarded after that date. The time limit for unopened bottles is 2 years at room temperature. Where needed comparisons of recovery of newly purchased lots of media against proven lots, using recent pure-culture isolates and natural samples, are performed.
Pure Biological Cultures	All organisms are stored on Tryptic Soy Agar at 4°C in a dedicated refrigerator located in the biology department

6.3.3 Disposal:

- All excess, out of date or unneeded chemicals, reagents and standards are sent to the ESSH Office to ensure proper disposal. Excess chemicals designated as hazardous waste are lab packed and disposed of according to local, State and Federal regulations. Final disposal method is dependant on the classification of each individual chemical. Some sample extracts, chemicals or standards designated as hazardous waste may be disposed of into appropriate satellite accumulation areas. Any additional EPA waste codes resulting from addition of standard are applied to the satellite container, if applicable.

- ESH prohibits the sink disposal of chemicals, the intentional release of chemicals through chemical fume hoods and mixing of nonhazardous lab trash with hazardous waste.
- Sample and reagent/solvent disposal is handled in different ways according to toxicity.
  - Solvents, reagents, samples and wastes are segregated according to base/acid, reactive/non-reactive, flammable/non-flammable, hazardous/non-hazardous, soil/liquid etc. Samples are grouped together relevant to these categories and are disposed of accordingly.
  -
  -
  - Table 6.1 lists waste disposal methods for various test byproducts.
- Upon receipt and login, each sample is coded by sample matrix type. The codes divide samples into the following groups: air, industrial hygiene, wastewater, cake sludge, soil, drinking water, food and miscellaneous. As laboratory personnel review the data reported, the method of disposal is also determined.
- The TSR is notified if samples are to be returned to the client.

6.4 CONTAMINATION CONTROL

6.4.1 Metals

The metals lab conducts quarterly wipe testing in order to ensure that the environment is contaminant free. All critical areas are included and a record is kept of the sampling plan (including locations) and results. Bench tops, balances, digestion equipment, and instrument areas are evaluated against the regulatory limit. Any detectable concentration must be $\leq$1/2 of the established regulatory limit for each metal being analyzed. If any detectable amount exceeds the established criteria, then the area must be cleaned and verified before analysis can resume.

6.4.2 VOC's

The VOC Lab is physically separated from the Extraction Laboratory in order to eliminate

contamination caused by the use of extraction solvents. Contamination is monitored daily through the use of instrument/method blanks.

6.4.3 Biological Lab

The aquatic toxicity testing, mold testing, and all other biological determinations are performed in the administrative building and are therefore physically separated from processes involving solvent or other chemical use. The mold lab conducts monthly analyses to ensure that the laboratory environment is contaminant free. All critical areas are included and a record is kept of the sampling plan (including locations) and results.

TABLE 6.1 - WASTE DISPOSAL

NOTE: This information is a general guide and is not intended to be inclusive of all waste or hazardous samples.

PARAMETER	WASTE PRODUCTS	WASTE CLASSIFICATION	DISPOSAL METHOD
Acidity	slightly alkaline water	none	neutralize-sanitary sewer
Alkalinity	slightly acidic	none	neutralize-sanitary sewer
BOD, 5-day	Sample waste only	none	sanitary sewer
COD	acid waste, Hg, Ag, Cr+6	corrosive, toxic	dispose via haz waste regulations
Conductivity	None		
Cyanide, Total	acidic waste	corrosive	neutralize-sanitary sewer
Cyanide, Amenable	acidic waste	corrosive	neutralize-sanitary sewer
Flashpoint	Misc. Organic waste contiaining Chlorobenzene	Flammable	Dispose via haz waste regulations
Fluoride, Electrode	neutral waste solution	none	sanitary sewer
Hardness, Total	pH 10.0 alkaline waste	none	neutralize-sanitary sewer
Extraction/prep	methylene chloride and hexane	toxic solvents	Reclaim for resale
Methylene Blue Active Sub.	Acidic Chloroform Waste	toxic & acidic	dispose via haz waste regulations
Nitrogen-Ammonia	alkaline liquids	corrosive	neutralize-sanitary sewer
Nitrogen-Total Kjeldahl	Trace Hg in alkaline liquid	corrosive toxic	neutralize-sanitary sewer
Nitrogen-Nitrate, Nitrite	mild alkaline waste	none	sanitary sewer
Oil & Grease and Petroleum/Mineral Oil & Grease	Hexane	Toxic solvent	dispose via haz waste regulations
pH	Sample waste only	none	sanitary sewer
Phenols	slightly alkaline, non-amenable CN-	none	sanitary sewer
Phosphate-Total and Ortho	combined reagent	listed	sanitary sewer
Reactive CN & S	Acidic waste	corrosive	Neutralize - sanitary sewer; waste is monitored for CN
Solids, Total (% solids)	None		
Solids, Total Dissolved	None		
Solids, Total Suspended	None		
Turbidity	None	none	none
Metals	acids, metal solutions	corrosive, toxic	highly toxic metal standards and samples - dispose via haz waste regulations
Volatile Organics	methanol	toxic solvents	dispose via haz waste regulations
Extractable Organics	solvents, standards	toxic solvents	dispose via haz waste regulations
Biological Non-biohazardous Waste	Food samples, enrichment broth,	none	Disinfect – sanitary sewer

PARAMETER	WASTE PRODUCTS	WASTE CLASSIFICATION	DISPOSAL METHOD
Biological Non-biohazardous Waste	Gloves, plastic containers	none	Standard refuse

FIGURE 6.1 (*reprint of excerpt – current as of 3/12/08*)

40 CFR PART 261-IDENTIFICATION AND LISTING OF HAZARDOUS WASTE

Subpart A-General Sec.

261.1 Purpose and definition.
261.2 Definition of solid waste.
261.3 Definition of hazardous waste.
261.4 Exclusions.
261.5 Special requirements for hazardous waste generated by conditionally exempt small quantity generators.
261.6 Requirements for recyclable materials.
261.7 Residues of hazardous waste in empty containers.
261.8 PCB wastes regulated under Toxic Substance Control Act.

Sec.261.4 Exclusions.

(d) **Samples.** (1) Except as provided in paragraph (d)(2) of this section, a sample of solid waste or a sample of water, soil, or air, which is collected for the sole purpose of testing to determine its characteristics or composition, is not subject to any requirements of this part or parts 262 through 268 or part 270 or part 124 of this chapter or to the notification requirements of section 3010 of RCRA, when:

(i) The sample is being transported to a laboratory for the purpose of testing; or

(ii) The sample is being transported back to the sample collector after testing; or

(iii) The sample is being stored by the sample collector before transport to a laboratory for testing; or

(iv) The sample is being stored in a laboratory before testing; or

(v) The sample is being stored in a laboratory after testing but before it is returned to the sample collector; or

(vi) The sample is being stored temporarily in the laboratory after testing for a specific purpose (for example, until conclusion of a court case or enforcement action where further testing of the sample may be necessary).

(2) In order to qualify for the exemption in paragraphs (d)(1) (i) and (ii) of this section, a sample collector shipping samples to a laboratory and a laboratory returning samples to a collector must:

(i) Comply with U.S. Department of Transportation (DOT), U.S. Postal Service (USPS), or any other applicable shipping requirements; or

(ii) Comply with the following requirements if the sample collector determines that DOT, USPS, or other shipping requirements do not apply to the shipment of the sample:

(A) Assure that the following information accompanies the sample:

(1) The sample collector's name, mailing address, and telephone number;

(2) The laboratory's name, mailing address, and telephone number;

(3) The quantity of the sample;

(4) The date of shipment; and

(5) A description of the sample.

(B) Package the sample so that it does not leak, spill, or vaporize from its packaging.

(3) This exemption does not apply if the laboratory determines that the waste is hazardous but the laboratory is no longer meeting any of the conditions stated in paragraph (d)(1) of this section.

ESC Site Plan

APPENDIX I TO THE ESC QUALITY ASSURANCE MANUAL

for

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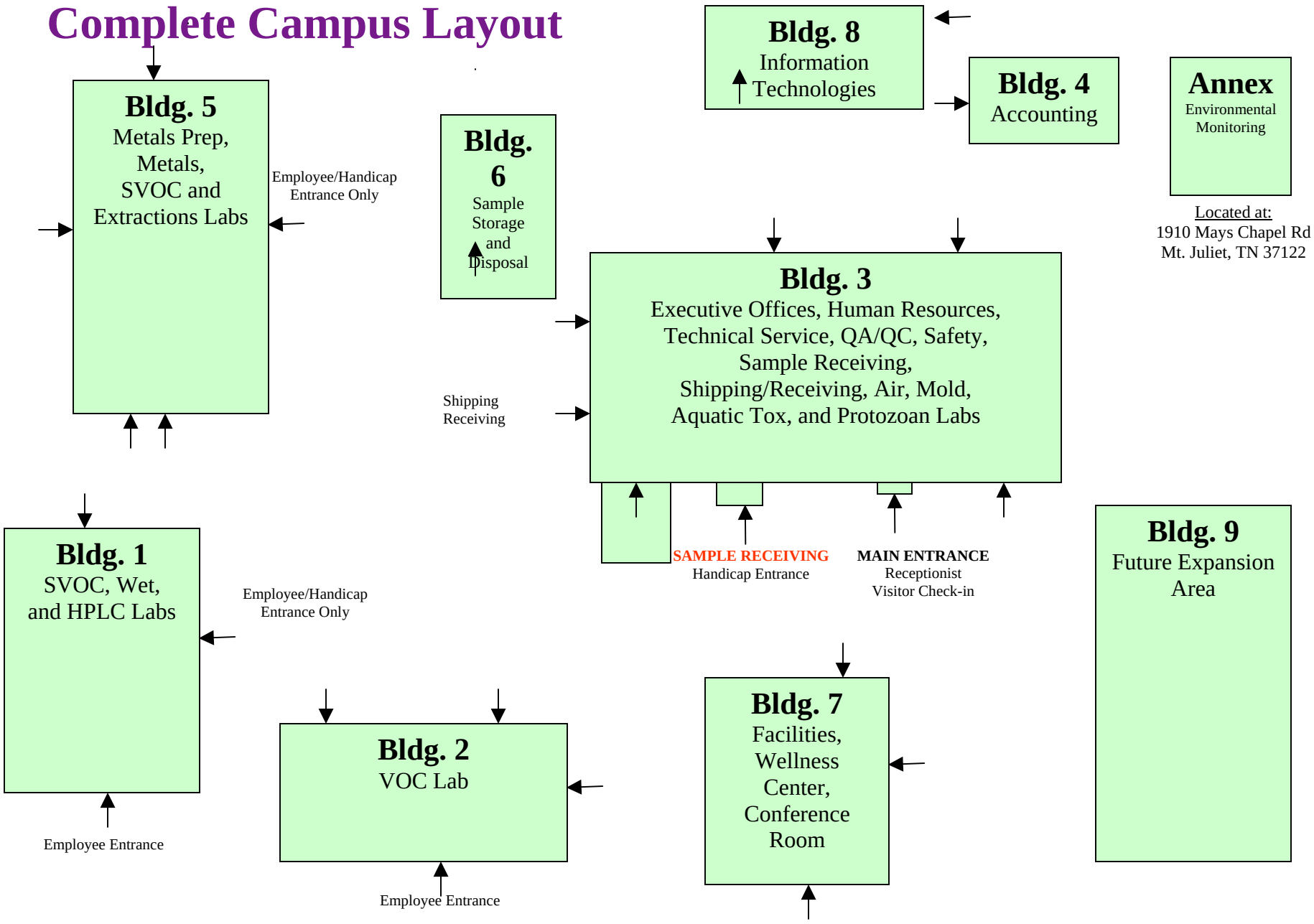
Complete Campus Layout



ESC Certifications

APPENDIX II TO THE ESC QUALITY ASSURANCE MANUAL

for

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ENVIRONMENTAL SCIENCE CORPORATION

Certification Summary

State/Agency	Certificate Number	Expiration Date/Status	Cert. REV. Date	Date Posted	Certified Programs	Approved Programs	Cert.Type	Cert. Authority
Alabama	40660	6/30/2011		7/23/2010	DW	WW, RCRA, UST	Reciprocity	TN
Alaska	UST-080	1/11/2011		4/20/2010	UST	UST	AK	AK
Arizona	AZ0612	6/25/2011		7/8/2010	AIR, DW, WW, RCRA, UST		Audit	AZ
Arkansas	88-0469	1/21/2011		2/18/2010	WW, RCRA, UST, BIoassay		NELAP	NJ
California	01157CA	8/31/2011		9/28/2010	WW, RCRA, UST		NELAP	NJ
Colorado	None	3/31/2011		6/2/2009	DW	WW, RCRA, UST	Reciprocity	TN
Connecticut	PH-0197	3/31/2011		4/16/2009	DW	WW, RCRA, UST	Reciprocity	TN, NJ
Florida	E87487	6/30/2011		7/8/2010	AIR, DW, WW, RCRA, UST		NELAP	NJ
Georgia DW	923	Renewal	Renewal	8/17/2007	DW		Reciprocity	TN
Georgia	None	6/30/2011		7/8/2010	WW, RCRA, UST		NELAP	NJ
Idaho	TN00003	6/1/2011		8/5/2010	DW	WW, RCRA, UST	NELAP	NJ
Illinois	200008	11/30/2011		11/22/2010	DW, WW, RCRA, UST		NELAP	NJ
Indiana	C-TN-01	6/16/2013		8/5/2010	DW	WW, RCRA, UST	Reciprocity	TN
Iowa	364	5/1/2012		8/5/2010	WW, RCRA, UST		NELAP	IA
Kansas	E-10277	10/31/2011		11/2/2010	DW, WW, RCRA, UST		NELAP	NJ
Kentucky DW	90010	12/31/2010		4/13/2010	DW	WW, RCRA	Reciprocity	TN
Kentucky UST	16	10/16/2011		11/3/2010	UST		Audit	A2LA
Louisiana	Agency ID 30792	6/30/2011		11/22/2010	WW, RCRA, UST, AIR		NELAP	NJ
Maine	TN0002	7/5/2011		8/4/2009	DW, WW	RCRA, UST	Reciprocity	TN, NJ
Maryland	324	12/31/2010		2/16/2010	DW		Reciprocity	TN
Massachusetts	M-TN003	6/30/2011	7/1/10	9/28/2010	DW,WW	RCRA, UST	Reciprocity	TN
Michigan	9958	6/16/2013		8/31/2010	DW	WW, RCRA, UST	Reciprocity	TN
Minnesota	047-999-395	12/31/2011		11/3/2010	WW, RCRA, UST		Audit	MN
Mississippi	None	6/16/2013		9/28/2010	DW	WW, RCRA, UST	NELAP	NJ
Missouri	340	6/16/2013		9/28/2010	DW	WW, RCRA, UST	NELAP	NJ
Montana	CERT0086	Renewal	Renewal	7/16/2007	DW	WW, RCRA, UST	Reciprocity	TN
Nebraska	NA	6/30/2011		8/31/2010	DW	WW, RCRA, UST	Reciprocity	TN
Nevada	TN-03-2002-34	7/31/2011	Extended	8/19/2010	WW, DW, RCRA, UST		NELAP	NJ
New Hampshire	2975	5/20/2011	Jul-10	7/8/2010	DW, WW	RCRA, UST	NELAP	NJ
New Jersey - NELAP	TN002	6/30/2011		7/8/2010	DW, WW, RCRA, UST, AIR		NELAP	NJ
New Mexico	None	6/30/2011		7/9/2010	DW	WW, RCRA, UST	NELAP	NJ

New York	11742	4/1/2011	6/3/10	6/9/2010	WW, RCRA, UST, AIR		NELAP	NJ
North Carolina DW	DW21704	7/31/2011		8/5/2010	DW		Audit	NC
North Carolina	Env375	12/31/2010		1/15/2010	WW, RCRA, UST		Audit	NC
North C. Aquatic Tox	41	11/1/2011		11/22/2010	Aquatic Toxicity		Audit	NC
North Dakota	R-140	6/30/2011		7/23/2010	DW, WW, RCRA		Reciprocity	TN, WI
Ohio VAP	CL0069	4/14/2011	Jan-08	6/2/2009	WW, RCRA, UST, AIR		Audit	OH
Oklahoma	9915	8/31/2011		11/3/2010	WW, RCRA, UST, BIOASSAY		NELAP	NJ
Oregon	TN200002	1/15/2011		2/16/2010	DW, WW, RCRA, UST		NELAP	NJ
Pennsylvania	68-02979	12/31/2010		1/15/2010	DW, WW, RCRA, UST		NELAP	NJ
Rhode Island	221	12/31/2010		2/16/2010	DW, Env. Lead	WW, RCRA, UST	Reciprocity	TN, AIHA
South Carolina	84004	6/30/2011		11/22/2010	WW, RCRA, UST		NELAP	NJ
South Dakota	Pending	Pending						
Tennessee DW	2006	6/16/2013		7/23/2010	DW	WW, RCRA, UST	Audit	TN
Tennessee DW Micro	2006	10/12/2012		2/16/2010	DW Micro		Audit	TN
Texas Mold	LAB0152	3/10/2011		5/7/2007	MOLD		NA	TX
Texas - Env	T 104704245-07-TX	10/31/2011		11/3/2010	DW, WW, RCRA, AIR		Reciprocity	NJ
Utah	6157585858	6/30/2011		8/5/2010	DW, WW, RCRA, UST		NELAP	NJ
Vermont	VT2006	1/5/2011	Jan-10	1/15/2010	DW	WW, RCRA, UST	Reciprocity	TN
Virginia	109	6/30/2011		7/8/2010	DW	WW, RCRA, UST	NELAP	NJ
Washington	C1915	8/19/2011	9/18/08	11/3/2010	DW, WW, RCRA, UST, AIR		Audit	A2LA
West Virginia	233	2/28/2011		4/9/2010	WW, RCRA, UST		Audit	WV
Wisconsin	998093910	8/31/2011		9/14/2009	WW, RCRA, UST		Audit	WI
Wyoming	A2LA	11/30/2011		7/23/2010	UST	WW, RCRA	Audit	A2LA
Other Agencies								
A2LA	1461.01	11/30/2011	4/30/2010	7/23/2010	DW, WW, RCRA, UST, AIR, MICRO		Audit	A2LA
AIHA*	100789	6/1/2012		6/1/2010	IHLAP, ELLAP, EMLAP		Audit	AIHA
DOD	1461.01	11/30/2011		3/8/2010	RCRA, UST		Audit	A2LA
EPA	TN00003	None			Cryptospiridium		Audit	EPA
USDA	S-67674	8/7/2012		8/7/2009	Quarantine Permit		Audit	USDA

(1) A2LA = American Association for Laboratory Accred.
(2) AIHA = American Industrial Hygiene Association
(3) NELAP = National Environmental Laboratory Accred. Program
(4) IHLAP = Industrial Hygiene Laboratory Accred. Program
(5) ELLAP = Environmental Lead Laboratory Accred. Program
(6) EMLAP = Environmental Microbiology Laboratory Accreditation Program
(7) USDA = United States Department of Agriculture
(8) Approved Programs = The state does not have a formal certification program.
(9) Pending = The state is processing our application.
(10) EPA = Environmental Protection Agency

1.0 SIGNATORY APPROVALS

SAMPLING PROTOCOL QUALITY ASSURANCE MANUAL

APPENDIX III TO THE ESC QUALITY ASSURANCE MANUAL

for

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2.0 APPENDIX TABLE OF CONTENTS

3.0 SCOPE AND APPLICATION

This appendix discusses the standard practices and procedures utilized by ESC personnel for site selection and sample collection of various matrices. Topics addressed include field QA/QC procedures, together with equipment care and calibration for field sampling activities. Proper collection and handling of samples is of the utmost importance to insure that collected samples are representative of the sampling site. With this goal, proper sampling, handling, preservation, and quality control techniques for each matrix must be established and strictly followed. Precise identification of the collected samples and complete field documentation including a chain of custody are also vital.

ESC Lab Sciences does not provide sampling services for Industrial Hygiene and Environmental Lead analyses. We do require that all samples collected for these programs be sampled using the guidelines established by NIOSH, OSHA or other published protocol.

In addition, ESC Lab Sciences personnel do not conduct sampling in conjunction with the Ohio Voluntary Action Program (VAP).

4.0 LIST OF SAMPLING CAPABILITIES

• Parameter Group	• Sample Source
Extractable Organics	Surface water, wastewater, groundwater, stormwater runoff, drinking water, sediments, soils, chemical/ hazardous wastes, domestic wastewater sludge, hazardous waste sludge
Volatile Organic Compounds (VOCs)	Surface water, wastewater, groundwater, stormwater runoff, drinking water, sediments, soils, chemical/ hazardous wastes, domestic wastewater sludge, hazardous waste sludge
Metals	Surface water, wastewater, groundwater, stormwater runoff, drinking water, sediments, soils, chemical/ hazardous wastes, domestic wastewater sludge, hazardous waste sludge
Inorganic Anions	Surface water, wastewater, groundwater, stormwater runoff, drinking water, sediments, soils, chemical/ hazardous wastes, domestic wastewater sludge, hazardous waste sludge
Organics	Surface water, wastewater, groundwater, stormwater runoff, drinking water, sediments, soils, chemical/ hazardous wastes, domestic wastewater sludge, hazardous waste sludge
Physical Properties	Surface water, wastewater, groundwater, stormwater runoff, drinking water, sediments, soils, chemical/ hazardous wastes, domestic wastewater sludge, hazardous waste sludge
Cyanide	Surface water, wastewater, groundwater, stormwater runoff, drinking water, sediments, soils, chemical/ hazardous wastes, domestic wastewater sludge, hazardous waste sludge
Microbiology	Surface water, groundwater, drinking water, wastewater
Macro Invertebrate Identification	Surface water, wastewater, sediments
Biotoxicity	Surface water and wastewater

5.0 GENERAL CONSIDERATIONS

The following procedures are used in all of ESC's sampling activities. These procedures must be considered in relation to the objectives and scope of each sampling event.

5.1 SELECTING A REPRESENTATIVE SAMPLING SITE

Selecting a representative sampling site is dependent upon the matrix to be sampled and type of analyses required. These matrix specific procedures are discussed in subsequent sections.

5.2 SELECTION AND PROPER PREPARATION OF SAMPLING EQUIPMENT

The type of sampling equipment to be used is specific to the sample matrix and the analyses to be conducted. These are discussed later in this section. Section 12.0 describes the equipment cleaning procedures utilized by ESC personnel.

5.3 SAMPLING PROCEDURES FOR INDUSTRIAL HYGIENE AND ENVIRONMENTAL LEAD SAMPLES

ESC does not provide sampling services for industrial hygiene and/or environmental lead analyses. Experienced laboratory personnel can assist with advice on sampling; however, the adequacy and accuracy of sample collection is the client's responsibility.

5.4 SAMPLING EQUIPMENT CONSTRUCTION MATERIALS

To prevent direct contamination or cross-contamination of the collected sample, great attention must be given to the construction material used for sampling equipment. Materials must be inert, non-porous and easy to clean. Preferred materials include Teflon®, glass, stainless steel and plastic. Plastics may not be used for collections where organics are the analytes of interest. Stainless steel may not be used where metallic compounds will be analyzed.

5.5 SELECTION OF PARAMETERS BEING ANALYZED

Parameters for analysis are usually dictated by and based on regulated monitoring conditions (i.e. NPDES or RCRA permits). If these do not apply, analyses are selected by ESC or the client based on federal regulations specific to the matrix being investigated.

5.6 ORDER OF SAMPLE COLLECTION

Unless field conditions demand otherwise, the order of sample collection is as follows:

1. Volatile organic compounds (VOCs)
2. Extractable Organics (includes Total Recoverable Petroleum Hydrocarbons [TRPH], Oil & Grease, Pesticides and Herbicides)
3. Total metals
4. Dissolved metals
5. Microbiological
6. Inorganic (includes Nutrients, Demand, and Physical Properties)
7. Radionuclides

5.7 SPECIAL PRECAUTIONS FOR TRACE CONTAMINANT SAMPLING

Many contaminants can be detected in the parts per billion or parts per trillion range and extreme care must be taken to prevent cross-contamination. Therefore, extra precautions apply where samples are collected for trace contaminants. These precautions include:

- A new pair of disposable latex gloves must be worn at each sampling location.
- Sample containers for samples suspected of containing high concentrations of contaminants shall be sealed in separate plastic bags immediately after collection and preservation.
- If possible, background samples and source samples should be collected by different field sampling teams. If different field teams are not possible, all background samples shall be collected first and placed in separate ice chests or shipping containers. Samples of waste or highly contaminated samples shall not be placed in the same container as environmental samples. Ice chests or shipping containers for source samples or samples that are suspected to contain high concentrations of contaminants are discarded after use.
- If possible, one member of the field team should handle all data recording, while the other members collect samples.
- When sampling surface waters, water samples should always be collected before sediment samples are collected.
- Sample collection activities should proceed from the suspected area of least contamination to the suspected area of greatest contamination.
- ESC personnel should use equipment constructed of Teflon®, stainless steel, or glass that has been properly pre-cleaned (Sections 12.3 & 12.4) for collecting samples for trace metals or organic compounds analyses. Teflon®, glass, or plastic is preferred for collecting samples where trace metals are of concern. Equipment constructed of plastic or PVC shall not be used to collect samples for trace organic compounds analyses.
- When fuel powered units are utilized, they will be placed downwind and away from any sampling activities.

- Monitoring wells with free product shall not be sampled for trace contaminant analysis.

5.8 SAMPLE HANDLING AND MIXING

Sample handling should be kept to a minimum. ESC personnel must use extreme care to avoid sample contamination. If samples are placed in an ice chest, personnel should ensure that sample containers do not become submerged or tip over as this may result in cross-contamination. Small sample containers (e.g., VOCs or bacterial samples) should be placed in airtight plastic bags to prevent cross-contamination.

Once a sample has been collected, it may have to be split into separate containers for different analyses. A liquid sample will be split by shaking the container or stirring the sample contents with a clean pipette or pre-cleaned Teflon® rod. Then the contents are alternately poured into respective sample containers. Items used for stirring must be cleaned in accordance with the guidelines set forth in Section 12.0. Samples for VOCs, Cyanide, Total Phenol, and Oil & Grease must be collected as discrete grabs.

A soil sample may be split but must first be homogenized as thoroughly as possible to ensure representative sub-samples of the parent material. This is accomplished using the quartering method. The soil is placed in a sample pan and divided into quarters. Each quarter is mixed separately then all quarters are mixed together. This is repeated several times until the sample is uniformly mixed. If a round bowl is used, mixing is achieved by stirring the material in a circular fashion with occasionally inversion of the material.

Soil and sediment samples collected for volatile organic compounds shall not be mixed. The appropriate sample container should be filled completely, allowing little to no headspace.

Moisture content inversely affects the accuracy of mixing and splitting a soil sample.

5.9 QUALITY CONTROL SAMPLES

Quality control samples must be collected during all sampling events to demonstrate that the sample materials have not been contaminated by sampling equipment, chemical preservatives, or procedures relating to the sample collection, transportation and storage. A summary of the recommended frequency for collecting field quality control samples is presented in the following:

5.9.1 Quality Control Samples

Number of samples	Precleaned equipment blank[1]	Field cleaned equipment blank	Trip blank (VOCs)	Duplicate
10 or more	minimum of 1 then 5%	minimum of 1 then 5%	one per cooler[2]	minimum one then 10%[3]
5 - 9	one	one	one per cooler[2]	one
less than 5	one	one	one per cooler[2]	Not required, but recommend a minimum of one. USACE projects require one. Client specific QAPP requirements must be considered.

[1] Pre-cleaned blanks are to be collected after the initial decontamination procedure has been completed but before the first sample is collected. Only one pre-cleaned or field-cleaned blank is required if less than 10 samples are collected. Only analyte-free water as defined in this document will be used in the preparation of any field and/or equipment blank.

[2] Where VOC methods are analyzed simultaneously, such as 601/602, only one (1) trip blank is required per cooler.

[3] Duplicate samples are collected for all VOC samples.

5.10 VOLATILE ORGANIC COMPOUND SAMPLING

Water Samples

Generally, groundwater, drinking water and wastewater samples for the analysis of volatile organic compounds are collected in duplicate pre-labeled 40mL vials. During bottle kit preparation in the laboratory, 200µL of concentrated HCl is added to each clean and empty vial. A Teflon® septum is placed in each cap and a cap is placed securely on each vial.

The sampler should check the water being sampled for residual chlorine content. This is done with residual chlorine testing strips. If no chlorine is present, the prepared vials may be filled as needed. If residual chlorine is present, add one crystal of sodium thiosulfate ($Na_2S_2O_3$) to each vial prior to sampling.

To fill the vial properly, the sample is poured slowly down the inside wall of the vial until a convex meniscus is formed. Care should be taken to minimize turbulence. The cap is then applied to the bottle with the Teflon® side of the septum contacting the sample. Some overflow is lost; however air space in the bottle should be eliminated. Check for air bubbles by inverting the capped vial and tapping against the heel of the hand. This will dislodge bubbles hidden in the cap. If any bubbles are present, repeat the procedure. If unsuccessful, discard the vial and re-sample with a new preserved vial and septum. At a minimum, duplicate vials should always be collected from each sample location.

For analysis using EPA Method 524.2, samples that are suspected to contain residual chlorine, 25mg of ascorbic acid per 40mL of sample is added to each sample vial prior to sampling. Additionally, if analytes that are gases at room temperature (i.e. vinyl chloride, etc.) or any of the analytes in following table are not to be determined, 3mg of sodium thiosulfate is recommended for use to remove residual chlorine during sampling. If residual chlorine is present in the field sample at >5mg/L, then add additional 25mg or ascorbic acid or 3mg of sodium thiosulfate for each 5mg/L of residual chlorine present. Sample vials are then filled as previously described. Following collection and dechlorination, Method 524.2 samples are adjusted to a pH of <2 with HCl.

Acetone	Acrylonitrile	Allyl chloride
2-Butanone	Carbon disulfide	Chloroacetonitrile
1-Chlorobutane	t-1,2-Dichloro-2-butene	1,1-Dichloropropanone
Diethyl ether	Ethyl methacrylate	Hexachloroethane
2-Hexanone	Methacrylonitrile	Methylacrylate
Methyl iodide	Methylmethacrylate	4-Methyl-2-pentanone
Methyl-tert-butyl ether	Nitrobenzene	2-Nitropropane
Pentachloroethane	Propionitrile	Tetrahydrofuran

For more detailed instructions, see the published method.

Soil Samples

Option 1 – Core Sampling Device
Soil samples for volatile organic analysis should be sampled using traditional core sampling methods. Once the core sample is collected, additional samples should be taken using an Encore™ sampler, either 5g or 25g, capped, sealed, and immediately cooled. The holding time for this method is 48 hours.

Option 2 – Pre-weighed Vial
In the other option for volatile soil sampling, 40mL vials with cap, Teflon® lined septum, preservative (5mL sodium bisulfate solution), and stir bar are pre-weighed, either by the user or the manufacturer. The vial is weighed on a balance capable of measuring to 0.01g and labeled with the pre-weighed value. In the field, place roughly 5g of sample into a pre-weighed vial, cap, and then immediately place on ice to achieve a temperature of 4ºC. Exact soil weights can be measured using the pre-weight of the vial and the post-sampling weight. The difference represents the actual weight of the soil sample. The holding time for this method is 14 days.

Unless specifically permitted by the regulatory authority, VOC samples (liquid or solid) should never be mixed or composited.

5.11 OIL AND GREASE SAMPLING

Aqueous samples collected for oil and grease analyses must be collected as discrete grab samples. Sample containers should not be rinsed with sample water prior to sample collection and samples should be collected directly into the sample container. Intermediate vessels should only be used where it is impossible to collect the sample directly into the sample container and, in this case, only Teflon® beakers should be used. Samples should be taken from well-mixed areas.

5.12 CYANIDE SAMPLING

Cyanide is a very reactive and unstable compound and should be analyzed as soon as possible after collection. Samples shall be collected in polyethylene or glass containers and shall be pretreated and preserved in the manner specified in the following paragraphs.

5.12.1 Test for Oxidizing Agents

1. Test the sample with residual chlorine indicator strips.
2. Add a few crystals of ascorbic acid and test until negative.
3. Add an additional 0.6 grams of ascorbic acid for each liter sampled to remove residual chlorine.
4. Preserve the pretreated sample by to a pH > 12.0 with NaOH and cool to 4 $\pm$ 2°C. Verify the pH of the samples as per Section 14.2.
5. Equipment blanks must be handled in the same manner as described in steps 1 through 4.

5.12.2 Test for Sulfide

1. Test the sample for sulfide using the sulfide test strip;(formally HACH KIT).
2. If sulfide is not removed by the procedure below, the sample must be preserved with NaOH to pH > 12.0 and analyzed by the laboratory within 24 hours.
3. Sulfide should be removed by filtering visible particulate. Retain filter (filter #1).
4. Remove the sulfide by adding lead carbonate powder to the filtrate to cause the sulfide to precipitate out.
5. Test the filtrate for the presence of sulfide. If sulfides are present, repeat steps 1 and 4 until no sulfides are shown present.
6. The precipitate can now be filtered from the sample and this filter is discarded.
7. The sample is then reconstituted by adding the sediment collected on filter #1 back to the filtrate.
8. Preserve the pretreated sample to a pH > 12.0 with NaOH and cool to 4 $\pm$ 2°C. Verify the pH of the samples as per Section 14.2

9. Equipment blanks must be handled in the same manner as described in steps 1 through 9.

5.13 BIOMONITORING SAMPLING

Aqueous samples collected for Bioassay can be collected in either glass or HDPE plastic. There is no chemical preservation for this type of sample and the required volume varies with each type of analysis. Following sampling, all samples must be cooled to 4°C and can be held for a maximum of 36 hours from the time of collection. Grab and composite sample protocols are utilized for acute and chronic bioassays and are chosen according to permit requirements. Samples should be collected with minimum aeration during collection and the container should be filled allowing no headspace. Samples may be shipped in one or more 4L (l gal.) CUBITAINERS® or unused plastic "milk" jugs. All sample containers should be rinsed with source water before being filled with sample. Containers are not reused. If the sample is a chlorinated effluent, total residual chlorine must be measured immediately following sample collection.

5.14 PROCEDURES FOR IDENTIFYING POTENTIALLY HAZARDOUS SAMPLES

Any sample either known, or suspected, to be hazardous shall be identified as such on the chain of custody. Information explaining the potential hazard (i.e., corrosive, flammable, poison, etc.) shall also be listed.

5.15 COLLECTION OF AUXILIARY DATA

All auxiliary data shall be entered in the field records. Auxiliary data relative to a particular sampling location should be recorded concurrent with the sample event. Matrix specific auxiliary data are discussed later in this section.

5.16 TIME RECORDS

All records of time shall be kept using local time in the military (24 hour) format and shall be recorded to the nearest minute.

5.17 REFERENCES

ESC maintains copies of the various sampling references in the sample equipment room. Pertinent pages of these documents may be photocopied and taken to the field during sampling investigations. A bibliography of references used in the development of this section is presented in Section 17.

6.0 ANCILLARY EQUIPMENT AND SUPPLIES

The equipment used to collect samples and conduct necessary purging activities is listed in subsequent sections for each type of sample. However, Section 6.1 lists some of the ancillary field equipment and instruments that may be required.

6.1 ANCILLARY EQUIPMENT AND SUPPLIES

Flow Measurement:	ISCO Continuous Flow Meters 3230, 3210, 2870; Flo-Poke pipe insert
Personal Protective Equipment:	Hard Hats, Face Shields, Half- and Full-Face Respirators, Rubber and Latex Gloves, Tyvex protective coveralls, rubber boots, safety glasses
Field Instruments:	Water Level Indicator, Continuous Recording pH Meter, Portable pH/Temperature Meters, Hach DR-100 Chlorine Analyzer, Hach CEL/700 Portable Laboratory, YSI Field Dissolved Oxygen/Temperature Meter w/ Submersible Probe, Portable Field Specific Conductance Meter, Hach 2100P Portable Turbidimeter
Chemical Supplies & Reagents:	Deionized Water, Tap Water, Liquinox Detergent, Isopropanol, Nitric Acid, Hydrochloric Acid, Sulfuric Acid, Sodium Hydroxide, Ascorbic acid, Sodium Thiosulfate, Ascorbic Acid, Zinc Acetate, pH calibration buffers (4.0, 7.0, and 10.0), Hach Sulfide Kit, lead carbonate powder, Specific Conductance Standard, Turbidity Standards
Tools:	Pipe Wrench, Bung Wrench, Crowbar, Hammer, Assorted Screwdrivers, Tape Measures, Channel Lock Pliers, Vise Grip Pliers, Duct Tape, Vinyl Pull Ties
Miscellaneous:	Cellular Phones, Pagers, Walkie Talkies, 12 Volt Batteries, Flashlights, Extension Cords, Brushes, Plastic sheeting, Fire extinguishers, Water Squeeze Bottles, First Aid Kit, lengths of rigid PVC conduit, aquatic sampling nets (Wildco)

7.0 WASTEWATER SAMPLING

7.1 SAMPLING EQUIPMENT

Type	Use	Materials	Permissible Parameter Groups
Continuous Wastewater Samplers-Peristaltic Pump	Sampling	Tygon tubing; glass or plastic sample container	All parameter groups except oil & grease, extractable organics, and VOCs
	Sampling	Teflon® tubing; glass sample container	All parameter groups except VOCs

7.2 GENERAL CONSIDERATIONS

The procedures used by ESC are generally those outlined in the NPDES Compliance Inspection Manual. Additional guidance is given in the EPA Handbook for Monitoring Industrial Wastewater. Some important considerations for obtaining a representative wastewater sample include:

- The sample should be collected where the wastewater is well mixed.
- Samples should not be collected directly from the surface/bottom of the wastestream.
- In sampling from wide conduits, cross-sectional sampling should be considered.
- If manual compositing is employed, the individual sample bottles must be thoroughly mixed before pouring the individual aliquot into the composite container.

7.3 SAMPLING SITE SELECTION

Wastewater samples should be collected at the location specified in the NPDES or sewer use permit if such exists. If the specified sampling location proves unacceptable, the project manager shall select an appropriate location based on site-specific conditions. An attempt should be made to contact the regulating authorities for their approval. The potential for this type of issue highlights the need for a site inspection prior to the scheduled sampling event.

7.3.1 Influent

Influent wastewaters should be sampled at points of high turbulence and mixing. These points are: (1) the upflow siphon following a comminutor (in absence of grit chamber); (2) the upflow distribution box following pumping from main plant wet well; (3) aerated grit chamber; (4) flume throat; or (5) pump wet well when the pump is operating. Raw wastewater samples should be collected upstream of sidestream returns.

7.3.2 Effluent

Effluent samples should be collected at the site specified in the permit or, if no site is specified, at the most representative site downstream from all entering wastewater streams prior to final discharge.

7.3.3 Pond and Lagoon Sampling

Composite samples of pond and lagoon effluent are preferred over grabs due to the potential for ponds and lagoons to short circuit the projected flow paths. However, if dye studies or facility data indicate a homogeneous discharge, grab samples may be taken.

7.4 SAMPLING TECHNIQUES: GENERAL

The choice of a flow-proportional or time-proportional composite sampling program depends upon the variability of flow, equipment availability, sampling point configuration and accessibility. Flow metered sampling is necessary for complete wastewater characterization and should be utilized where possible. If not feasible, a time-proportional composite sample is acceptable.

A time-proportional composite sample consists of aliquots collected at constant time intervals and can be collected either manually or with an automatic sampler.

A flow-proportional composite sample consists of aliquots collected automatically at constant flow intervals with an automatic sampler and a flow-measuring device. Prior to flow-proportional sampling, the flow measuring system (primary flow device, totalizer, and recorder) should be examined. The sampler may have to install flow measurement instrumentation if automatic sampling is to be used.

7.5 USE OF AUTOMATIC SAMPLERS

7.5.1 General

Automatic samplers are used when several points are sampled at frequent intervals, with limited personnel, or when a continuous sample is required. Automatic samplers used by ESC must meet the following requirements:

- Must be properly cleaned to avoid cross-contamination from prior sampling events.
- No plastic or metal parts shall come into contact with the sample when parameters to be analyzed could be impacted by these materials.
- Must be able to provide adequate refrigeration. Commercially available ice is placed in the sampler base and packed around the container approximately half way up the sample container.
- Must be able to collect a large enough sample for all required analyses. Composite sample containers (glass or plastic) hold up to 10 liters.
- A minimum of 100 milliliters should be collected each time the sampler is activated.
- Should provide a lift of at least 20 feet and be adjustable so that sample volume is not a function of pumping head.
- Pumping velocity must be adequate to transport solids without settling.
- The intake line must be purged a minimum of one time before each sample is collected.
- The minimum inside diameter of the intake line should be 1/4 inch.
- Have a power source adequate to operate the sampler for 48 hours at 15-minute sampling intervals.
- Facility electrical outlets may be used if available.
- Facility automatic samplers may be used for conventional parameters if they meet ESC QA/QC criteria.

Specific operating instructions, capabilities, capacities, and other pertinent information for automatic samplers presently used by ESC are included in the respective operating manuals and are not presented here.

All data relative to the actual use of automatic equipment on a specific job is recorded in sampling logbooks.

7.5.2 Equipment Installation

7.5.2.1 Conventional Sampling

Automatic samplers may be used to collect time-proportional composite or flow-proportional composite samples. In the flow-proportional mode, the samplers are activated by a compatible flow meter. Flow-proportional samples can also be collected using a discrete sampler and a flow recorder and manually compositing the individual aliquots in flow-proportional amounts.

Installation procedures include cutting and installing the proper length of tubing, positioning it in the wastewater stream, and sampler programming. All new tubing (Dow® Corning Medical Grade Silastic, or equal, in the pump and Tygon®, or equal, in the sample train) will be used for each sampler installation.

For a time-proportional composite, the sampler should be programmed to collect 100mL samples at 15-minute intervals into a refrigerated 10L plastic or glass jug, as appropriate for the particular parameters being analyzed.

For a flow-proportional composite, the sampler should be programmed to collect a minimum of 100mL for each sample interval. The sampling interval should be based on the flow of the waste stream.

7.5.3 Automatic Sampler Maintenance, Calibration, and Quality Control

To ensure proper operation of automatic samplers, the procedures outlined in this section shall be used to maintain and calibrate ESC automatic samplers. Any variance from these procedures will be documented.

Proper sampler operation will be checked by ESC personnel prior to each sampling event. This includes checking operation through three cycles of purge-pump-purge; checking desiccant and replacing if necessary; checking charge date on NiCad batteries to be used; and repairing or replacing any damaged items.

Prior to beginning sampling, the purge-pump-purge cycle shall be checked at least once. The sample volume will be calibrated using a graduated cylinder at least twice, and the flow pacer that activates the sampler shall be checked to be sure it operates properly.

Upon return from a field trip, the sampler shall be examined for damage. The operation will be checked and any required repairs will be performed and documented. The sampler will then be cleaned as outlined in Section 12.

7.6 MANUAL SAMPLING

Manual sampling is normally used for collecting grab samples and for immediate in-situ field analyses. Manual sampling may also be used when it is necessary to evaluate unusual waste stream conditions. If possible, manually collected samples should be collected in the actual sample container that will be submitted to the laboratory. This minimizes the possibility of contamination from an intermediate collection container.

Manual samples are collected by (1) submerging the container neck first into the water; (2) inverting the bottle so that the neck is upright and pointing into the direction of wastewater flow; (3) quickly returning the sample container to the surface; (4) shake to rinse. Pour the contents out downstream of sample location; (5) collect sample as described in steps 1, 2, and 3; pour out a few mLs of sample downstream of sample collection. This allows for addition of preservatives and sample expansion.

Exceptions to the above procedure occur when preservatives are present in the sampling container or when oil & grease, microbiological, and/or VOC analyses are required. In these cases, sample shall be collected directly into the container with no pre-rinsing.

If the water or wastewater stream cannot be physically or safely reached, an intermediate collection container may be used. This container must be properly cleaned (Section 12) and made of an acceptable material. A separate collection container should be used at each sampling station to prevent cross-contamination between stations. The sample is collected by lowering a properly cleaned Teflon®, plastic, or glass collection vessel into the waste stream. The intermediate vessel may be lowered by hand, pole or rope.

7.7 SPECIAL SAMPLE COLLECTION PROCEDURES

7.7.1 Trace Organic Compounds and Metals

Due to the ability to detect trace organic compounds and metals in extremely low concentrations, care must be taken to avoid contamination of the sample. All containers, composite bottles, tubing, etc., used in sample collection for trace organic compounds and metals analyses should be prepared as described in Section 12.

Personnel handling the sample should wear a new pair of disposable latex gloves with each set of samples collected to prevent cross-contamination. A more detailed discussion is given in Section 5.7 under special precautions for trace contaminant sampling.

7.7.2 Bacterial Analysis

Samples for bacterial analysis will always be collected directly into the prepared glass or plastic sample bottle. The sample bottle should be kept closed until immediately prior to sampling and never rinsed with sample. When the container is opened, care should be taken not to contaminate the cap or the inside of the bottle. The bottle should be held near the base and plunged, neck downward, below the surface and turned until the neck points upward and upstream. The bottle should be filled to within one-inch of the top and capped immediately.

Section 14 presents preservation procedures and holding times. As holding times are limited to 6 hours for microbiological analyses, special arrangements may be required to ensure that these samples reach the laboratory within this timeframe.

7.7.3 Immiscible Liquids/Oil and Grease

Oil and grease may be present in wastewater as a surface film, emulsion, solution, or a combination of these forms. A representative sample for oil and grease analysis is difficult to collect. The sampler must carefully evaluate the location of the sampling point to find the area of greatest mixing. Quiescent areas should be avoided.

Because losses of oil and grease will occur on sampling equipment, collection by composite sampler is not practical. Intermediate sampling vessels should not be used if possible. If intermediate collection vessels are required they should be made of Teflon® and be rinsed with the sample three times before transferring any sample to the sample container. Sample containers, however, should never be rinsed.

7.7.4 Volatile Organic Compounds Analyses

Water samples to be analyzed for volatile organic compounds are collected in 40mL pre-preserved (200uL of concentrated HCl) vials with screw caps. A Teflon®-silicone septum is placed in each cap prior to the sampling event. The Teflon® side must be facing the sample side.

Sampling containers with preservatives are pre-labeled prior to any field activities to reduce the chances of confusion during sampling activities. A complete list of sample preservatives, containers, holding times, and volumes is found in Section 14.

The sampler should check the water to be sampled for chlorine. This is done with residual chlorine indicator strips. If no chlorine is found, the vials may be filled. If residual chlorine is present, the sampling and preservation procedures listed in Section 5.10 of this manual must be performed.

7.8 AUXILIARY DATA COLLECTION

While conducting wastewater sampling, the following information may also be gathered:

- Field measurements -- pH, DO, conductivity, temperature
- Flows associated with the samples collected -- continuous flows with composite samples and instantaneous flows with grab samples
- Diagrams and/or written descriptions of the sample locations
- Photographs of pertinent wastewater-associated equipment, such as flow measuring devices, treatment units, etc.
- Completion of applicable forms required during specific investigations.

All observations, measurements, diagrams, etc., will be entered in field logbooks or attached thereto.

8.0 SURFACE WATER AND SEDIMENT SAMPLING

8.1 EQUIPMENT

Equipment Type	Use	Material	Permissible Parameter Groups
Surface Water Sampling			
Kemmerer Sampler	Depth sampling	PVC	All parameter groups except extractable organics, VOCs, and oil & grease
Automatic Samplers	Sampling	Teflon®	All parameter groups except VOCs, oil & grease, & micro
	Sampling	PVC	All parameter groups except extractable organics, VOCs, oil & grease, and micro
Sample Collection Container	Sampling	Stainless steel	All parameter groups
Bailers	Sampling	Teflon®	All parameter groups
	Sampling	PVC	All parameter groups except extractable organics, VOCs, and oil & grease
Sediment Sampling			
Hand Augers	Sampling	Carbon Steel	Demand, nutrients, and extractable organics (for hard packed soils only)
Sediment Core Sampler	Sampling	Stainless Steel, Teflon®	All parameter groups
Encore™	Sampling	Teflon®	VOC Sediment/soil
Scoops	Sampling	Teflon® coated	All parameter groups
Mixing Bowl	Compositing	Glass	All parameter groups except VOCs
Spoons, spatula	Sampling, compositing	Stainless Steel	All parameter groups

8.2 GENERAL

Selection of surface water sampling locations for water quality studies are determined by the objective of the study and waterway type. Factors that impact and alter water quality and characteristics (dams, bridges, discharges, etc.) must be considered. Accessibility is important.

8.3 SAMPLE SITE SELECTION

Fresh water environments are commonly divided into two types: (1) rivers, streams, and creeks; and (2) lakes, ponds, and impoundments. Since these waterways differ considerably in general characteristics, site selection must be adapted to each.

Prior to conducting a sampling event, an initial survey should be conducted to locate prime sampling points. Bridges and piers provide ready access to sampling points across a body of water. However, they should only be used when at otherwise acceptable locations and are found not to be detrimentally impacting stream characteristics.

If wading for water samples must be done, caution should be used to avoid disturbing bottom deposits that could result in increased sediment in the sample. Shallow areas may be best for sediment sampling.

8.3.1 Rivers, Streams, and Creeks

Sampling sites should be located in areas possessing the greatest degree of cross-sectional homogeneity. Such points are easily found directly downstream of a riffle or rapid. These locations are also good for sediment sampling. In the absence of turbulent areas, a site that is clear of immediate point sources, such as tributaries and effluent discharges, may be used.

Typical sediment deposition areas are located at the inside of river bends and down-stream of islands or other obstructions. Sites immediately upstream or downstream from the confluence of two streams or rivers should be avoided due to inadequate mixing of the combining flows. Also, backflow can upset normal flow patterns.

Great attention should be given to site selection along a stream reach:

- Sites should be spaced at intervals based on time-of-water-travel. Sampling sites may be located at about one-half day time-of-water-travel for the first three days downstream of a waste source for the first six sites and then approximately one day for the remaining distance.
- If the study data is for comparison to previous study data, the same sampling sites should be used.
- Sites should be located at marked physical changes in the stream channel.
- Site locations should isolate major discharges as well as major tributaries.

Dams and weirs usually create quiet, deep pools in river reaches that would otherwise be swift and shallow. When times of travel through them are long, sites should be established within them.

Some structures, such as dams, permit overflow that may cause significant aeration of oxygen deficient water. Sites should be located short distances upstream and downstream of these structures to measure the rapid, artificial increase in dissolved oxygen (DO), which is not representative of natural aeration.

A minimum of three sites should be located between any two points of major change in a stream, even if the time-of-travel between the points of change is short. Major changes include, but are not limited to, a waste discharge, a tributary inflow, or a significant change in channel characteristics. Sampling three sites is also important when testing rates of change of unstable constituents. Results from two of three sites will usually support each other and indicate the true pattern of water quality in the sampled zone. If the effect of certain discharges or tributary streams of interest is desired, sites should be located both upstream and downstream of these points.

Due to the tendency of the influent from a waste discharge or tributary to slowly mix, cross-channel, with the main stream, it is nearly impossible to measure their effect immediately downstream of the source. Thus, samples from quarter points may miss the wastes and only indicate the quality of water above the waste source. Conversely, samples taken directly in the stream portion containing the wastes would indicate excessive effects of the wastes with respect to the river as a whole.

Tributaries should be sampled as near the mouth as possible. Often, these may be entered from the main stream for sampling by boat. Care should be taken to avoid collecting water from the main stream that may flow back into the tributary as a result of density differences created by temperature, salinity, or turbidity differences.

Actual sampling locations will vary with the size and amount of turbulence in the stream or river. Generally, with streams less than 20 feet wide, well mixed areas and sampling sites are readily found. In such areas, a single grab sample taken at mid-depth at the center of the channel is adequate. A sediment sample can also be collected at the center of the channel. For slightly larger streams, at least one vertical composite should be taken from mid-stream. It should be composed of at least one sub-surface, mid-depth, and above the bottom sample. Dissolved oxygen, pH, temperature, conductivity, etc. should be measured on each aliquot of the vertical composite. Several locations should be sampled across the channel width on the larger rivers. Vertical composites across the channel width should be located proportional to flow, i.e., closer together toward mid-channel where flow is greater and less toward the banks where the flow proportionally lower.

The field crew will determine the number of vertical composites and sampling depths for each area. They should base their decisions upon two considerations.

1. The larger the number of sub-samples, the more nearly the composite sample will represent the water body.
2. Taking sub-samples is time consuming and expensive, and increases the chance of contamination.

A number of sediment samples should be collected along a cross-section of a river or stream to adequately characterize the bed material. The normal procedure is to sample at quarter points along the cross-section of the site. When the sampling technique or equipment requires that the samples be extruded or transferred at the site, they can be combined into a single composite sample. However, samples of dissimilar composition should not be combined. They should be kept separate for analysis in the laboratory. To ensure representative samples, coring tubes are employed. The quantity of each sub-sample that is composited shall be recorded.

8.3.2 Lakes, Ponds, and Impoundments

Lakes, ponds, and impoundments have a much greater tendency to stratify than rivers and streams. This lack of mixing requires that more samples be obtained from the different strata. Occasionally, extreme turbidity differences occur vertically where a highly turbid river enters a lake. This stratification is caused by temperature differences where the cooler, heavier river water flows beneath the warmer lake water. A temperature profile of the water column and visual observation of lake samples can detect these layers. Each layer of the stratified water column should be sampled.

The number of sampling sites on a lake, pond, or impoundment is determined by the objectives of the investigation dimensions of the basin. In small bodies of water, a single vertical composite at the deepest point may be sufficient. Dissolved oxygen, pH, temperature, etc., should be conducted on each vertical composite aliquot. In naturally formed ponds, the deepest point is usually near the center; in impoundments, the deepest point is usually near the dam.

In lakes and larger impoundments, several vertical sub-samples should be composited to form a single sample. These vertical sampling locations should be along a transaction or grid. The field crew will determine the number of vertical composites and sampling depths for each area. In some cases, separate composites of epilimnetic and hypolimnetic zones may be required. Additional separate composite samples may be needed to adequately represent water quality in a lake possessing an irregular shape or numerous bays and coves. Additional samples should always be taken where discharges, tributaries, agriculture, and other such factors are suspected of influencing water quality.

When collecting sediment samples in lakes, pond, and reservoirs, the sample site should be as near as possible to the center of the water mass, especially for impoundments of rivers or streams. Generally, coarser grained sediments are deposited at the headwaters of a reservoir, and the finer sediments are near the center. The shape, inflow pattern, bathymetry, and circulation affect the location of sediment sampling sites in large bodies of water.

8.3.3 Control Sites

The collection of samples from control sites is necessary to compile a basis of comparison of water quality. A control site above the point of interest is as important as the sites below, and must be chosen with equal care. Two or three sites above the waste inflow may be necessary to establish the rate at which any unstable material is changing. The time of travel between the sites should be sufficient to permit accurate measurement of the change in the material under consideration.

8.4 SAMPLING EQUIPMENT AND TECHNIQUES

8.4.1 General

Any equipment or sampling techniques used to collect a sample must not alter the integrity of the sample and must be capable of providing a representative sample.

8.4.2 Water Sampling Equipment/Techniques

The physical location of the collector will dictate the type of equipment needed to collect samples. Surface water samples may be collected directly into the sample container when possible. Pre-preserved sample containers shall never be used as intermediate collection containers. Samples collected in this manner shall use the methods specified in Section 7.6 of this manual. If wading into the stream is required, care should be taken not to disturb bottom deposits, which could be unintentionally collected, and bias the sample. Also, the sample should be collected directly into the sample bottle and **up current** of the wader. If wading is not possible or the sample must be collected from more than one depth, additional sampling equipment may be used. If sampling from a powerboat, samples must be collected upwind and upstream of the motor.

8.4.2.1 Sampling Procedure Using a Teflon® or PVC Bailer

If data requirements of surface water sampling do not necessitate sampling from a strictly discrete interval of the water column, Teflon® or PVC constructed bailers can be used for sampling. The type bailer used is dependent on the analytical requirements. A closed top bailer utilizing a bottom check valve will be sufficient for many surface water studies. Water is continually displaced through the bailer as it is lowered down through the water column until the specified depth is attained. At this point, the bailer is retrieved back to the surface. There is the possibility of contamination to the bailer as it is lowered through the upper water layers. Also, this method may not be successful in situations where strong currents are found or where a discrete sample at a specified depth is needed.

If depth specific, discrete samples are needed and the parameters do not require Teflon® coated sampling equipment, a standard Kemmerer sampler may be used. A plastic bucket can also be used to collect surface samples if parameters to be analyzed do not preclude its use. The bucket shall always be rinsed twice with the sample water prior to collection and the rinse water be disposed of downstream from the sample collection point. All field equipment will be cleaned using standard cleaning procedures.

8.4.2.2 Sampling Procedure Using a Kemmerer Sampler

Due to the PVC construction of the Kemmerer sampler, it shall not be used to collect samples for extractable organics, VOCs, and/or oil & grease analysis. The general collection procedure is as follows:

1. Securely attach a suitable line to the Kemmerer bottle.
2. Lock stoppers located at each end of the bottle on the open position. This allows the water to be drawn around the bottom end seal and into the cylinder at the specified depth.
3. The bottle is now in the set position. A separate "messenger" is required to activate the trip mechanism that releases the stopper and closes the bottle.
4. When the bottle is lowered to the desired depth, the messenger is dropped. This unlocks the trip mechanism and forces the closing of both end seals.
5. Raise the sampler, open one of the end seal, and carefully transfer the sample to the appropriate sample container.

8.4.2.3 Sampling Procedures Using Sample Collection Containers

In most cases, sample collection containers are used to collect surface water from easily accessible sampling points. This means that the sample is collected manually, always upstream of the sampling person's position. An extension may be added to the container to make the sampling point more accessible for manual sampling. Extensions can be constructed of aluminum, PVC, steel, or any other suitable material. The sample container is normally attached to the extension using a clamp, vinyl pull ties, or duct tape. Samples collected in this way are done so in the following manner:

1. Place the inverted sample container into the water and lower to the desired depth. Never use a pre-preserved container as an intermediate sample collection device.
2. Re-invert the container with the mouth facing into the direction of flow and at the appropriate depth to collect the desired sample.
3. Carefully raise the container to the surface and transfer to the appropriate container.

8.4.3 Sediment Sampling Equipment/Techniques

A variety of methods can be used to collect sediment samples from a streambed. ESC utilizes corers and scoops. Precautions must be taken to ensure that the sample collected is representative of the streambed. These methods are discussed in the following paragraphs.

8.4.3.1 Sediment Core Samplers

Core sampling is used to collect vertical columns of sediment from the stream or lakebed. Many types of coring devices are available for use depending on the depth of water from which the sample is obtained, the type of bottom material, and the length of the core to be collected. Some devices are weight or gravity driven while others are simple hand push tubes. These devices minimize the loss of fine particles and should always be used when collecting sediment samples from flowing waters.

Coring devices are particularly useful in pollutant monitoring because the shock wave created by sampler descent is minimized and the fines at the sediment-water interface are only slightly disturbed. The sample can be withdrawn primarily intact removing only the layers of interest. Core liners manufactured of Teflon® or plastic can be purchased. These liners reduce the possibility of contamination and can be delivered to the laboratory in the tube they were collected in. Coring devices sample small surface areas and small sample sizes and often require repetitive sampling to obtain a sufficient amount of sample. This is the primary disadvantage to these devices but they are recommended in the sampling of sediments for trace organic compounds or metals analyses.

When sampling sediments in shallow water, the direct use of a core liner is recommended. Stainless steel push tubes are also used because they provide a better cutting edge and higher tensile strength than Teflon® or plastic. One advantage to using the Teflon® or plastic tubes is the elimination of possible metals contamination of the sample from the core barrels or cutting heads. The length of the corer tube should correspond to the desired depth of the layer being sampled. In general, soft sediments adhere better to the inside of the tube and a larger diameter tube can be used. Coarser sediments require the use of a smaller diameter tube of two inches or less to prevent the sample from falling out of the tube. The inside bottom wall of the tube can be filed down to allow easier entry into the substrate.

When samples are obtained by wading, caution should be used to minimize disturbance in the area sampled. Core tubes are pushed directly down into softer substrates until four inches or less of the tube is above the sediment-water interface. A slight rotation of the tube may be necessary to facilitate ease of entry into harder substrates and reduce compaction of the sample. The tube is then capped and slowly extracted and the bottom of the corer is capped before it is pulled above the water surface.

Sub-sampling is performed for VOC samples using an Encore™ sampling device. This device is used to collect soil/sediment samples, while preventing container headspace. Once the core sample is collected, additional samples should be taken using an Encore™ sampler, either 5g or 25g, capped, sealed, and immediately chilled to 4ºC. The holding time for this sampling method is 48 hours. Alternatively, weigh 5g of sample into a pre-weighed vial (with a Teflon® lined screw cap) containing, 5mL sodium bisulfate solution and a magnetic stir bar, cap, and then ice to 4ºC. The holding time for this method is 14 days.

8.4.3.2 Scooping Samples

The easiest and quickest way to collect a sediment sample in shallow water is with a Teflon® coated scoop or stainless steel spoon. This type of sampling should be limited to quiescent (i.e., non-flowing) waters such as lakes or reservoirs.

8.4.3.3 Mixing

As specified in Section 5.8, sediment samples, collected for chemical analysis, should be thoroughly mixed (except for volatile organic compounds analysis) before being placed in the sample containers.

8.5 SPECIAL SAMPLE COLLECTION TECHNIQUES

8.5.1 Trace Organic Compounds and Metals

Samples for trace pollutant analyses in surface water should be collected by dipping the sample containers directly into the water. Sometimes samples are split for enforcement or quality control purposes. A sufficient volume of sample for all containers should be collected in a large glass container and then, while mixing, be alternately dispensed into the appropriate bottles. This cannot be done for volatile organic compound samples due to potential loss of volatile compounds.

Only Teflon® or stainless steel should be used in sediment sampling for trace contaminant analyses. Teflon® coring tubes are the preferred technique.

8.5.2 Bacterial Analysis

Samples for bacteriological examination must be collected in sterilized bottles and protected against contamination. The preferred technique is to collect sample directly into the sample bottle. Hold the bottle near the base and plunge, neck downward, below the surface. The container is then turned with the neck pointed slightly upward and the mouth directed toward the current. The bottle is filled to about ½ inch from the top and recapped immediately. While the bottle is open, extreme care should be used to protect both the bottle and stopper against contamination. The ½ inch air space is left in the bottle to facilitate subsequent shaking in the laboratory.

If sampling with an intermediate sampling device (i.e. bailer), the device shall be thoroughly rinsed with sample water prior to collecting the sample. For this reason, microbiological samples are among the final samples collected from a sampling site. Begin pouring sample out of the sampling device before collecting into the sterilized container. Continue pouring sample out of the device, place the container under the flowing stream, and fill the container to ½ inch from the top. Flow should remain continuous before and during the filling process.

When sampling from a bridge, the sterilized sample bottle can be weighted and lowered to the water on a rope. Collectors must be careful not to dislodge debris from the bridge that could fall into the bottle.

8.6 AUXILIARY DATA COLLECTION

A field logbook will be used to record data pertinent to sampling activities. This data shall describe all sampling locations and techniques, list photographs taken, visual observations, etc. Visual observations of sample site conditions, including weather and overall stream conditions, recorded during the investigation can be valuable in interpreting water quality study results.

8.7 SPLIT AND DUPLICATE SAMPLE COLLECTION

Split samples measure variability between analysts, methods, and laboratories and are taken as subsamples from a single sample. This is unlike duplicate samples that measure variability inherent in the collection method or waste stream and are obtained in close succession during the same sampling event.

8.7.1 Split Sample Collection

Split samples are collected as follows:

1. Sample must be collected in a properly cleaned container constructed of acceptable materials. The volume should be more than twice the volume required for one sample.
2. Add appropriate preservative where required.
3. Mix thoroughly.
4. Alternately, decant sample into subsample containers in increments of approximately 10% of total subsample volume until containers are full.
5. Seal the sample containers with appropriate, airtight caps.
6. Label each sample container with a field number and complete a chain of custody.

NOTE: Volatile organic samples shall not be collected in this manner. Samples for VOC's must be collected as simultaneous, discrete grab samples.

8.7.2 Duplicate Sample Collection

1. Collect two samples in rapid succession.
2. Preserve where required.
3. Mix thoroughly.
4. Seal the sample containers with appropriate, airtight caps.
5. Label each sample container with a field number and complete a chain of custody.

9.0 GROUNDWATER AND DRINKING WATER SAMPLING

9.1 GROUNDWATER AND DRINKING WATER SAMPLING EQUIPMENT

Equipment type	Purpose	Component(s)	Allowable Parameter Groups
Bailers (disposable and non-disposable)	Purging	Teflon® & SS	All parameter groups
	Sampling	Teflon®	All parameter groups
Peristaltic Pump[1]	Purging[2]	Tygon Tubing	All parameter groups except organics
	Purging	Teflon®	All parameter groups
		Silastic Rubber	All parameter groups except organics
ISCO Bladder Pump[3]	Sampling	Stainless Steel, Teflon®	All parameter groups

[1] New or dedicated tubing must be used at individual monitoring well sites.

[2] If sample is not collected immediately after evacuation, tubing shall be withdrawn from the well prior to pump being turned off to prevent back flowing into the well.

[3] Pump will be cleaned after each use.

9.2 GENERAL GROUNDWATER SAMPLING

Groundwater sampling is necessary for a number of purposes. These include, but are not limited to, evaluating potable or industrial water sources, mapping contaminant plume movement at a land disposal or spill site, RCRA compliance monitoring (landfills), or examining a site where groundwater contamination may have or may be occurring.

Normally, groundwater is sampled from a permanent monitoring well. However, this does not exclude collection of samples from a sinkhole, pit, or other drilling or digging site where groundwater is present.

Monitoring wells are not always at the optimum. In these situations, additional wells may need to be drilled. Experienced, knowledgeable individuals (hydrologists, geologists) are needed to site the well and oversee its installation so that representative samples of groundwater can be collected.

ESC utilizes the procedures being reviewed in this section. Further guidance is available in the RCRA Groundwater Monitoring Technical Enforcement Guidance Document (TEGD); ESC field personnel will at a minimum meet, and when possible exceed, the requirements of this document.

9.3 MEASUREMENT OF WELL WATER LEVEL AND STAGNANT WATER VOLUME CALCULATION

The sampling and analysis plan provides for measurement of standing water levels in each well prior to each sampling event. Field measurements will include depth to standing water surface and total depth of the well. This data will then be utilized to calculate the volume of stagnant water in the well and provide a check on the integrity of the well (e.g., silt buildup). The measurement should be taken to 0.01 foot when possible. A battery powered level sensor will be used to measure depth to the surface of the groundwater. Equipment shall be constructed of inert materials and will be cleaned per sample equipment cleaning procedures prior to use at another well. Field data will be recorded on the Monitoring Well Data Sheet (Figure 2).

9.3.1 Procedure For Water Level Measurement

1. Clear debris from area around well (lay plastic sheathing around well pad as an option).
2. Remove protective casing lid.
3. Open monitoring well lid.
4. Lower the clean water level indicator probe down into the well. A beep will sound upon contact with the water surface. False readings can be made from the wetted side of the well so it will be necessary to check the level several times until a consistent reading is achieved. Record the distance (to the nearest 0.01 ft.) from the top of the well casing to the water surface on the Monitoring Well Data Sheet.
5. Continue to lower the probe until it reaches the well bottom. Record the distance (to the nearest 0.01 ft) from the top of the well casing to the bottom of the well on the Monitoring Well Data Sheet.
6. All water level and well depth measurements shall be made from the top of the well casing unless specified otherwise by the project manager or DER.
7. The wetted depth is obtained by subtracting total well depth from the surface level depth.

9.3.2 Calculating Water Volume

Total volume of standing water in a well is calculated by the following formula:

$$V = \pi r^2 h \times 7.48 \text{ gallons/ft}^3$$

where;

V	=	volume of standing water in the well (gallons)
r	=	radius of well (ft)
h	=	depth of water column in the well (ft)
π	=	3.14
7.48	=	conversion factor

9.4 WELL EVACUATION: WELLS WITHOUT IN-PLACE PLUMBING

Water standing in a well may not be representative of actual groundwater conditions. The standing water in a well should be removed to allow representative formation water to supplant the stagnant water. The evacuation method depends on the hydraulic characteristics of the well but the following general rules apply.

The total amount of water purged must be recorded. Therefore, the volume must be measured during the purging operation. This may be determined by:

1. Collecting the water in a graduated or known volume container (i.e., bucket);
2. Calculate the volume based on the pump rate; however pump rate may not be constant and field personnel should be aware of this;
3. Record the time that the actual purging begins in the field record.

Purging is considered complete if any one of the following criteria is satisfied:

1. Three well volumes are purged and field parameters (pH, temperature, conductivity) stabilize within 5% in consecutive readings at least 5 minutes apart. If field parameters have not stabilized after 5 well volumes, the purging is considered complete and sampling can begin.
2. Five well volumes are purged with no monitoring of field parameters.
3. At least one fully dry purge. A second dry purge may be necessary in some situations.

FIGURE 2
MONITORING WELL DATA SHEET

Site location:

ESC Project name/#:

Well Number	Depth to water surface (ft)	Depth to bottom of well (ft)	Length of water column (ft)	Volume of water evacuated (gal)	Time/date

Well Number	Temperature (^{0}F)	pH (S.U.)	Conductivity (Tmho/cm)	Time/Date

Well casing material / diameter:

Sampled by / signature:

NOTES / CALCULATIONS:

Except for low recovery wells, all wells shall be sampled within 6 hours of purging. Low recovery wells may be sampled as soon as sufficient sample matrix is available or up to 10 hours after purging. Wells that do not recover sufficiently within 10 hours should not be sampled.

Purging equipment includes Teflon® or stainless steel bailers or a peristaltic pump. Any fuel-powered pumping units shall be placed downwind of any sampling site. If purging equipment is reused, it shall be cleaned following standard procedures. Disposable latex gloves shall be worn by sampling personnel and changed prior to starting work at each sampling site.

If bailed water is determined to be hazardous, it should be disposed of in an appropriate manner.

The Florida Department of Environmental Regulation requires that during purging of the well, the purging device should be placed just below the surface of the water level and be lowered with the falling water level. For high yield wells, three casing volumes should be evacuated prior to collecting samples. Purging should be conducted at a rate to minimize agitation of the recharge water. Conductivity, pH, and temperature measurement during purging is necessary to monitor variability of the groundwater. **Samples should be collected within 6 hours of purging high yield wells.**

Low-yield wells (incapable of yielding three casing volumes) should be evacuated to dryness at a rate that does not cause turbulence. When the well recovers sufficiently, the first sample should be analyzed for pH, temperature, and conductivity. When recovery exceeds two hours, the sample should be collected as soon as sufficient volume is available. **If recovery is longer than 10 hours, the well should not be tested**. The project manager may wish to review available information to determine if obtaining a representative sample is possible.

9.4.1 Procedure for Well Evacuation: Teflon® Bailer

1. Clear the area around the well pad; cover with plastic if necessary.
2. Slowly lower the bailer to the water surface and remove it when full.
3. Reel or pull bailer to the surface using caution to not allow the lanyard (cable or string) to touch the ground.
4. Use the bailer volume and number of bails removed to determine volume of water removed. Excess hazardous material should be poured into a container for later disposal.
5. Repeat steps 2 and 3 until 1.5 well volumes have been removed.
6. Begin monitoring for pH, temperature, and conductivity. Record on Monitoring Well Data Sheet. Discard the sample into the collection pail. Purge until the change between samples of each parameter is less than 5 percent.
7. Continue until at least three well volumes have been evacuated and the parameters pH, temperature, and conductivity are within 5 percent, or until a low yield well has been evacuated to dryness.
8. Record date and time the well was purged on the Monitoring Well Data Sheet.

NOTE: For wells sampled in the State of Florida, three well volumes will be purged prior to pH, temperature, and conductivity screening. Following evacuation of three well volumes, purge water will be screened for these parameters at regular intervals until two consecutive measurements are within 5 percent. The intervals may be time-based (at least 5 min) or represent a portion of the well volume (at least 0.5 well volume)

Compliance with more stringent local, State, or Regional guidelines will be maintained where required.

9.4.2 Procedure for Well Evacuation: Peristaltic Pump

1. Clean area around the well pad.
2. Install the appropriate length of Tygon® or Teflon® tubing into the pump mechanism.
3. Insert the uncontaminated sampling end of the tubing into the well surface.
4. Connect the pump to the power supply.
5. Operate the pump at a flow rate that does not cause excessive agitation of the replacement water.
6. Determine the pump flow rate.
7. Purge until 1.5 well volumes have been evacuated.
8. Collect samples at a rate of one per well volume evacuated. Monitor these samples for pH, temperature, and conductivity. Record these measurements on the Monitoring Well Data Sheet. Monitor until the difference in each parameter is less than 5 percent.
9. Continue purging until three well volumes have been evacuated and the parameters pH, temperature, and conductivity are within 5 percent, or until a low yield well has been evacuated to dryness.
10. Record the date and time the well was purged on the Well Sampling Field Data Sheet.

9.5 PURGING TECHNIQUES: WELLS WITH IN-PLACE PLUMBING

9.5.1 General

The volume to be purged depends on whether the pumps are running continuously or intermittently and how close to the source samples can be collected. If storage/pressure tanks are present, a volume must be purged to totally exchange the volume of water in the tank.

9.5.2 Continuously Running Pumps

For continuously running pumps, the well should be purged by opening the valve and allowing it to flush for 15 minutes, if the well volume is unknown. If the sample is collected after a holding tank, the volume of the tank should also be purged.

9.5.3 Intermittently Running Pumps

Wells shall be purged at the maximum rate for at least 15 minutes. Monitoring of field parameters will continue until two consecutive measurements within 5% are measured at 5-minute intervals.

9.6 SAMPLE WITHDRAWAL

Technique for withdrawal is dependent on the parameters to be analyzed. To collect a representative sample and minimize the possibility of sample contamination:

- Use Teflon® or stainless steel sampling devices when organics are an analyte of concern.
- Use dedicated tubing or samplers for each well. If a dedicated sampler is not available, clean the sampler between sampling events. Analyze equipment blanks to ensure cross-contamination has not occurred.

The preferred sample collection order is as follows (decreasing volatility):

1. Volatile organic compounds (VOCs)
2. Extractable Organics (includes Total Recoverable Petroleum Hydrocarbons [TRPH], Oil & Grease, Pesticides and Herbicides)
3. Total metals
4. Dissolved metals
5. Microbiological
6. Inorganics (includes Nutrients, demands, and Physical Properties)
7. Radionuclides

The following items are acceptable sampling devices for all parameters:

- A gas-operated, Teflon® or stainless steel squeeze pump (also referred to as a bladder pump with adjustable flow control) should be dedicated or completely cleaned between sampling events. If it is dedicated, the protocols on use, flow rates, and flow controls should be discussed.
- A Teflon® bailer with check valves and a bottom emptying device. Dedicated or disposable bailers should not be cleaned between purging and sampling operations.

ESC generally supplies sampling devices for wells sampled by ESC. However, some clients have wells equipped with dedicated sampling devices. All dedicated equipment will be cleaned between sampling events with the exception of dedicated pump systems or dedicated pipes that are never removed. ESC will evaluate the device and the project manager shall approve/disapprove of the dedicated device prior to sampling.

If sampling includes dissolved parameters, samples shall be filtered in the field in the following manner:

1. Use a one piece, molded, in-line high capacity disposable 1.0 micron filter when collecting samples for dissolved trace metals analysis. Use a 0.45 micron filter when sampling for all other (i.e., orthophosphorous, silica, etc.) dissolved parameters.
2. Filter material should be non-contaminating synthetic fibers.
3. Filter should be placed on the positive pressure side of the peristaltic pump.
4. If well is deeper than 25 feet; a submersible bladder pump may be necessary to bring the sample to the surface. Samples shall not be collected in an intermediate container.
5. At least one filtered equipment blank using deionized water must be collected and analyzed.
6. The sample shall be preserved as required following filtration.
7. Unfiltered samples will be collected in conjunction with filtered samples.

NOTE: Filtered samples will be collected only at the request of DER and will not be collected for turbid samples only.

9.6.1 Sample Removal: With In-Place Plumbing

Samples should be collected following purging from a valve or tap as near to the well as possible, and ahead of all screens, aerators, filters, etc. Samples shall be collected directly into the sampling containers. Flow rate should not exceed 500 mL/min.

9.6.2 Sample Removal: Without In-Place Plumbing

1. Following purging, collect the sample and pour it directly from the bailer into the sample container. If a peristaltic pump is used, pump the sample directly into the container. Collect the samples in order of decreasing volatility.
2. Measure the conductivity, pH, and temperature of the samples and record the results on the Monitoring Well Data Sheet.
3. If a bailer is not dedicated, clean field equipment using standard procedures. Collect blanks at a rate of one per type of equipment cleaned. If a piece of equipment is cleaned more than twenty times, collect blanks at a rate of 10 percent. An equipment blank must be taken and preserved for each analyte method group.
4. If a bailer is used to collect samples, replace the bailer string. Take precautions not to allow the string to touch the ground. Dispose of the used string properly. If Teflon® or stainless steel cable is used, clean according to standard procedures and do not let it touch the ground.
5. Replace the well cap and close and lock the protective casing lid.

9.7 SPLIT AND DUPLICATE SAMPLE COLLECTION

Split samples measure variability between analysts, methods, and laboratories and are taken as subsamples from a single sample. Duplicate samples measure variability inherent in the collection method or waste stream and are obtained in close succession during the same sampling event.

9.7.1 Split Sample Collection

1. Collect sufficient volume in a container constructed of appropriate materials. The volume should be more than twice the volume required for one sample.
2. Preserve as necessary.
3. Mix well.
4. Alternately decant 10% of the sample volume into each container and mix well.
5. Continue until each container is filled with an adequate sample volume.
6. Seal the containers, assign a field number, and complete the chain of custody.

9.7.2 Duplicate Sample Collection

1. Collect two samples in rapid succession into separate containers.
2. Preserve as necessary.
3. Mix well.
4. Seal the containers, assign a field number, and complete the chain of custody.

9.8 DRINKING WATER SAMPLING

9.8.1 General Concerns

Containers and preservatives must be selected prior to sampling.

- Containers and preservatives shall comply with Tables 1 and 2.
- It is recommended that the appropriate preservative be added to the container by the laboratory.

9.8.2 Sampling Drinking Water Wells

1. Purging and sampling should be from a spigot closest to the wellhead.
   - The spigot should be located before the holding tank and filters. If this is not possible, the holding tank must also be purged.
   - All aerators and filters should be removed if possible.
2. Depending on the running schedule of the well and the placement of the pressure tank, the system will be purged as described in Section 9.5.
3. If volume of the pressure tank is not known, the well is purged for at least 15 minutes at maximum rate.
4. The flow is reduced to approximately 500 mL/minute.
5. Sample containers with no preservatives:
   - The interior of the cap or the container should not come in contact with anything.
   - The sample container is rinsed and the water is discarded.
   - Containers are not rinsed if collecting for oil and grease, total recoverable hydrocarbons, volatile organics (including trihalomethanes) or microbiologicals.
   - The container should be tilted to minimize agitation.
6. Sample containers with preservatives:
   - The above protocol is followed but **DO NOT** rinse the container.
   - The open end of the container should be held away from the face while filling.
   - The container should be gently tipped several times to mix the preservatives.
7. Place the bottle in a plastic bag and cool to 4°C.

9.8.3 Sampling Drinking Water Within A Facility/Residence for the Lead/Copper Rule

1. The appropriate sampling point depends on whether the sample is being taken to monitor compliance with Drinking Water Regulations for Lead and Copper. If so, the sample must be taken from a cold water tap in the kitchen or bathroom of residential housing or from an interior tap where water is used for consumption in a non-residential building.
2. Samples must be collected after the water has stood in the pipes for at least six hours.
3. THE SYSTEM SHOULD NOT BE FLUSHED.
4. The first flush should be collected immediately into the sample container. DO NOT RINSE THE CONTAINER PRIOR TO COLLECTING THE SAMPLE.
5. The container should be tilted to minimize agitation.
6. If the container contains preservative, hold the open end away from the face.
7. Add preservative as needed.
8. Replace cap and gently tip the container several times to mix the preservatives.
9. Place in a plastic sample bag.

9.8.4 Sampling a Lead Service Line in a Facility/Residence for the Lead/Copper Rule

1. When sampling for compliance, the sampling point is normally designated by the permit or the municipality.
2. For Lead & Copper samples, each sample shall have stood in the line for at least six hours and shall be collected in one of the following ways:
   a. At the tap, after flushing the volume of water between the tap and the lead service line. The volume of water shall be calculated based upon the inner diameter and length of the pipe between the tap and the service line.
   b. By tapping directly into the service line.
   c. In a single-family residence, allow the water to run until a significant temperature change indicates water standing in the service line is being sampled.
3. The flow shall be reduced to less than 500 mL/min before collecting samples.
4. Test for the presence of residual chlorine using residual chlorine indicator strips or a Hach DR-100 chlorine analyzer.
5. If residual chlorine is present and the parameter being analyzed requires removal of chlorine, collect the sample in the appropriate sample container(s) using the required preservatives.
   a. Add 0.008% $Na_2S_2O_3$ or 100mg of $Na_2S_2O_3$ per 1L of sample water directly into the sample container.
   b. After replacing the cap, tip the container several times to mix the preservative.

10.0 SOIL SAMPLING

Soil samples are preserved as per Section 14. When compositing subsamples, the quantity of each subsample used shall be measured and recorded in the field logbook.

10.1 SAMPLING EQUIPMENT

Type	Use	Materials	Allowable Parameter Groups[1]
Hand Auger (Bucket type)	Sampling	PVC	All parameter groups except VOC's, extractables and organics
Encore™ Sampler	VOC soil subsampling	Teflon®	VOC's only
Split Spoons	Sampling	Carbon Steel	All parameter groups
Trowel, Spatula	Sampling and Compositing*	Chrome-Plated Steel	All parameter groups
Spoons	Sampling and Compositing*	Stainless Steel	All parameter groups
Shovel	Sampling	Carbon Steel	All parameter groups
Mixing Pan	Compositing*	Pyrex & Aluminum	All parameter groups except metals in aluminum pan

[1] Carbon steel & Chrome-plated steel tools may be used for collecting soils where trace metal concentrations are not a concern. When these tools are used, samples should be taken from soils not in contact with the tool surface.

***** Compositing is not suitable for VOC's

10.2 HAND AUGER SAMPLING PROCEDURE

This procedure is used when only relatively shallow samples are required or when the use of heavy equipment is not practical. The hand auger may be used to collect samples of soils or other materials at various depths by adding extensions as necessary.

1. Remove surface debris from the location of the sampling hole using a clean shovel or spoon.
2. Disturbed portions of soil should be discarded and not taken as part of the sample.
3. Using a clean auger, drill to the desired sample depth. Confirm depths using a tape measure or other appropriate device.
4. Use a clean planer auger to clean and level the bottom of the boring.
5. All grab samples should be mixed thoroughly prior to placement in containers (except VOCs).

6. Using a clean auger, extract the desired sample. Subsampling is performed for VOC sample collection using an Encore™ sampling device. Once the core sample is collected, additional samples should be taken using an Encore™ sampler, either 5g or 25g, capped, sealed, and immediately cooled to 4ºC. The holding time for this method is 48 hours. Alternatively, weigh 5g of sample into a pre-weighed vial (with a Teflon® lined screw cap) containing 5mL sodium bisulfate solution and a magnetic stir bar, cap, and then ice to 4ºC. The holding time for this method is 14 days.
7. If less than the collected volume of material is desired or if multiple containers are required, subsampling shall be conducted. The collected material shall be placed in a clean mixing pan and thoroughly mixed using a clean, stainless steel spoon. The mixed material will then be quartered, removed and recombined before samples are collected. For clay soils, representative aliquots of the entire sample should be removed from the auger using stainless steel spoons. Samples for chemical analyses shall not be collected from auger flights or cuttings from hollow stem auger flights. Samples used for vapor meter determinations will not be used for trace contaminant analyses.
8. Samples should then be labeled. The depth range from which the samples were taken should be included in the sample description.
9. Repeat steps (2) through (6) as necessary to obtain samples at all desired depths.
10. When preparing composite samples, the quantity of each subsample shall be measured and recorded in the field logbook.

10.3 SPLIT AND DUPLICATE SAMPLE COLLECTION

Split samples measure variability between analysts, methods, and laboratories and are taken as subsamples from a single sample. This is unlike duplicate samples that measure variability inherent in the collection method or waste stream and are obtained in close succession during the same sampling event. True split samples are difficult to collect for soils, sediment, and sludge under field conditions. Split samples for these materials are therefore considered duplicate samples.

The collection procedure is as follows:

1. Collect the appropriate volume of sample into a clean disk constructed of a non-reactive material.
2. Mix the material with a clean utensil and separate into 4 to 10 equal portions.
3. Alternate placing a portion of the subdivided material into each container.
4. Repeat until each container is filled.
5. Assign each container a field sample number and complete the chain of custody.

11.0 WASTE SAMPLING

11.1 SAMPLING EQUIPMENT

Type	Use	Materials	Allowable Parameter Groups[1]
Shovel	Sampling	Carbon Steel	All parameter groups except metals
Split Spoons	Sampling	Carbon Steel	All parameter groups except metals
Trowel, Spatula	Sampling and Compositing*	Stainless Steel	All parameter groups
Spoon	Sampling and Compositing*	Stainless Steel	All parameter groups
Drum Pump	Sampling	Polypropylene	All parameter groups
Mixing pan	Compositing*	Pyrex or aluminum	All parameter groups except metals in aluminum pan
Coliwasa	Sampling	Glass	All parameter groups

[1]Carbon steel tools may be used for collecting wastes when trace metal concentrations are not a concern.
*Compositing is not suitable for VOC's

11.2 GENERAL

This section discusses the collection of samples from drums, tank trucks, and storage tanks, and samples from waste piles and landfills. All ESC personnel consider sampling from closed containers as a hazardous operation.

11.2.1 Specific Quality Control Procedures for Sampling Equipment

Sampling equipment used during waste sampling must be cleaned as specified in Section 12 of this manual before being returned from the field to minimize contamination.

Contaminated disposable equipment must be disposed of as specified in the sampling plan.

All field equipment shall be cleaned and repaired before being stored at the conclusion of a field study. Special decontamination procedures may be necessary in some instances and will be developed on a case-by-case basis. Any deviation from standard cleaning procedures and all field repairs shall be documented in field logbooks. Equipment that has not been properly cleaned must be tagged and labeled.

11.2.2 Collection of Supplementary Information

The collection of supplementary data is important when collecting waste samples. Any field analyses shall be recorded in field logbooks. Sketches of sampling locations and layout shall be documented in the logbooks. Photographs shall be used extensively.

11.3 OPEN AND CLOSED CONTAINER SAMPLING

11.3.1 General

When sampling containers, open containers should be sampled first since they generally present less of a hazard. Closed containers must be considered as extremely hazardous. Due to the dangers involved with container sampling, the sampling of drums or other containers containing either unknown materials or known hazardous materials shall be considered a hazardous duty assignment.

One problem with container sampling is stratification and/or phase separation. Care must be taken to ensure that the sample collected is representative. If only one layer or phase is sampled, this should be noted when interpreting analytical results.

If no stratification is present, representative samples may be composited by depth. When a drum or cylindrical container is standing vertically, depth compositing provides a good quantitative estimate of the containers contents. In other cases where containers are tipped, horizontal, deformed, etc., and stratification may not be present, vertical compositing will at least provide a qualitative sample.

11.3.2 Sampling Equipment

The following equipment is available for use in collecting waste samples: barrel bung wrenches, adjustable wrenches, etc.; coliwasa samplers for drum sampling; and peristaltic pumps for liquid waste sampling from containers.

11.3.3 Sampling Techniques

Containers containing unknown materials or known hazardous materials shall be opened using only spark proof opening devices from a grounded container.

The coliwasa sampler is a single use glass sampler, consisting of an outer glass tube with one end tapered and a separate inner glass tube with a small bulb on one end. The outer tube is slowly lowered into the drum, tapered end first. Slowly lowering the tube allows the liquid phases in the drum to remain in equilibrium. The inner glass tube is inserted into the outer tube. After both inner and outer tubes are inserted into the drum to be sampled, the inner tube bulb end is pressed gently against the tapered end of the outer tube, forming a seal. Both tubes are withdrawn from the drum and the ends of the tubes are held over the sample container.

Drum samples can also be collected using a length of glass tube (1/2-inch or less inside diameter). The tube is inserted into the drum as far as possible and the open end is sealed to hold the sample in the tube. The sample is then placed in the appropriate container. Sample volumes shall be the absolute minimum required.

Tank truck and storage tank samples may be collected from access ports on top of these tanks or trucks using the above techniques. Tank trucks are often compartmentalized, and each compartment should be sampled. Sampling from discharge valves is not recommended due to stratification possibilities and possibilities of sticking or broken valves. If the investigator must sample from a discharge valve, the valving arrangement of the particular tank truck being sampled must be clearly understood to ensure that the contents of the compartments of interest are sampled. The investigator must realize that samples obtained from valves may not be representative.

If stratification or phase separation of waste samples is suspected, the sample collected should be representative of container contents. Samples should be depth composited when possible and number and types of layers shall be noted when interpreting analytical results.

11.4 WASTE PILES AND LANDFILLS

11.4.1 General

Waste piles consist of sludge and other solid waste, liquid waste mixed with soil, slag, or any type of waste mixed with construction debris, household garbage, etc. The sampling personnel must be aware that landfills were not and are often still not selective in the types of materials accepted. Sampling at landfills could involve sampling operations that are potentially dangerous to sampling personnel.

11.4.2 Sampling Locations

Sampling locations should be selected that will yield a representative sample of the waste. Exceptions are situations in which representative samples cannot be collected safely or when the team is purposely determining worst-case scenarios.

11.4.2.1 Waste Piles

A representative sample from a small waste pile can be obtained by collecting a single sample. Collecting representative samples from large waste piles requires a statistical approach in selecting both the numbers of samples and sample location. A discussion of statistical methods is outlined in the Test Methods for Evaluating Solid Waste (SW-846) issued by the EPA Office of Solid Waste and Emergency Response.

11.4.2.2 Landfills

Representative samples from landfills are difficult to achieve to due to the heterogeneous nature of the wastes. A statistical approach should be used in

selecting both the number of samples and the sample location. Statistical methods are given in Test Methods for Evaluating Solid Waste (SW-846) issued by the EPA Office of Solid Waste and Emergency Response. Landfills often generate leachate at one or more locations downgradient of the fill material that can provide some insight into the materials contained in a landfill that are migrating via groundwater.

11.4.3 Sampling Techniques

All samples collected should be placed into a Pyrex® or aluminum mixing pan and mixed thoroughly. Samples for volatile organic compounds analyses must not be mixed or composited. Stainless steel spoons or scoops should be used to clear away surface materials before samples are collected. Near surface samples can then be collected with a clean stainless steel spoon. Depth samples can be collected by digging to the desired depth with a carbon steel shovel or scoop and removing the sample with a stainless steel spoon.

12.0 STANDARD CLEANING PROCEDURES

12.1 GENERAL

12.1.1 Introduction

ESC personnel use the procedures outlined in this section to clean field equipment prior to use. Ideally, a sufficient amount of clean equipment is carried to the field so that the project can be conducted without the need for field cleaning. This is not always the case. ESC's policy regarding cleaning field equipment is as follows:

1. Equipment used in the field must be thoroughly cleaned in a controlled environment using prescribed procedures. This minimizes the potential for contaminants being transferred to equipment, vehicles, and the laboratory.
2. All equipment will be rinsed immediately with tap water after use, even if it is to be field cleaned for other sites.
3. If equipment is used only once (i.e., not cleaned in the field), it will be labeled as "dirty" or "contaminated equipment" in the field and transported separately from clean equipment.
4. All cleaning procedures shall be documented. Field decontamination shall be documented in the field records. These records will specify the type of equipment cleaned and the specific protocols that are used. In-house cleaning records must identify the type of equipment, date it was cleaned, SOP used, and person that cleaned it.
5. Unless justified through documentation (i.e., company written protocols and analytical records) and historic data (i.e., absence of analytes of interest in equipment blanks), the protocols in Sections 12.1.2 through 12.7.11 shall be followed without modification.
6. All field sampling equipment shall be pre-cleaned in-house.

12.1.2 Cleaning Materials

Use a phosphate-free, laboratory detergent such as Liquinox®. The use of any other detergent is noted in field logbooks and summary reports.

Ten percent nitric acid solution shall be made from reagent-grade nitric acid and deionized water.

The standard cleaning solvent used will be pesticide-grade isopropanol. Other solvents (acetone and/or hexane) may be substituted as necessary. The use of other solvents must be documented in field logbooks and summary reports.

Tap water may be used from any potable water system. Untreated water is not an acceptable substitute for tap water.

Deionized water is tap water that has been passed through a deionizing resin column and should contain no inorganic compounds at or above analytical detection limits. Organic-free water is tap water that has been de-ionized and treated with activated carbon. Organic-free water should contain no detectable levels of organic compounds, and less than 5 ug/L of VOCs.

Analyte-free water is water in which all the analytes of interest and all interferences are below the method detection limits. Analyte-free water is always used for blank preparation and for the final in-house decontamination rinse.

Substitution of a higher grade water (i.e., deionized or organic-free water for tap water) is permitted and need not be recorded. Solvent, nitric acid, detergent, and rinse water used to clean equipment shall not be reused.

12.1.3 Marking Clean Equipment

Equipment that is cleaned by these methods shall be marked with the date and time that the equipment was cleaned.

12.1.4 Marking Contaminated or Damaged Field Equipment

Field equipment that needs repair will be tagged and repairs or symptoms noted on the tag. Field equipment that needs cleaning **will not** be stored with clean equipment. All wrapped equipment not used in the field may be placed back in stock after equipment is inspected to ensure that contamination has not taken place.

12.1.5 Decontamination of Equipment Used With Toxic or Hazardous Waste

Equipment used to collect hazardous or toxic wastes or materials from hazardous waste sites, RCRA facilities, or in-process waste streams shall be decontaminated prior to leaving the site. This decontamination procedure shall consist of washing with laboratory detergent and rinsing with tap water. More stringent procedures may be required depending on the waste sampled.

If equipment is heavily contaminated, an acetone or acetone/hexane/acetone pre-rinse may be necessary prior to regular decontamination procedures. It is not recommended that this type of cleaning be performed in the field.

12.1.6 Disposal of Cleaning Materials

See Section 16.

12.1.7 Safety Procedures For Cleaning Operations

All applicable safety procedures shall be followed during cleaning operations. The following precautions shall be taken during cleaning operations:

- Safety glasses or goggles, gloves, and protective clothing will be worn during all cleaning operations.
- Solvent rinsing operations will be conducted under a hood or in an open, well ventilated area.
- No eating, smoking, drinking, chewing, or hand to mouth contact shall be permitted during cleaning operations.

12.1.8 Storage of Field Equipment

All clean field equipment shall be stored in a designated, contaminant-free area.

12.2 QUALITY CONTROL PROCEDURES FOR CLEANING

12.2.1 General

This section establishes quality control methods to monitor the effectiveness of the equipment cleaning procedures. The results of these methods will be monitored by the ESC Quality Assurance Department. All quality control procedures are recorded in a logbook and maintained in a quality assurance file. If contamination problems are detected, the ESC QA Department shall determine the cause(s) of the problem(s) and take immediate corrective action.

12.2.2 Rinse Water

The quality of water used shall be monitored once per quarter by placing water in standard, precleaned sample containers and submitting them to the ESC laboratory for analysis. Organic-free water will also be submitted for analyses of the various organic compounds.

12.3 PROCEDURES FOR CLEANING TEFLON® OR GLASS EQUIPMENT USED IN THE COLLECTION OF SAMPLES FOR TRACE ORGANIC COMPOUNDS AND/OR METALS ANALYSES

1. Equipment will be washed with laboratory detergent and hot water using a brush to remove any particulate matter or surface film. If oil, grease, or other hard to remove residues are present on the equipment, an acetone/hexane/acetone pre-wash and/or steam cleaning may be necessary.
2. Rinse the equipment with hot tap water.
3. Rinse or soak, if necessary, equipment with a 10% nitric acid solution. If nitrogen-containing compounds are analytes of concern, hydrochloric acid must be used as a substitute or subsequent equipment rinse.
4. Rinse equipment with tap water.
5. Rinse equipment with deionized water.
6. Rinse equipment twice with solvent and allow to dry.
7. If equipment cannot be cleaned effectively, discard properly.
8. Wrap equipment in aluminum foil. Seal in plastic and date.

12.4 PROCEDURES FOR CLEANING STAINLESS STEEL OR METAL SAMPLING EQUIPMENT USED IN TRACE ORGANIC AND/OR METALS SAMPLE COLLECTION

1. Equipment will be washed with laboratory detergent and hot water using a brush to remove any particulate matter or surface film. If oil, grease, or other hard to remove materials are present, a acetone/hexane/acetone pre-wash and/or steam cleaning may be necessary.
2. Rinse equipment with hot tap water.
3. Rinse equipment with deionized water.
4. Rinse equipment twice with solvent and allow to dry.
5. If equipment cannot be cleaned effectively, discard properly.
6. Wrap equipment in aluminum foil. Seal in plastic and date.

12.5 CLEANING PROCEDURES FOR AUTOMATIC SAMPLING EQUIPMENT

12.5.1 General

All automatic wastewater samplers will be cleaned as follows:

- The exterior and accessible interior portions of automatic samplers will be washed with Liquinox and rinsed with tap water.
- The electronics casing will be cleaned with a clean damp cloth.
- All vinyl sample tubing will be discarded after each use.
- Teflon® tubing will be cleaned using procedures found in Section 12.6.2.
- Silastic pump tubing will be cleaned and re-used after each use, if possible. Tubing will be cleaned using cleaning procedures specified in Section 12.6.1 of this document. Tubing shall be checked on a regular basis and will be changed if it has become discolored or loses elasticity.

12.5.2 Reusable Glass Composite Sample Containers

1. If containers are used to collect samples that contain hard to remove materials (i.e., oil and grease) it is rinsed as necessary with reagent grade acetone prior to the detergent wash. If material cannot be removed, the container is discarded.
2. Wash containers thoroughly with hot tap water and Liquinox and rinse thoroughly with hot tap water.
3. If metals are to be sampled, rinse with 10% nitric acid. If nutrients are to be sampled, follow with a 10% hydrochloric acid rinse.
4. Rinse thoroughly with tap water.
5. Rinse thoroughly with DI water.
6. If organics are to be sampled, rinse twice with isopropanol and allow to air dry for 24 hours or more. Cap the container with the decontaminated Teflon® lined lid.
7. After use rinse with tap water in the field and cover to prevent drying of material onto the interior surface.
8. Containers that have a visible scale, film, or discoloration after cleaning or were used at a chemical manufacturing facility should be properly discarded at the conclusion of the sampling activities.

12.5.3 Reusable Plastic Composite Sample Containers

1. Wash containers with hot tap water and laboratory detergent using a bottlebrush to remove particulate matter and surface film.
2. Rinse containers with hot tap water.
3. Rinse containers with 10% nitric acid. If nitrogen containing compounds are analytes of concern, hydrochloric acid must be used as a substitute or subsequent equipment rinse.
4. Rinse containers with tap water.
5. Rinse containers with deionized water.
6. Cap with aluminum foil.
7. Plastic sample containers used at facilities that produce toxic compounds will be properly disposed of at the conclusion of the sampling activities. Containers that have a visible film, scale, or other discoloration remaining after cleaning will be discarded.

12.5.4 Plastic Sequential Sample Bottles for Automatic Sampler Base

1. Rinse bottles in field with potable or de-ionized water when possible.
2. Wash in dishwasher at wash cycle, using laboratory detergent cycle, followed by tap and deionized water rinse cycles. Alternatively, hand wash using the same procedure.
3. Rinse with 10% nitric acid. If nitrogen containing compounds are analytes of concern, hydrochloric acid must be used as a substitute or subsequent equipment rinse.
4. Rinse with tap water.
5. Replace bottles in sampler base; cover with aluminum foil before storing.

12.6 CLEANING PROCEDURES FOR SAMPLING TUBING

12.6.1 Silastic Rubber Pump Tubing Used In Automatic Samplers

Silastic pump tubing used in automatic samplers need not be replaced in pumps where the sample does not contact the tubing, where the sampler is being used solely for purging purposes (i.e., not being used to collect samples). Tubing must be changed on a regular basis, if used for sampling purposes, and should be cleaned in this manner:

1. Flush tubing with laboratory grade detergent and hot tap water
2. Rinse thoroughly with hot tap water
3. Rinse thoroughly with DI water
4. If used to collect metals samples, the tubing shall be flushed with 1+5 nitric acid, followed by a thorough rinsing with DI water
5. Install the tubing in the automatic wastewater sampler
6. Cap both ends with aluminum foil or equivalent

Tubing should always be replaced at automatic sampler manufacturer's recommended frequencies. If tubing cannot be adequately cleaned, it shall be discarded.

12.6.2 Teflon® Tubing

New Teflon® tubing shall be pre-cleaned as follows:

1. Rinse outside of the tubing with pesticide-grade solvent.
2. Flush interior of the tubing with pesticide-grade solvent.
3. Let dry overnight in drying oven or equivalent.
4. Wrap tubing in aluminum foil and seal in plastic.

Reused tubing shall be transported to the field in pre-cut and pre-cleaned sections. Field cleaning of Teflon® is not recommended. The following steps describe in-house cleaning procedures:

1. Exterior of tubing must be cleaned first by soaking in hot, soapy water in a stainless steel or non-contaminating sink. Particulate may be removed with a brush.
2. Clean inside of tubing ends with a small bottlebrush.
3. Rinse surfaces and ends with tap water.
4. Rinse surfaces and ends with nitric acid, tap water, isopropanol, and analyte-free water.
5. Place on fresh aluminum foil, connect all sections with Teflon® couplings.

6. Cleaning configuration:
   a. Cleaning solutions are placed in a clean, 2-liter glass jar.
   b. Place one end of tubing in the solution, the other in the **INFLUENT** end of a peristaltic pump.
   c. Effluent from the pump can be recycled through the glass cleaning solution jar. All cleaning solutions can be recycled EXCEPT the final isopropanol and analyte-free water rinses.
7. The above configuration is used as follows:
   a. Pump generous amounts of hot, soapy water through the tubing.
   b. Follow this with tap water, 10% nitric acid, tap water, isopropanol, and analyte-free water.
   c. The nitric acid and isopropanol rinses should be allowed to remain in the tubing for 15 minutes with the pump shut off then continue with subsequent rinses
   d. Leave any couplings in and connect or cover the remaining ends.
8. After cleaning the interior, rinse the exterior with analyte-free water.
9. The cleaned lengths are wrapped in aluminum foil and stored in a clean, dry area until use.

12.7 FIELD EQUIPMENT CLEANING PROCEDURES

12.7.1 General

It is the responsibility of field personnel to properly clean equipment in the field. The following procedures shall be observed when cleaning equipment in the field.

12.7.2 Conventional Equipment Use

Remove deposits with a brush if necessary. If only inorganic anions are of interest, equipment should be rinsed with analyte-free water and with the sample at the next sampling location prior to collection. Clean equipment for the collection of samples for organic compounds or trace inorganic analyses according to Section 12.7.3.

12.7.3 Equipment Used to Collect Organic Compounds and Trace Metals Samples

1. Clean with tap water and laboratory detergent. If necessary, use a brush to remove particulate and surface films then rinse with tap water.
2. Rinse with 10 to 15% nitric acid solution followed by 10% hydrochloric acid rinse (unless equipment is made of metal) followed by tap water and DI water.
3. Rinse twice with solvent.
4. Rinse with organic-free water and allow to air dry.
5. If organic-free water is unavailable, let air dry. Do not rinse with deionized or distilled water.

6. Wrap with aluminum foil or plastic.

12.7.4 Teflon®, Glass, Stainless Steel or Metal Equipment Used to Collect Samples for Metal Analyses

1. Remove particulate matter and surface films. Clean with laboratory detergent and tap water.
2. Rinse with tap water.
3. Ten percent nitric acid solution (skip 3 and 4 if equipment is made of metal and/or stainless steel).
4. Rinse with tap water.
5. Rinse with deionized water then let air dry.

12.7.5 Instruments Used to Measure Groundwater Levels

1. Wash with laboratory detergent and tap water.
2. Rinse with tap water.
3. Rinse with deionized water.
4. Allow to dry.

12.7.6 Field Filtration Apparatus

1. A new, disposable filtration unit will be used for each site. Filter pore size will be dependent on parameter being monitored as per Section 9.6.
2. The peristaltic pump is cleaned as described in Section 12.7.7.
3. Silastic pump tubing will be cleaned as described in Section 12.6.1.
4. If Teflon® tubing is used, it will be cleaned as described in Section 12.6.2.
5. Other tubing types must be cleaned following the appropriate regimen described in Section 12.6. In general, non-Teflon® type tubing (e.g., HDPE) will not be re-used.

12.7.7 Flow Meters, Above Ground Pumps, Bladder Pumps and Other Field Instrumentation

The exterior of equipment such as flow meters should be washed with a mild detergent and rinsed with tap water before storage. The interior of such equipment may be wiped with a damp cloth.

Other field instrumentation should be wiped with a clean, damp cloth. Meter probes should be rinsed with deionized water before storage.

Equipment desiccant should be checked and replaced as necessary.

Peristaltic pumps used for purging must be free of oil and grease on the exterior. They must be cleaned on the outside with Liquinox and rinsed with tap water followed by DI water.

12.7.8 In-Field Decontamination For Submersible Purging Pump and Tubing

ESC uses the submersible bladder pump listed in Section 9.1 only for purging and not for sample collection. The pump and tubing shall be decontaminated between wells in the following manner:

1. Interior of the pump and tubing shall be thoroughly flushed with a soapy water solution.
2. Wipe or scrub the exterior of the pump and tubing as necessary with the appropriate soap solution.
3. Rinse exterior and interior of pump and tubing thoroughly with tap water followed by a deionized water rinse.
4. Allow remaining water to drain from tubing and pump and allow to air dry as long as possible in a contaminant free area before purging the next well.

12.7.9 Shipping Containers

All reusable shipping containers shall be washed with laboratory detergent, rinsed with tap water, and air dried before storage or re-use. Extremely contaminated shipping containers shall be cleaned as thoroughly as possible and properly disposed.

12.7.10 Analyte Free Water Containers

Analyte-free water containers can be made of glass, Teflon®, polypropylene, or high density polyethylene (HDPE). Inert glass or Teflon® are recommended for holding organic-free sources of water. Polypropylene can be used when organics are not analytes of concern. HDPE is not normally recommended but is acceptable for use. Water should not be stored in these containers for extended periods. Containers of water should only be used for a single event and should be disposed of at the end of the sampling day. The procedure for cleaning analyte-free water containers is as follows:

1. For new containers, follow instructions in Section 12.3 of this manual. Delete the solvent rinse if containers are made of plastic.
2. Cap with Teflon® film, aluminum foil, or the Teflon® lined bottle cap (aluminum foil or Teflon® film may also be used as a cap liner).

If water is being stored in reused containers, the following cleaning procedures should be followed:

1. After emptying, cap the container.
2. Wash exterior of the container with Liquinox and rinse with DI water.
3. Rinse the interior twice with isopropanol unless the container is made of plastic.
4. Rinse the interior thoroughly with analyte-free water.
5. Invert and allow to dry.
6. Fill the container with analyte-free water and cap with aluminum foil, Teflon® film, or a Teflon® lined bottle cap.
7. Water shall not be stored prior to a sampling event for more than 3 days.

12.7.11 Vehicles

Field vehicles used by ESC personnel should be washed at the conclusion of each sampling event. This should reduce the risk of contamination due to transport on a vehicle. When vehicles are used at hazardous waste sites or on studies where pesticides, herbicides, organic compounds, or other toxic materials are known or suspected to be present, a thorough interior and exterior cleaning is mandatory at the conclusion of the site visit.

Vehicles are equipped with trash containers. ESC personnel are responsible for cleanliness of each vehicle.

13.0 SAMPLE HISTORY

Sample chronology is recorded and kept on the ESC chain of custody, field logbooks and laboratory notebooks. These are discussed in detail in Section 9.0.

14.0 SAMPLE CONTAINERS, PRESERVATION METHODS AND HOLDING TIMES

14.1 GENERAL CONSIDERATIONS

The following section contains information regarding sample containers, preservation methods, and holding times. Refer to SW-846, Table II-1 and Chapter 3, Page 3 for solid waste and RCRA projects and 40 CFR Part 136, Table II for water and wastewater projects.

The provisions of 40 CFR Part 136, Table II shall take precedence over requirements given in any approved method when sampling in the State of Florida for water and wastewater.

Proper sample preservation is the responsibility of the sampling team and it is their responsibility to assure that all samples are preserved according to 40 CFR Part 136. For the purposes of this manual, "immediately" will be defined as within 15 minutes.

Sample preservation is accomplished either by obtaining prepreserved containers from an acceptable source or by adding preservatives in the field.

It is the responsibility of the field team accepting prepreserved containers to make sure that the proper preservatives are used and desired results are achieved. The laboratory shall also supply additional preservatives from the same source in suitable containers.

14.2 SAMPLE PRESERVATION

The following protocols apply for sample containers preserved in the field after the sample has been added:

1. Preservatives shall be at least reagent grade or higher. The acid for metals shall be suitable for trace metals analyses.
2. Fresh preservatives shall be obtained prior to each sampling event. Remaining preservatives that are not sealed must be discarded in an acceptable manner.
3. Preservatives are transported in pre-measured glass ampules and added directly to the sample.
4. A corresponding amount of preservative shall be added to associated equipment blanks.
5. The pH is checked on all pH preserved samples with the exception of VOC, oil and grease, and TRPH.

Effectiveness of pH adjustment is made in the following manner:

1. Narrow range pH paper is used to test a small aliquot of the preserved sample.
2. A small portion of sample is placed into a container, checked with pH paper, and compared against the color chart.
3. Discard the aliquot properly, but do not pour back into the sample container.
4. If pH is acceptable, document in field log and prepare for transport to laboratory.

If pH is unacceptable, continue to add additional preservative in measured increments using the methods described above until an acceptable pH has been reached. Record the total amount of preservative used in the field log. Always use additional preservative from the same source as the initial preservation attempt.

In some cases, an extra dummy sample can be used to test pH preservation. Content should be suitably discarded.

If equipment blanks or field blanks are used, the maximum amount of preservative that was used to preserve any single sample in the set shall be added to the equipment or field blank.

Samples requiring temperature preservation shall be cooled to 4°C. The cooler will be checked to ensure that the ice has not melted.

14.3 SAMPLE CONTAINERS

ESC does not clean and re-use sample containers. ESC purchases all sample collection containers precleaned. All used sampling containers are discarded after use. The cleaning criteria of all containers must meet EPA analyte specific requirements.

QEC provides written certification that containers do not contain analytes of concern above method detection levels

ESC maintains records for these containers (lot numbers, certification statements, date of receipt, etc.) and intended uses are documented.

14.4 FIELD REAGENT HANDLING

Reagents, cleaning materials, and preservatives that are maintained by a field team will be stored, transported, and handled in such a way as to prevent and/or minimize contamination. The following storage and use protocols will be observed:

1. Chemicals will be stored in-house and transported to the field segregated by reactivity.
2. Acids are stored in an acid storage cabinet and solvents are stored in a vented, explosion proof solvent storage cabinet.
3. All chemicals transported to the field are stored in bottles and packed to avoid breaks.
4. When reagents are transferred from an original container, the transport container must be pre-cleaned and of compatible material as the original container.
5. Chemicals shall be separated from sample containers and samples to avoid reaction and possible contamination.
6. Analyte free water shall be segregated from solvents to prevent contamination.

14.4.1 Reagent and Standard Storage

Chemical	Method of Storage
Nitric acid	Stored separated from other acids in original container in vented cabinet.
Sulfuric acid	See above
Hydrochloric acid	See above
Isopropanol	Stored in original glass container in vented and explosion proof solvent storage cabinet.
pH calibration buffers, turbidity standards, conductivity standards	Stored in cabinet designated for standard and reagent storage. Stored in temperature-controlled area of laboratory.
Sodium hydroxide	Stored in original container in designated cabinet in laboratory.
Sodium thiosulfate, zinc acetate, ascorbic acid, lead acetate	Stored in original containers in designated area of laboratory. Reagent solutions made fresh prior to use.

14.5 SAMPLE TRANSPORT

In the majority of situations, samples will be delivered directly to the laboratory by the field sampling team or field courier following standard chain of custody protocols. Samples will be preserved immediately (i.e., within 15 minutes) and packed with ice prior to transport. The field team will relinquish custody to the login sample custodian upon arrival at the laboratory.

Certain situations require that the field sampling team ship samples to the laboratory utilizing common carrier (UPS, FEDEX, etc.). If samples are sent by common carrier, all documentation (transmittal form, chain of custody, field data, analyses request, etc.) shall be placed in a ziplock bag and placed inside the sample container. The container is then sealed closed and sent to the laboratory in the required time frame to meet requirements of time-sensitive analyses.

14.6 BIOMONITORING SAMPLING

Preservation and Sample Volume
Aqueous samples collected for Bioassay can be collected in either glass or HDPE plastic. There is no required chemical preservation for this type of sample but the sample must be kept at 4 ± 2°C. The required volume varies independently with each type of analysis but the minimum collected is 250mL. The samples can be held for a maximum of 36 hours from the time of collection until first use.

Sample Collection
Grab sample protocols are utilized for acute bioassay unless otherwise specified in permit requirements. Composite sampling protocols are utilized for chronic bioassays unless otherwise specified in permit requirements. (Actual sampling protocols are discussed in detail throughout this appendix) ESC field collection personnel are required to collect all bioassay samples by completely filling the sample bottle and leaving no headspace. It is important that bottles be filled completely to reduce possible aeration that may reduce the toxic properties of the sample. If a client chooses to collect the samples, a trained ESC field collection person will explain in detail the importance of reducing aeration by filling the sample bottle completely.

14.6.1 Biomonitoring Sampling Containers

All bioassay glassware are cleaned using the following EPA protocol:

- soak for 15 minutes in hot tap water with detergent and scrub
- rinse thoroughly with hot tap water
- rinse thoroughly with dilute nitric acid (10%)
- rinse thoroughly with deionized water
- rinse thoroughly with pesticide grade acetone

- rinse well with deionized water then rinse with dilution water

New glassware will be cleaned according to the same procedure as listed above except the first step will be preceded by soaking the glassware overnight in 10% HNO_3. Sample collection containers used for automatic sampling devices are cleaned according to the same protocol listed above.

ESC does not reuse sample transport containers. All bottles used for sample transport are new.

TABLE 14.6A: PRESERVATION, HOLDING TIME AND SAMPLE CONTAINERS (SOLID WASTE AND SOIL SAMPLES)

PARAMETER	PRESERVATIVE	HOLDING TIME	CONTAINER(S)
Metals	Cool, 4°C	* 6 Months	Plastic, glass
Volatile Organic Compounds in Water, Includes TPH GRO/BTEX	Cool, 4°C	14 Days	Glass, Teflon®-lined septum
Volatile Organic Compounds in Soil/Solid Includes TPH GRO/BTEX	Cool, 4°C (If using vials, then Sodium Bisulfate is used)	48 hours (using Encore™ sampler) 14 Days (using pre-weighed, preserved, vials)	Encore™ Sampler or Pre-weighed glass vials (Teflon®-lined septum) with magnetic stir bar
Semi-volatiles, non-volatile organics Includes TPH DRO	Cool, 4°C	14 Days until extraction, 40 days after extraction	Glass, Teflon®-lined cap
Solids	Cool, 4°C	7 Days	Plastic, glass
Cyanides	Cool, 4°C	14 Days	Glass
Oil and Grease	Cool, 4°C	28 Days	Glass, Teflon®-lined cap

* Maximum holding time for mercury is 28 days.

TABLE 14.6B: WASTEWATER PRESERVATION, HOLDING TIME AND SAMPLE CONTAINERS (OTHER PARAMETERS)

Parameter	Container[1]	Preservation[2,3]	Maximum Holding Time[4]	Required Sample Volume
Biomonitoring				
Biomonitoring Acute and Chronic	P, G	Cool, 4°C	36 hours to first use	Determined by analysis req. Min. 250 mL.
		Bacteriological		
Coliform, Fecal and Total Fecal Streptococci	P, G	Cool, 4°C, 0.008% $Na_2S_2O_3$[5]	6 hours	150 mL
Inorganics				
Acidity	P, G	Cool, 4°C	14 days	250 mL
Alkalinity	P, G	Cool, 4°C	14 days	250 mL
Ammonia	P, G	Cool, 4°C, H_2SO_4 to pH <2	28 days	500 mL Distilled-1000 mL
Biochemical Oxygen Demand	P, G	Cool, 4°C	48 hours	2000 mL
Bromide	P, G	None Required	28 days	200 mL

Parameter	Container[1]	Preservation[2,3]	Maximum Holding Time[4]	Required Sample Volume
Biochemical Oxygen Demand, Carbonaceous	P, G	Cool, 4°C	48 hours	2000 mL
Chemical Oxygen Demand	P, G	Cool, 4°C, H_2SO_4 to pH <2	28 days	100 mL
Chloride	P, G	None Required	28 days	200 mL
Chlorine, Total Residual	P, G	None Required	Immediately	200 mL
Color	P, G	Cool, 4°C	48 hours	250 mL
Cyanide, Total and Amenable	P, G	Cool, 4°C, NaOH to pH >12, 0.6 g/l ascorbic acid[5]	14 days[6]	2000 mL
Fluoride	P	None Required	28 days	100 mL
Hardness	P, G	HNO_3 to pH <2, H_2SO_4 to pH <2	6 months	100 mL
Hydrogen Ion (pH)	P, G	None Required	Immediately	100 mL
Kjeldahl and Organic Nitrogen	P, G	Cool, 4°C, H_2SO_4 to pH <2	28 days	500 mL
Chromium VI	P, G	Cool, 4°C	24 hours	500 mL
Mercury[7]	P, G	HNO_3 to pH <2	28 days	500 mL
Metals[7], except Chromium VI and Mercury	P, G	HNO_3 to pH <2	6 months	1000 mL
Nitrate	P, G	Cool, 4°C	48 hours	500 mL
Nitrate-Nitrite	P, G	Cool, 4°C, H_2SO_4 to pH <2	28 days	500 mL
Nitrite	P, G	Cool, 4°C	48 hours	200 mL
Oil and Grease	G	Cool, 4°C, HCl/H_2SO_4 to pH <2	28 days	1000 mL
Organic Carbon	P, G	Cool, 4°C, HCl/H_2SO_4 to pH <2	28 days	100 mL
Orthophosphate	P, G	Filter Immediately, Cool, 4°C	48 hours	200 mL
Oxygen, Dissolved Probe	G Bottle and Top	None Required	Immediately	Not Applicable
Phenols	G only	Cool, 4°C, H_2SO_4 to pH <2	28 days	1000 mL
Phosphorus (elemental)	G	Cool, 4°C	48 hours	2000 mL
Phosphorus, Total	P, G	Cool, 4° C, H_2SO_4 to pH <2	28 days	500 mL
Residue, Total	P, G	Cool, 4°C	7 days	500 mL
Residue, Filterable	P, G	Cool, 4°C	7 days	500 mL
Residue, Nonfilterable (TSS)	P, G	Cool, 4°C	7 days	500 mL
Residue, Settleable	P, G	Cool, 4°C	48 hours	1000 mL
Residue, Volatile	P, G	Cool, 4°C	7 days	500 mL
Specific Conductance	P, G	Cool, 4°C	28 days	500 mL
Sulfate	P, G	Cool, 4°C	28 days	500 mL
Sulfide	P, G	Cool, 4°C, add zinc acetate plus NaOH to pH >9	7 days	300 mL
Sulfite	P, G	None Required	Immediately	250 mL
Surfactants	P, G	Cool, 4°C	48 hours	500 mL
Temperature	P, G	None Required	Immediately	Not Applicable
Turbidity	P, G	Cool, 4°C	48 hours	200 mL
Organics[8]				
Volatile Halocarbons	G, Teflon®-lined septum	Cool, 4°C, 0.008% $Na_2S_2O_3$[5]	14 days	2 x 40 mL
Volatile Aromatic Hydrocarbons	G, Teflon®-lined septum	Cool, 4°C, 0.008% $Na_2S_2O_3$[5], HCl to pH 2[9]	14 days	2 x 40 mL

Parameter	Container[1]	Preservation[2,3]	Maximum Holding Time[4]	Required Sample Volume
Acrolein and Acrylonitrile	G, Teflon®-lined septum	Cool, 4°C, 0.008% $Na_2S_2O_3$[5]. Adjust pH to 4-5[10]	14 days	2 x 40 mL
Phenols[11]	G, Teflon®-lined cap	Cool, 4°C, 0.008% $Na_2S_2O_3$[5]	7 days to ext. then 40 days	3000 mL
Benzidines[11]	G, Teflon®-lined cap	Cool, 4°C, 0.008% $Na_2S_2O_3$[5]	7 days to ext.[13]	3000 mL
Phthalate esters[11]	G, Teflon®-lined cap	Cool, 4°C	7 days to ext. then 40 days	3000 mL
Nitrosamines[11, 14]	G, Teflon®-lined cap	Cool, 4°C, store in dark, 0.008% $Na_2S_2O_3$[5].	7 days to ext. then 40 days	3000 mL
PCBs[11], Acrylonitrile	G, Teflon®-lined cap	Cool, 4°C	7 days to ext. then 40 days	3000 mL
Nitroaromatics and Isophorone[11]	G, Teflon®-lined cap	Cool, 4°C, 0.008% $Na_2S_2O_3$[5], store in dark	7 days to ext. then 40 days	3000 mL
Polynuclear Aromatic Hydrocarbons[11]	G, Teflon®-lined cap	Cool, 4°C, 0.008% $Na_2S_2O_3$[5], store in dark	7 days to ext. then 40 days	3000 mL
Haloethers[11]	G, Teflon®-lined cap	Cool, 4°C, 0.008% $Na_2S_2O_3$[5]	7 days to ext. then 40 days	3000 mL
Chlorinated Hydrocarbons[11]	G, Teflon®-lined cap	Cool, 4°C	7 days to ext. then 40 days	3000 mL
TCDD[11]	G, Teflon®-lined cap	Cool, 4°C, 0.008% $Na_2S_2O_3$[5]	7 days to ext. then 40 days	3000 mL
Pesticides[11]	G, Teflon®-lined cap	Cool, 4°C, pH 5-9[15]	7 days to ext. then 40 days	2000 mL
Radiological Analyses: Alpha, beta and Radium	P, G	HNO_3 to pH <2	6 months	3000 mL

NOTES:

[1] Polyethylene (P) or Glass (G).

[2] Sample preservation should be performed immediately upon sample collection. If using an automatic sampler, preserve by maintaining at 4 deg. C until compositing and sample splitting is completed.

[3] Samples shipped by common carrier or sent through the United States Mail must comply with the Department of Transportation Hazardous Materials Regulations (49 CFR Part 172). For the preservation requirements of Table II, the Office of Hazardous Materials, Materials Transportation Bureau, Department of Transportation has determined that the Hazardous Materials Regulations do not apply to the following materials: Hydrochloric acid (HCl) in water solutions at concentrations of 0.04% by weight or less (pH about 1.96 or greater); Nitric acid (HNO_3) in water solutions at concentrations of 0.15% by weight or less (pH about 1.62 or greater); Sulfuric acid (H_2SO_4) in water solutions at concentrations of 0.35% by weight or less (pH about 1.15 or greater); and Sodium hydroxide (NaOH) in water solutions at concentrations of 0.080% by weight or less (pH about 12.30 or less).

[4] Samples should be analyzed as soon as possible after collection. The times listed are the maximum times that samples may be held before analysis and still be considered valid for analytical and regulatory purposes.

[5] Only to be used in the presence of residual chlorine.

[6] Maximum holding time is 24 hours when sulfide is present. Optionally all samples may be tested with lead acetate paper before pH adjustment in order to determine if sulfide is present. If sulfide is present, it can be removed by the addition of cadmium nitrate powder until a negative spot test is obtained. The sample is filtered and pH adjusted to 12.

[7] Dissolved metals samples should be filtered immediately on-site before adding preservative.

[8] Applies to samples to be analyzed by GC, LC, or GC/MS for specific compounds.

[9] Sample receiving no pH adjustment must be analyzed within seven days after collection.

[10] pH adjustment is not required if acrolein will not be measured.

[11] When the extractable analytes of concern fall within a single chemical category, the specified preservative and maximum holding times should be observed for sample integrity. When the analytes of concern fall within two or

more chemical categories, the sample may be preserved by cooling to 4° C, reducing residual chlorine, storing in the dark, and adjusting the pH to 6-9; samples preserved in this manner may be held for seven days before extraction and for forty days after extraction.

[12] 1,2-diphenylhydrazine is likely to be present, adjust the pH of the sample to 4.0 plus or minus 0.2.

[13] Extracts may be stored up to seven days before analysis if stored in an oxidant-free atmosphere.

[14] For the analysis of diphenylnitrosamine, add 0.008% $Na_2S_2O_3$ and adjust pH to 7-10 with NaOH within 24 hours of sampling.

[15] The pH adjustment may be performed upon receipt at the laboratory and may be omitted if the samples are extracted within 72 hours of collection. For the analysis of aldrin, add 0.008% $Na_2S_2O_3$.

14.7 SAMPLE CONTAINER PACKING PROCEDURES

ESC routinely sends sample containers to clients. Standard operating procedure determines the containers needed for the requested analyses. A sample request form is completed to document what is needed, the destination, the date prepared and the initials of the preparer. Containers are prepared, with appropriate preservatives, labels, and custody seals, and organized for the client's convenience in a cooler. The cooler also contains a temperature blank, chain of custody, a return address label, and applicable instructions. The cooler is bound with packaging tape (and a custody seal if requested) and shipped UPS.

15.0 SAMPLE DISPATCH

Samples collected during field investigations or in response to a hazardous materials incident are classified by the project manager, prior to shipping, as either environmental or hazardous material samples. The shipment of samples, designated as environmental samples, is not regulated by the U.S. Department of Transportation.

Samples collected from certain process streams, drums, bulk storage tanks, soil, sediment, or water samples from suspected areas of high contamination may need to be shipped as hazardous. These regulations are promulgated by the US-DOT and described in the Code of Federal Regulations (49 CFR 171 through 177). The guidance for complying with US-DOT regulations in shipping environmental laboratory samples is given in the "National Guidance Package for Compliance with Department of Transportation Regulations in the Shipment of Environmental Laboratory Samples."

15.1 SHIPMENT OF ENVIRONMENTAL SAMPLES

Shipping receipts are maintained at the ESC laboratory. The shipment of preserved sample containers or bottles of preservatives (i.e., NaOH pellets, HCl, etc.) which are designated as hazardous under the US-DOT, Hazardous Materials Table, 49 CFR 171.101, must be transported pursuant to the appropriate US-DOT regulations.

Samples packaged for shipment by ESC shall be segregated by sample type, preservation requirements, and potential contaminant level. During events in which large numbers of samples will be collected, samples are segregated by analyses required. If multiple sites are sampled, or if specific and separate areas of interest are identified, samples will be further segregated for packaging prior to shipment.

Environmental samples shall be packed prior to shipment using the following procedures:

1. Select a cooler (clean and strong). Line the cooler with a large heavy-duty plastic bag.
2 Allow sufficient headspace (except VOC's or others with zero headspace requirements) to compensate for any pressure and temperature changes.
3. Be sure the lids on all bottles are tight.
4. Place all bottles in appropriately sized polyethylene bags.
5. Place VOC vials in foam material transport sleeves.
6. Place foam padding in the bottom of the cooler and then place the bottles in the cooler with sufficient space to allow for the addition of more foam between the bottles.
7. Put ice on top of and/or between the samples.
8. Place chain of custody in a clean dry bag and into the cooler. Close the cooler and securely tape the cooler shut. The chain of custody seals should be affixed to the top and sides of the cooler so that the cooler cannot be opened without breaking the seal.
9. The shipping containers must be marked "THIS END UP". The name and address of the shipper shall be placed on the outside of the container. Labels used in the shipment of hazardous materials are not permitted to be on the outside of the container used to transport environmental samples and shall not be used.

16.0 INVESTIGATION WASTE

16.1 GENERAL

Field surveys conducted by ESC may generate waste materials. Some of these waste materials may be hazardous requiring proper disposal in accordance with EPA regulations.

16.1.1 Types of Investigation Derived Wastes (IDW)

Materials which may be included in the IDW category are:

- Personnel protective equipment (PPE)
- Disposable sampling equipment (DE)

- Soil cuttings
- Groundwater obtained through well purging
- Spent cleaning and decontamination fluids
- Spent calibration standards

16.1.2 Managing Non-hazardous IDW

Disposal of non-hazardous IDW should be addressed prior to initiating work at a site. Facility personnel should be consulted and wastes handled in an appropriate manner as directed by the client.

For development and purge water generated in the State of Florida, specific disposal requirements apply. The water shall be contained on-site in temporary storage until it is characterized. Appropriate disposal and/or treatment methods will then be determined. Possible disposal options are:

- Direct discharge on-site to infiltrate the same or a more contaminated source
- Transportation to an off-site facility

In no case shall the water be discharged into any surface water unless permitted.

16.1.3 Management of Hazardous IDW

Disposal of hazardous or suspected hazardous IDW (as defined in 40 CFR 261.30-261.33 or displaying the characteristics of ignitability, corrosivity, reactivity, or TC toxicity) must be specified in the sampling plan. Hazardous IDW must be disposed in compliance with USEPA regulations. If appropriate, these wastes may be taken to a facility waste treatment system. These wastes may also be disposed of in the source area from which they originated if state regulations permit.

If on-site disposal is not feasible, appropriate analyses must be conducted to determine if the waste is hazardous. If so, they must be properly contained and labeled. They may be stored on the site for a maximum of 90 days before they must be manifested and shipped to a permitted treatment or disposal facility. Weak acids and bases may be neutralized in lieu of disposal as hazardous wastes. Neutralized wastewaters may be flushed into a sanitary sewer.

If possible, arrangements for proper containment, labeling, transportation, and disposal/treatment of IDW should be anticipated beforehand.

Investigation derived wastes should be kept to a minimum. Most of the routine studies conducted by ESC should not produce any IDW that are hazardous. Many of the above PPE and DE wastes can be deposited in municipal dumpsters if care is taken to keep them segregated from hazardous waste contaminated materials. Disposable equipment can often be cleaned to render it nonhazardous, as can some PPE, such as splash suits. The volume of spent solvent waste produced during equipment decontamination can be reduced or eliminated by applying only the minimum amount of solvent necessary.

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1.0 SIGNATORY APPROVALS

WET LAB QUALITY ASSURANCE MANUAL

APPENDIX IV TO THE ESC QUALITY ASSURANCE MANUAL

for

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2.0 APPENDIX TABLE OF CONTENTS

3.0 SCOPE AND APPLICATION

This manual discusses specific QA requirements for general analytical protocols to ensure analytical data generated from the Wet Chemistry Laboratory, or Wet Lab, are scientifically valid and are of acceptable quality. Any deviations from these requirements and any deviations that result in nonconforming work must be immediately evaluated and their corrective actions documented.

4.0 LABORATORY ORGANIZATION AND RESPONSIBILITIES

ESC Lab Sciences offers diverse environmental capabilities that enable the laboratory to provide the client with both routine and specialized services, field sampling, and broad laboratory expertise. A brief outline of the organization and responsibilities as they apply to the ESC Quality Assurance Program is presented in *Section 4.0 in the ESC Quality Assurance Manual Version 8.0.*

5.0 PERSONNEL AND TRAINING

5.1 PERSONNEL

Kenneth W. Buckley, with a B.S. degree in General Science, is the Department Manager of the Organics and Wet Chemistry laboratories. Mr. Buckley reviews and approves all data reduction associated with analyses in these areas and is responsible for the overall production of these laboratories; including the management of the staff and scheduling. Mr. Buckley has over 9 years of environmental laboratory experience. In his absence, Chad Pfalmer assumes responsibility for departmental decisions in the Wet Lab.

5.2 TRAINING

5.2.1 All new analysts to the laboratory are trained by a primary analyst or Manager according to ESC protocol. Performance is documented using an initial demonstration of capability (IDOCs) and continuing demonstration of capability (CDOC). On-going acceptable capability in Wet Lab analyses is demonstrated by acceptable participation in multiple proficiency testing programs (PTs) and daily Quality Control sample analyses. Documentation of analyst training is maintained on file within the department.

6.0 FACILITIES AND LABORATORY SAFETY

6.1 FACILITIES

The main area of the laboratory has approximately 2800 square feet with roughly 750 square feet of bench area. There is an additional 400 square feet of storage space and the lighting standard is fluorescence. The air system is a 5-ton Trane package unit and a 10-ton Trane package unit with natural gas for heating. The laboratory reagent water is provided through the US Filter deionizer system with a Millipore Milli-Q Academic A-10 system for finished water. Waste disposal containers are located in the laboratory and Clean Harbors serves as ESC's waste disposal contractor. ESC's building information guides and site plan are shown in Appendix I.

6.2 LABORATORY SAFETY

- Laboratory access is limited when work is being performed.
- All procedures where chemicals are prepared or splashes may occur are conducted in laboratory exhaust hoods.
- ESC's laboratory safety guidelines are detailed in the *ESC Chemical Hygiene and Safety Plan.*

7.0 SAMPLING PROCEDURES

7.1 FIELD SAMPLING PROCEDURES, SAMPLE STORAGE, AND HANDLING

- Field Sampling procedure is described in Appendix III of this ESC Quality Assurance Manual. Sample information is recorded and kept on the ESC chain of custody and field logbooks.
- Matrices for Wet Lab environmental analyses include groundwater, wastewater, drinking water, soil, and sludge. The Wet Lab also performs analyses on sorbent media and air filters for Industrial Hygiene monitoring.
- Sample containers, preservation methods and holding times vary depending on analyses requested. Please see the determinative procedures for specific directions.

8.0 EQUIPMENT

8.1 EQUIPMENT LIST

LABORATORY EQUIPMENT LIST: MAJOR ITEMS – Wet Lab
This table is subject to revision without notice

Item	*Manufacturer*	*Model*	*Instrument Name*	*Serial #*	*Location*
Analytical Balance	Mettler	AT200	Balance	m26291	Wet Lab
Analytical Balance	Mettler	AG204 Delta Range	Balance	118420883	Wet Lab
Analytical Balance	Mettler	xs204	OG balance	11619	Wet Lab
Autoanalyzer	Lachat	Quikchem 8000	Lachat 2	1027	Wet Lab
Autoanalyzer	Lachat	Quikchem 8000	Lachat 3	1638	Wet Lab
Autoanalyzer	Lachat	Quikchem 8500	Lachat 4	060900000341	Wet Lab
Autoanalyzer	Lachat	Quikchem 8500	Lachat 5	060900000342	Wet Lab
Autoanalyzer - digestor	Lachat	BD-46	DIG1	1800-772	Wet Lab
Autoanalyzer - digestor	Lachat	BD-46	DIG2	1800-7m21	Wet Lab
Automated distiller	Skalar	SAN++ system	Kelada 1	09719	Wet Lab
Automated titrator	Metrohm	855 titrosampler	Titrando	3256	Wet Lab
Centrifuge	Beckman	Spinchron R	Centrifuge	100515	Wet Lab
Class “I” weights	Troemner	Serial #7944		7944	Wet Lab
COD Reactor	HACH	45600	COD1	22195	Wet Lab
COD Reactor	HACH	45600	COD2	985	Wet Lab
Conductivity Meter	ORION	MODEL 170		32470007	Wet Lab
Distillation Unit - Cyanide	Kontes Glass Co.	Model Cal 3200		501	Wet Lab
Distillation Unit - Cyanide	Kontes Glass Co.	Model Cal 3200		2193	Wet Lab
Distillation Unit - Cyanide	Kontes Glass Co.	Model Cal 3200			Wet Lab
Distillation Unit - Phenol	Westco Scientific	Model EASY-DIST		1062	Wet Lab
Distillation Unit - Phenol	Westco Scientific	Model EASY-DIST		Spare	Wet Lab
Flash Point Tester	Koehler	Pensky-Martens K16200	Manual	13576	Wet Lab
Flash Point Tester	Petrolab	Petrotest	Auto	8851	Wet Lab
Hot Plate	Thermolyne Fisher	Type 2200	Hot	16237	Wet Lab
Hot Plate	Thermolyne Fisher	Type 2200	Hot	16240	Wet Lab
Ion Chromatograph	Dionex	DX1500	IC1	08100010	Wet Lab
Ion Chromatograph	Metrohm	850 Professional	IC2	1860	Wet Lab
Ion Chromatograph	Dionex	ICS 2000	IC3	06050731	Wet Lab
Ion Chromatograph	Dionex	ICS 2000	IC4	08090820	Wet Lab
Ion Chromatograph	Dionex	ICS 1500	IC5	08090871	Wet Lab

LABORATORY EQUIPMENT LIST: MAJOR ITEMS – Wet Lab
This table is subject to revision without notice

Item	*Manufacturer*	*Model*	*Instrument Name*	*Serial #*	*Location*
Muffle Furnace	Thermolyne	(1) 30400		23231	Wet Lab
Oven - Drying	VWR	1305U	#1	202597	Wet Lab
Oven - Drying	Equatherm	D1576	#3	not available	Wet Lab
Oven - Drying	Equatherm	D1576	#4	74845	Wet Lab
Oven - Drying	VWR	1305U	#6	not available	Wet Lab
Oven - Drying	Fisher	Isotemp 655G	OG oven	00127	Wet Lab
pH Meter	Fisher	AB15		AB92322670	Wet Lab
pH Meter	Orion	410A		05074	Wet Lab
Refrigerated Recirculator	VWR	117P		not available	Wet Lab
Refrigerated Recirculator	VWR	117P		not available	Wet Lab
Spectrophotometer (UV/Vis)	Hach	DR 4000U		not available	Wet Lab
Spectrophotometer (UV/Vis)	Hach	DR 4000U		not available	Wet Lab
Total Organic Carbon Analyzer	Shimadzu	Model TOC-VWS	Persulfate	39830872	Wet Lab
Total Organic Carbon Analyzer	Shimadzu	5000A	Combustion	36301649	Wet Lab
Total Organic Halogen Analyzer	Mitsubishi	TOX-100		02909	Wet Lab
Total Organic Halogen Analyzer	Mitsubishi	TOX-100		00247	Wet Lab
Turbidimeter	Hach	2100N		not available	Wet Lab

8.2 EQUIPMENT PREVENTIVE MAINTENANCE, EQUIPMENT CALIBRATION

INSTRUMENT	*P. M. DESCRIPTION*	*FREQUENCY*
Analytical Balances	•Check with Class "I" weights	Daily
Analytical Balances	•Service/Calibration (semi-annual contract maintenance and calibration check)	Tolerance - ±0.1%
Analytical Balances	•Service/Calibration (semiannual contract maintenance and calibration check)	Semi-annually
Refrigerators & Incubators	•Maintenance service	As needed - determined by daily temperature performance checks
Water Bath	•Check thermometer vs. NIST	Once/year
Water Bath	•Remove from service when not maintaining temperature and send off for repair or replace	As needed
Flash Point Tester	•Check thermometer vs. certified traceable	Once/year
Lachat Autoanalyzer	•Check pump tubes, change valve flares	At least 1/month
Pensky Martens	•Check fuel level, refill	As needed
Pensky Martens	•Clean cup thoroughly	Between each test and after use
TOC	•Maintain manufacturer's service contract	Renew each year
Turbidimeter - Hach 2100A	•Illumination lamp or window (alignment and/or replacement)	Erratic or poor response
pH Meters	•Reference junction & electrode replacement	As needed
pH Meters	•Probe stored in KCl	At all times when not in use
pH Meters	•Other	As described in the manufacturer's O & M manual

8.3 STANDARDS AND REAGENTS

Table 8.3A lists standard sources, receipt, and preparation information. Table 8.3B is designed to provide general calibration range information. These ranges may change depending on regulatory requirements, procedural changes, or project needs. Table 8.3C indicates the procedures and frequency for the standardization of laboratory solutions used for titrations.

Table 8.3A: Standard sources, description and calibration information.
This table is subject to revision without notice

Instrument Group	Standard Source	How Received*	Source/ Storage	Preparation from Source	Lab Stock Storage	Preparation Frequency
Alkalinity, Acidity	Lab preparation	Acidity-matrix standard grade KHP	Room temp.	0.0500N	4°± 2°C	6 months
Ammonia-Nitrogen and Total Kjeldahl Nitrogen	Lab preparation	ACS grade NH4Cl	Room temp.	1,000ppm stock standard	Room temp.	Annually or sooner if check samples reveal a problem
Ammonia-Nitrogen and Total Kjeldahl Nitrogen				Working Standards	Not stored	Prepared fresh as needed
BOD	Lab preparation	As dry glucose and glutamic acid	Dessicator	150mg of each/L	4°± 2°C	Made fresh daily
COD	Lab preparation	Acid grade KHP	Dessicator	Stock solution (10,000ppm)	4°± 2°C	When absorbance of curve changes or check samples are out of control
Cyanide (Autoanalyzer)	Lab preparation	KCN	Reagent shelf	Stock solution (1,000ppm)	4°± 2°C	6 months. Working dilutions prepared daily as needed
Fluoride	Inorganic Standard. NSI Lab preparation	ACS grade KF	Room temp.	100ppm stock solution	Room temp.	1 year or as needed when reference standard fails
Fluoride				Dilute standards	Not stored	Prepared fresh daily
Hardness	Lab preparation	Chelometric Std. $CaCO_3$	Room temp.	1mg/mL as CaCO3	Room temp.	Annually or sooner if check samples reveal a problem
IC (Chloride, Nitrate, Nitrite, Bromide, Sulfate, Fluoride)	Commercial source	Varies	4°± 2°C	Working Standards as needed per analyte	4°± 2°C	6 months or sooner if check samples reveal a problem
IC (Chloride, Nitrate, Nitrite, Bromide, Sulfate, Fluoride)	Inorganic Standards	Varies	4°± 2°C	Working Standards as needed per analyte	4°± 2°C	Midpoint standard prepared weekly or sooner if necessary
IC (Chloride, Nitrate, Nitrite, Bromide, Sulfate, Fluoride)	NSI (2nd source)	Varies	4°± 2°C	Working Standards as needed per analyte	4°± 2°C	Prepared weekly or sooner if necessary
MBAS	Lab preparation	LAS Reference Material	4°± 2°C	1,000mg/mL working standards	4°± 2°C Wet Stored	6 months or when check standards are out of control. Prepared fresh.

Table 8.3A: Standard sources, description and calibration information.

This table is subject to revision without notice

Instrument Group	Standard Source	How Received*	Source/ Storage	Preparation from Source	Lab Stock Storage	Preparation Frequency
Nitrite-Nitrate (autoanalyzer)	Lab preparation	ACS grade KNO3	Reagent shelf	Stock solution (1000ppm)	4°± 2°C	When absorbance of curve changes or check samples are out of control
pH Meter	Commercial Source	pH 4.0 Buffer	Room temp.	No prep required	NA	Annual/Expiration Date
pH Meter	Commercial Source	pH 7.0 Buffer	Room temp.	No prep required	NA	Annual/Expiration Date
pH Meter	Commercial Source	pH 10.0 Buffer	Room temp.	No prep required	NA	Annual/Expiration Date
Phenols (autoanalyzer)	Lab preparation	ACS Certified Phenol	Reagent shelf	Stock solution (1000ppm)	4°± 2°C	Every month. Working solutions prepared daily as needed.
Phosphate	(H2O) - Prepared in Lab Total Phos. (soils) RICCA, ERA	KH2PO4	Reagent shelf	Stock solution (50ppm as P)	Room temp.	When absorbance of curve changes or check samples are out of control. Working solutions prepared daily as needed.
Specific Conductivity Meter	NSI-Primary	ACS Certified KCl	Room temp.	Working Standard (0.01M)	Room temp.	As needed
Specific Conductivity Meter	ERA-2nd Source	ACS Certified KCl	Room temp.	Working Standard (0.01M)	Room temp.	As needed
Sulfate	Inorganic Standards, NSF Prepared in Lab	Anhydrous Na2SO4	Reagent shelf	Stock solution (100ppm)	Room temp.	When visible microbiological growth or check samples are out of control
Turbidimeter	Commercial Source Hach	Hach	Room temp.	No prep required	NA	Checked daily against Formazin Standards

TABLE 8.3B: WORKING STANDARD CALIBRATION	
Analysis	**Calibration Standard**
Alkalinity, Acidity- Titrimetric	Primary standard grade Na_2CO_3.
Alkalinity - Methyl orange Autoanalyzer	Primary standard grade Na_2CO_3: 0, 10, 25, 50,100, 250, 375, 500 mg/L
BOD	D.O.-Barometric pressure/temp., Glucose and Glutamic acid reference standard.
Bromate IC	Low Range – 5.0, 10, 20, 30, 50, 100 ug/L
Bromide IC	Range –1.0, 5.0, 10, 50, 100, 150, 200 mg/L
Chlorate IC	Low Range – 5.0, 10, 20, 30, 50, 100 ug/L High Range – 10, 20, 50, 100, 200, 400, 600 ug/L
Chloride IC	Range –1.0, 5.0, 10, 50, 100, 150, 200 mg/L
Conductivity	Standard KCl solution: 1413
Cyanides	Blank, 0.0025 – 0.40ppm. Distill one standard as check with each batch.
COD	KHP (Potassium hydrogen phthalate) standards 20 – 1000 mg/L
Chromium – Hexavalent (Colorimetric)	Blank, 0.0101, 0.0202, 0.0505, 0.1010, 0.2525, 0.5050, 1.010 mg/L
Chromium – Hexavalent (IC)	Blank, 0.5, 1.0, 2.0, 10, 20, 50, 100 ug/L
Fluoride – IC	Range –0.10, 0.50, 1.0, 5.0, 10.0, 15.0, 20.0 mg/L
Hardness	$CaCO_3$, chelometric standard.
Hardness (Colorimetric)	Range – 30, 50, 60, 100, 150, 200, 300 mg/L
MBAS	LAS reference material: 0.0, 0.1, 0. 5, 1.0, 1.5, 2.0 mg/L
Nitrogen-Ammonia – Autoanalyzer	Calibration standards: 0.05, 0.10, 0.50, 1.0, 2.0, 5.0, 10, 20 mg/L
Nitrogen-Nitrate, Nitrite – Autoanalyzer	Blank, 0.1, 0.50, 1.00 5.0, 7.0, 10.0 mg/L
Nitrogen-Nitrate – IC	Range –0.10, 0.50, 1.0, 5.0, 10.0, 15.0, 20.0 mg/L
Nitrogen-Nitrite – IC	Range –0.10, 0.50, 1.0, 5.0, 10.0, 15.0, 20.0 mg/L
Orthophosphate, Total Phosphate	Blank, 0.025, 0.10, 0.25, 0.50, 0.75, 1.0mg/L diluted from standard KH_2PO_4
Perchlorate	Range – 0.5, 1.0, 3.0, 5.0, 10, 20, 25 mg/L
pH	Buffers1.0, 4.0, 7.0, 10, 13
Phosphate, Total	Range – 0.0, 0.1, 0.5, 1.0, 2.5, 5.0 mg/L
Phosphate – IC	Range –0.10, 0.50, 1.0, 5.0, 10.0, 15.0, 20.0 mg/L
Phenols (chloroform ext.)	Blank 0.04, 0.05, 0.10, 0.50, 1.0, 2.0mg/L Distill one standard with each batch
Solids	Gravimetric balance calibrated charts, checked with Class “I” weights in range of sample tare weights.
Sulfate – IC	Range –1.0, 5.0, 10, 50, 100, 150, 200 mg/L
Sulfide (Colormetric)	Range –0.0, 0.05, 0.1, 0.5, 1.0, 1.5, 2.0 mg/L
Sulfite	Titration
TKN	Range – 0.0, 0.1, 0.5, 1.0, 2.5, 5.0, 10, 20 mg/L
Turbidity	Range –0, 20, 200, 1000, 4000NTU
TOC	Range –0, 1.0, 2.5, 5.0, 7.5, 10, 20, 50, 75, 100 mg/L
ToX	Cell checks at 1, 20, 40 ug

TABLE 8.3C: STANDARDIZATION OF TITRATION SOLUTIONS		
Solution	**Primary Standard**	**Frequency**
0.0200 N NaOH	0.050 N KHP	Daily as needed
0.0200 N H_2SO_4	Freshly prepared and standardized NaOH (from KHP standard)	6 months or with each new batch
0.0141 N Hg $(NO_3)_2$	Standard NaCl solution 500 ug Cl/ml	Daily as used
0.0100 M EDTA	Standard $CaCO_3$ solution 1 mg $CaCO_3$/liter	Daily as used

8.4 INSTRUMENT CALIBRATION

Total Organic Carbon Analyzer (TOC) – SOP Number 340356A

The TOC standard curve is prepared using a minimum of five standards. Linear regression is used for quantitation with the correlation coefficient being at least 0.995. The calibration range is 1.0mg/L to 100mg/L. During the analytical sequence, the stability of the initial calibration is verified, following every 10th sample and at the end of the sequence, by the analysis of continuing calibration verification (CCV) standards. The CCV must recovery within 10% of the expected value for each analyte.

A laboratory control standard (LCS) is prepared from a source that is independent from the calibration standards and used to verify that the calibration curve is functioning properly and that the analytical system performs acceptably within a clean matrix. The LCS must recover within ±15% of the expected concentration.

Total Organic Halogen Analyzer (TOX) – SOP Number 340360

The cell performance of the TOX analyzer is verified at the beginning of each analytical sequence in the low, mid and high ranges. The verifications must recover within 3% of the expected target value. The instrument performs a linear regression using the values determined with the required correlation coefficient being at least 0.995. During the analytical sequence, the stability of the initial calibration is verified, following every 10th sample and at the end of the sequence, by the analysis of continuing calibration verification (CCV) standards. The CCV must recovery within 10% of the expected value for each analyte.

A laboratory control standard (LCS) is prepared from a source that is independent from the calibration standards and used to verify that the calibration curve is functioning properly and that the analytical system performs acceptably within a clean matrix. The LCS must recover within ±15% of the expected concentration.

Anions by Ion Chromatography – SOP 340319

Quadratic Fit is the primary method of quantitation; however Linear Regression is required for sample analyzed in conjunction with the Ohio VAP program. When using quadratic fit a minimum of six standards are used. If linear regression is used for quantitation, a minimum of five standards is used and the correlation coefficient must be at least 0.995 for each analyte of interest. The calibration range varies depending upon the analyte(s) to be determined. During the analytical sequence, the stability of the initial calibration is verified, following every 10th sample and at the end of the sequence, by the analysis of continuing calibration verification (CCV) standards. The CCV must recovery within 10% of the expected value for each analyte, except during the analysis of groundwater and soil using EPA Method 9056 that recover within 5%.

A laboratory control standard (LCS) is prepared from a source that is independent from the calibration standards and used to verify that the calibration curve is functioning properly and that the analytical system performs acceptably within a clean matrix. The LCS must recover within ±10% for water samples and 15% of the expected concentration for soil samples.

Auto-Analyzer (Lachat) – Various SOPs

The Autoanalyzer calibration curve is prepared using a minimum of five standards. For most analyses, linear regression is used for quantitation with the correlation coefficient being at least 0.995. The calibration range varies depending upon the analyte to be determined. During the analytical sequence, the stability of the initial calibration is verified, following every 10th sample and at the end of the sequence, by the analysis of continuing calibration verification (CCV) standards. Routinely, the CCV must recovery within 15% of the expected value for each analyte, but is dependent on the analyte of concern, the matrix of the sample and the determinative method.

A laboratory control standard (LCS) is prepared from a source that is independent from the calibration standards and used to verify that the calibration curve is functioning properly and that the analytical system performs acceptably within a clean matrix. The LCS must recover within ±15% of the expected value, except for cyanide where ±10% applies.

Gravimetric Analyses – Various SOPs

Gravimetric analyses are performed using several different published methods, including TDS, TSS, TVDS, TS, TVS, VSS, Settleable Solids, Total Particulates, Respirable Particulates. Calibration for these methods require use of Class I weights and a properly performing and verified balance. Where possible, laboratory control standards are analyzed in conjunction with field sample analysis to verify that the analytical process is performing accurately. Sample duplicate analyses also provide verification that the analytical process is performing as required.

Perchlorate in Drinking Water – ESC SOP 340370

The Ion Chromatograph calibration curve is prepared using a minimum of five standards. The instrument performs a linear regression using the values determined with the required correlation coefficient being at least 0.995. During the analytical sequence, the stability of the initial calibration is verified, following every 10th sample and at the end of the sequence, by the analysis of continuing calibration verification (CCV) standards. The CCV must recovery within 15% of the expected value for each analyte.

A laboratory control standard (LCS) is prepared from a source that is independent from the calibration standards and used to verify that the calibration curve is functioning properly and that the analytical system performs acceptably within a clean matrix. The LCS must recover within ±15% of the expected concentration.

8.5 ACCEPTANCE/REJECTION OF CALIBRATION

The initial calibration curve is compared with previous curves for the same analyte. The curve is checked for linearity and the response must be within 10% of the previous curve. All new standard curves are immediately checked with a laboratory control standard from a separate source than that used for calibration. All curves are visually reviewed to ensure that acceptable correlation represents linearity. Calibration curves may be rejected for nonlinearity, abnormal sensitivity, or poor response of the laboratory control standard. Specific criteria for each instrument are outlined in Table 8.5.

Continuing calibration is performed following every tenth sample. If a check standard does not perform within established criteria then the instrument is evaluated to determine the problem. Once the problem is corrected, all samples between the last “in control” sample and the out of control check are re-analyzed.

TABLE 8.5: INSTRUMENT CALIBRATION

Instrument (Analysis)	Calibration Type	Number of Standards	Type of Curve	Acceptance/Rejection Criteria	Frequency
pH Meter*	Initial	5 (buffers) 1 reference buffer	Log.	Third pH of a different value buffer must read within 0.05 units of true value	Daily as used
	Continuing	1 buffer (may be any certified buffer)		Buffer solution must read within 0.05 units of true value	Every 10th sample; Field**
Conductivity Meter*	Initial	1	1 point	Calculation of cell constant between 0.95 - 1.05	Daily as used
	Continuing	1		Must be within 5% of true value	Every 10th sample; Field**
Turbidimeter *	Initial	5	Linear	Formazin-confirmed Gelex standards in appropriate range. Check with second standard must be within 5%	Daily as used
	Continuing	1 reference of different value, 1 (high-level)		Must be within 5% of true value	Every 10th sample; Field**
UV/VIS Spec.	Initial	At least 5 standards calibration standards 2 laboratory control standard	Linear	Calibration Curve must have a correlation of 0.995 or better Must be within ± 15% of the calibration curve.	Daily as used Daily as used
	Continuing	1 mid-level reference std.		Must be within 90 – 110%	Every 10th sample
Total Organic Halogen Analyzer	Initial	3 calibration standards 1 laboratory control standard	Linear	Calibration Curve must have a correlation of 0.995 or better Laboratory control standard must agree within ± 15% of calibration curve	Daily as used Daily as used
	Continuing	1 mid-level reference std.		Must be within 90 – 110%	Every 10th sample
Total Organic Carbon Analyzer	Initial	5 calibration standards 2 laboratory control standard	Linear	Calibration Curve must have a correlation of 0.995 or better Laboratory control standard must agree within ± 15% of calibration curve	Every 6 months or as needed Daily as used
	Continuing	1 mid-level reference std.		Must be within 90 – 110%	Every 10th sample

Note: ESC defines a "laboratory control standard" as a standard of a different concentration and source than those stock standards used for calibration.
*This equipment is also calibrated and used in the field.
**Field equipment must be checked every 4 hours and at the end of the day.

9.0 LABORATORY PRACTICES

9.1 REAGENT GRADE WATER

Reagent grade water is obtained from either a Barnstead NANOpure Diamond system or the Millipore Milli-Q Academic A-10 system.

9.2 GLASSWARE WASHING AND STERILIZATION PROCEDURES

General

Routine laboratory glassware is washed in a non-phosphate detergent and warm tap water. Before washing all labeling and large deposits of grease are removed with acetone. Glassware is then rinsed with: tap water, "No Chromix" solution, tap water, and deionized (DI) water. Glassware is stored in designated drawers or on shelves, inverted when possible. All glassware is rinsed with the required solvent, prior to use. DI water is then used as a precaution against airborne contamination

Phosphate Glassware

Glassware involved in phosphate analysis is marked and segregated. All labels and markings are removed from the glassware prior to washing. The glassware is then washed using hot water and a non-phosphorus detergent. It is then rinsed thoroughly in hot water followed by a rinse in DI water. It is rinsed in 1:1 HCl followed by a final rinse of DI water. If the phosphate glassware has not been used recently, it is the responsibility of the analyst to rinse the glassware with warm 1+9 hydrochloric acid prior to use.

Nutrients and Minerals Glassware

All labels and markings are removed from the glassware prior to washing. The glassware is then washed using hot water and detergent. It is then rinsed thoroughly in hot water followed by a rinse in DI water. It is rinsed in 1:1 HCl followed by a final rinse of DI water. Immediately prior to use, the ammonia glassware is rinsed in DI water. Routine blanks are run on ammonia glassware to ensure that the detergent is contaminant free.

Non-Metals (CN, BOD, COD) Glassware

All labels and markings are removed prior to washing. The glassware is soaked in hot soapy water followed by a thorough rinse with hot tap water. A final rinse of DI water is then performed.

BOD analysis is performed in disposable, pre-sterilized bottles. In the event that glass bottles must be used, the BOD glassware is washed in a commercial laboratory dishwasher using a phosphate free detergent, followed by a nitric acid rinse, with a final rinse of laboratory DI water.

10.0 ANALYTICAL PROCEDURES

10.1 A list of laboratory SOP's associated with the Wet Lab can be found in the following table:

TABLE 10.1: WET LAB DEPARTMENT SOP'S

This table is subject to revision without notice

SOP #	Title
340300	Acidity
340301	Alkalinity (Titrimetric)
340302	Alkalinity - Lachat
340303	Biochemical Oxygen Demand
340305	Chlorine, Total Residual
340306	Corrosivity
340307	Cyanide- All Forms (Colorimetric Automated UV) - Lachat
340309	Chemical Oxygen Demand
340310	Color by Visual Comparison
340313	Density (Specific Gravity)
340317	Total Hardness by Lachat
340317	Total Hardness (mg/l as CaCO3) - (Titrimetric)
340318	Hexavalent Chromium (Colorimetric) Water/Soil
340319	Ion Chromatography - Anions
340325	MBAS (Methylene Blue Active Substances)
340327	Ammonia, Phenolate (Lachat)
340328	Organic Nitrogen
340331	Threshold Odor Test
340333	Nitrate/Nitrite (Lachat Autoanalyzer)
340334	Paint Filter Test
340335	pH
340336	Phenol - 4AAP (Lachat Autoanalyzer)
340338	Orthophosphate Colorimetric
340338	Total Phos. Colorimetric
340339	Reactivity
340340	Reactive Cyanide/Sulfide Distillation
340342	Specific Conductance

SOP #	Title
340344	Sulfide (Colorimetric Methylene Blue)
340344	Sulfide Acid-soluble,and acid-insoluble
340345	Sulfite
340346	Settleable Solids
340347	Total Dissolved Solids
340348	Total Suspended Solids (Non-Filterable Residue)
340349	Total Solids/Percent Moisture
340350	Total Volatile Solids
340352	Total Kjeldahl Nitrogen
340356	Total Organic Carbon In Soils (loss of weight on ignit.)
340356	TOC for Drinking Water only
340356	Total Organic Carbon (TOC) and Total Inorganic Carbon (TIC)
340357	Ignitability
340357	Ignitability
340359	UV254
340360	TOX (total organic halides)
340361	Ferrous Iron
340362	Heat of Combustion
340365	Particles Not Otherwise Regulated, Total (PNOR)
340366	Oxidation Reduction Potential
340367	Extractable Organic Halides
340368	TOC in Soil (Walkley-Black)
340369	Carbon Dioxide by Calculation
340370	Perchlorate in DW
340371	Chlorine in Oil
340372	Hexavalent Chromium in Water by IC
340373	Organic Matter (FOM) and Fractional Organic Carbon (FOC)
340374	Total Volatile Dissolved Solids (TVDS)
340375	Hexavalent Chromium in Air by IC
340376	Total Organic Halides in Oil
340377	Manual Nitrocellulose Analysis
340378	Volatile Suspended Solids
340379	Guanidine Nitrate by IC

11.0 QUALITY CONTROL CHECKS

NOTE: For specific guidance on each determinative method, including required quality control and specific state requirements/modifications, refer to the relevant laboratory standard operating procedure(s).

11.1 ESC participates in proficiency testing (PT's) in support of various laboratory accreditations/recognitions. Environmental samples are purchased from Environmental Resource Associates (ERA). The WS, WP and solid matrix studies are completed every 6 months.

11.2 Initial Demonstrations of Capability (IDOC's) are performed during new analyst training and/or prior to acceptance and use of any new method/instrumentation. Continuing Demonstration of Capability (CDOC's) must be updated at least annually. The associated data is filed within the department and available for review.

11.3 Where appropriate, Matrix Spike and Matrix Spike Duplicates are performed on each batch of samples analyzed, depending on analytical method requested.

11.4 A Laboratory Control Sample (LCS) is analyzed once per batch of samples. Where appropriate, an LCS Duplicate may also be analyzed.

11.5 Where appropriate, a method preparation blank is performed per batch of samples processed. If one-half the reporting limit [RL] is exceeded, the laboratory shall evaluate whether reprocessing of the samples is necessary, based on the following criteria:

- The blank contamination exceeds a concentration greater than 1/10 of the measured concentration of any sample in the associated preparation batch or
- The blank contamination is greater than 1/10 of the specified regulatory limit.

The concentrations of common laboratory contaminants shall not exceed the reporting limit. Any samples associated with a blank that fail these criteria shall be reprocessed in a subsequent preparation batch, except when the sample analysis resulted in non-detected results for the failing analytes.

12.0 DATA REDUCTION, VALIDATION AND REPORTING

12.1 DATA REDUCTION

The analyst performs the data calculation functions and is responsible for the initial examination of the finished data. Data reduction steps applied to the raw data are outlined in ESC SOP #030201, *Data Handling and Reporting.* The Quality Control Department performs the secondary review of the data package using the ESC SOP #030227, *Data Review*. The QC Reviewer verifies that the analysis has performed as required and meets method criteria, all associate data is present and complete, and also ensures that any additional documentation is completed as required (i.e. Ohio VAP checklists, required flags on test reports, etc.)

TABLE 12.1: Data Reduction Formulas

PARAMETER	FORMULA
Acidity, Alkalinity	mL titrant x normality titrant x 50,000 / mL sample
BOD, 5-day	Initial D.O. - Final D.O. - CF / % Dilution Sample *Calculations are performed by computer software*
Boron, COD, Sulfate	Concentration from curve x dilution factor
Nitrogen-Nitrate, Nitrite, Nitrogen-Nitrite, Ortho and Total Phosphate, Phenols, Chloride	Calculated by computer software as provided by Lachat Corp.
Fluoride**, Nitrogen-Ammonia**, Nitrogen-Total Kjeldahl**	Calculated by computer software as provided by Lachat Corp.
Anions	Calculated by computer software as provided by Dionex
Conductivity*, pH, Turbidity,	Directly read from instrument
Cyanide, Total and Amenable	µg from standard curve x mL total volume absorbing solution / mL volume sample x mL volume of absorbing solution colored *Calculated by software as provided by Lachat Corp.*
Solids, Total and Total Dissolved	((mg wt of dried residue + dish) - mg wt of dish) x 1000 / mL sample
Solids, Total Suspended	((mg wt of dried residue + filter) - mg wt of filter) x 1000 / mL sample

12.2 VALIDATION

The validation process consists of data generation, reduction review, and reporting results. Once data reduction is complete, validation is conducted by verification that the QC samples are within acceptable QC limits and that all documentation is complete, including the analytical report and associated QC. See Table 12.3 by method for current QC targets, controls and current reporting limits.

12.3 REPORTING

Reporting procedures are documented in *SOP 030201 Data Handling and Reporting.*

Inorganic Control Limits: Inorganic QC targets are statutory. The laboratory calculated limits verify the validity of the regulatory limits. The Wet Lab QC targets for all inorganic analyses are within the range of $\pm$ 5 to 15% for accuracy, depending on determinative method requirements, and, where applicable, $\leq$20 RPD for precision, unless laboratory-generated data indicate that tighter control limits can be routinely maintained. When using a certified reference material for QC sample analysis, the acceptance limits used in the laboratory will conform to the provider's certified ranges for accuracy and precision.

Table 12.3: QC Targets for Wet Lab Accuracy (LCS), Precision and RL's
This table is subject to revision without notice

Analyte	Analysis Method	Matrix	Accuracy Range (%)	Precision (RPD)	RL (ppb)
Acidity	SM 2310B	w	85 - 115	<20	1000
Alkalinity	SM 2320	w	85 - 115	<20	10000
Ammonia	350.1, SM 4500-NH3-H	w	85 - 115	<20	100
Ammonia	350.1 (mod.)	s	Certified Values	<20	500
Bromide	300.0/9056/9056A	w	90 - 110	<20	1000
Bromide	SM 4110B	w	90 - 110	<20	1000
Bromide	300.0	s	Certified Values	<20	10000
Chloride	300.0/9056/9056A	w	90 - 110	<20	1000
Chloride	SM 4110B	w	90 - 110	<20	1000
Chloride	300.0	s	Certified Values	<20	10000
Color	SM 2120-E	w	n/a	<20	1 pCu
Conductivity	120.1/9050A, 2510	w	85 - 115	<20	1000
Cyanide	335.3, 335.4, 335.2 (CLP-M), 9012A	w	90 - 110	<20	5
Cyanide	SM 4500-CN-E	w	90 - 110	<20	5
Cyanide	EPA 9012A	s	Certified Values	<20	250
Ferrous Iron	3500FE B	w	85 - 115	<20	50
Fluoride	300.0/9056/9056A	w	90 - 110	<20	100
Fluoride	SM 4110B	w	90 - 110	<20	100
Fluoride	9056A	s	Certified Values	<20	1000
Hardness	130.1	w	85 - 115	<20	30000
Hardness	SM 2340	w	85 - 115	<20	1000
Hexavalent Chromium	SM3500 CrD/7196A	w	85 - 115	<20	10
Hexavalent Chromium	7196A	s	Certified Values	<20	2000
Ignitability	1010	ws	$\pm$3 degrees C	<20	n/a
Methylene Blue Active Substances	5540C SM20th	w	85 - 115	<20	100
Nitrate-Nitrite	300	w	90 - 110	<20	100
Nitrate-Nitrite	SM 4110B	w	85 - 115	<21	100
Nitrate-Nitrite	9056/9056A	w	90-110	<20	100
Nitrate-Nitrite	9056/9056A	s	Certified Values	<20	1000
Nitrite	300.0/9056/9056A	w	90 - 110	<20	100
Nitrite	SM 4110B	w	90 - 110	<21	100
Nitrite	300.0/9056/9056A	s	Certified Values	<20	1000
Nitrate	300.0/9056/9056A	w	90 - 110	<20	100
Nitrate	SM 4110B	w	90 - 110	<20	100
Nitrate	300.0/9056/9056A	s	Certified Values	<20	1000
Moisture	Karl Fisher	ws	n/a	<20	n/a
pH	SM 4500-H, 9040B	w	n/a	<1	n/a
pH	9045C	s	n/a	<1	n/a

Table 12.3: QC Targets for Wet Lab Accuracy (LCS), Precision and RL's
This table is subject to revision without notice

Analyte	Analysis Method	Matrix	Accuracy Range (%)	Precision (RPD)	RL (ppb)
Phosphate (ortho)	SM 4500-P	w	85 - 115	<20	25
Phosphorous/Total	365.4, SM 4500-P	w	85 - 115	<20	25
Phosphorous/Total	365.4	s	Certified Values	<20	1000
Phosphorous/Total	9056/9056A	s	Certified Values	<20	1000
Residual Chlorine	SM 4500Cl G 20th	w	90 - 110	<20	100
Residue, Total (TS)	SM 2540-B, SM2540-G	w	85 - 115	<20	1000
Residue, Filterable (TDS)	SM 2540-C	w	95 - 105	<20	1000
Residue Non-Filterable (TSS)	SM 2540-D	w	95 - 105	<20	1000
Residue, Total Volatile (TVS)	160.4, SM 2540-E, SM2540-G	w,s	80 - 120	<20	1000
Sulfate	300.0/9056/9056A	w	90 - 110	<20	5000
Sulfate	SM 4110-B	w	90 -110	<20	5000
Sulfate	300.0/9056/9056A	s	Certified Values	<20	50000
Sulfide	SM 4500S2 D 20th	w	85 - 115	<20	100
Sulfite	SM 4500-SO3	w	85 - 115	<20	500
Total Kjeldahl Nitrogen	351.2	w	85 - 115	<20	500
Total Kjeldahl Nitrogen	351.2	s	Certified Values	<20	50000
Total Organic Carbon	415.1, SM 5310B&C, 9060	w	85 - 115	<20	1000
Total Organic Carbon	LOI	s	Certified Values	<20	10000
Dissolved Organic Carbon	415.1, SM 5310B&C, 9060	w	85 - 115	<20	1000
Total Inorganic Carbon	415.1, SM 5310B&C, 9060	w	85 - 115	<20	1000
Total Organic Halogens	9020A, SM 5320B	w	85 - 115	<20	10
EOX	9023	s	85 - 115	<20	20000
Total Phenol	420.2	w	85 - 115	<20	50
Total Phenol	9066	s, ws	Certified Values	<20	50
Turbidity	180.1, SM 2130	w	n/a	<20	1 NTU

13.0 CORRECTIVE ACTIONS

13.1 In the event that a nonconformance occurs in conjunction with the analytical batch, a corrective action response (CAR) form must be completed. The reason for the nonconformance is stated on the form and the measures taken to correct the nonconformance clearly defined. The effectiveness of the corrective action must be assessed and noted. The CAR is kept on file by the QA department. Corrective action procedures are documented in SOP 030208, *Corrective and Preventive Action*

13.2 Required Corrective Action

Control limits have been established for each type of analysis. When these control limits are exceeded, corrective action must be taken. Calculated sample spike control limits are also used.

All samples and procedures are governed by ESC's quality assurance program. General corrective actions are as follows; however additional and more specific direction is provided in the specific determinative procedure. For more information, see the appropriate determinative SOP.

13.2.1 Laboratory QC Criteria and Appropriate Corrective Actions

If the analytical method contains acceptance/rejection criteria and it is more stringent than those controls generated by the laboratory the method criteria take precedence.

13.2.2 Calibration Verification Criteria Are Not Met: Inorganic Analysis

Rejection Criteria - See Table 8.5.

Corrective Action - If a standard curve linearity is not acceptable and/or the absorbance for specific standard(s) is not analogous to historic data, the instrument settings, etc. are examined to ensure that nothing has been altered, clogged, etc. Check the standard curve for linearity and re-analyze the standards once. If the failure persists, the working standards will be made fresh, intermediate dilutions will be re-checked and the instrument will be re-calibrated. If a problem persists, the group supervisor or QA Department is notified for further action.

If the initial reference check sample is out of control, the instrument is re-calibrated and the check sample is re-analyzed. If the problem continues the check sample is re-prepared. If the problem still exists then the standards and reagent blank are re-prepared. If the problem persists, the group supervisor or QA Department is notified for further action.

13.2.3 Out Of Control Blanks: Applies to Method, Trip, Rinsate & Instrument Blanks

Rejection Criteria - Blank reading is more than twice the background absorbance or more than 1/2 RL.

Corrective Action - Blanks are re-analyzed and the response is assessed. Standard curves and samples are evaluated for any obvious contamination that may be isolated or uniform throughout the run. If necessary, reagents are re-prepared. Field sample analyses are not started until the problem is identified and solved. If samples have already been partially prepared or analyzed, the group leader or QA Department will be consulted to determine if data needs to be rejected or if samples need to be re-prepped.

13.2.4 Out Of Control Laboratory Control Standards (LCS)

Rejection Criteria - If the performance of associated laboratory control sample(s) is outside of lab-generated control limits calculated as the mean of at least 20 data points $\pm$ 3 times the standard deviation of those points. (Listed in Section 12).

Corrective Action - Instrument settings are checked, LCS standard is re-analyzed. If the LCS is still out of control, re-calibration is performed, and samples affected since the last "in control" reference standard are re-analyzed. The group leader, lab supervisor, or QA Department will be consulted for further action.

13.2.5 Out Of Control Matrix Spike Samples

Rejection Criteria - If either the MS or MSD sample is outside the established control limits from accuracy charts on matrix spike samples of a similar matrix (i.e., water, solid, etc). Limits are calculated as the mean ± three times the standard deviations.

Corrective Action - Spiking technique is assessed to ascertain if the sample has been spiked correctly. The spiked sample should be 1 – 5 times the concentration of the client sample; otherwise, the percent recovery (%R) or relative percent difference (RPD) of the MS/MSD should be flagged as not meaningful or usable The sample is re-spiked and re-analyzed, along with several other similar samples in subset. If an "out of control" situation persists, sample matrix interference is indicated. Samples to be analyzed by standard additions are prepared (where appropriate), and the group leader, lab supervisor, or QA Department is notified.

13.2.6 Out Of Control Duplicate Samples

Rejection Criteria - Lab-generated maximum RPD limit (as listed under precision in Section 12)

Corrective Action - Instrument and samples checked to see if precision variance is likely (i.e., high suspended solids content, high viscosity, etc.). They are re-analyzed in duplicate and samples just preceding and following the duplicated sample are re-analyzed. If problem still exists, lab supervisor or QA Department is notified to review the analytical techniques.

13.2.7 Out Of Control Matrix Spike Duplicates

These QC samples can be out of control for accuracy, precision, or both. The appropriate corrective actions listed for either matrix spikes, duplicate samples, or both are followed.

Analysis-specific corrective action lists are available for each type of analysis performed by ESC.

13.2.8 Out Of Control Calibration Standards: ICV, CCV, SSCV

Rejection Criteria - If the performance is outside of method requirements.

Corrective Action - Instrument settings are checked, calibration verification standard is reanalyzed. If the standard is still out of control, re-calibration is performed, and samples affected since the last "in control" reference standard are re-analyzed. The group leader, lab supervisor, or QA Department will be consulted for further action.

14.0 RECORD KEEPING

Record keeping is outlined in SOP *#010103 Document Control and Distribution,* SOP *#030203 Reagent Logs and Records* and SOP *#030201 Data Handling and Reporting*

All calibration data and graphs generated for wet chemistry are kept in a calibration notebook with the following information: date prepared, calibration concentrations, correlation, and analyst initials. The analyst reviews the calibration and evaluates it against acceptance criteria before placing it in the calibration notebook. Data on initial and continuing reference standards, as well as matrix spikes and duplicates, are entered in the QC box generated on each analysis page. If a test allows the use of a previously established calibration curve then the calibration check standard is reviewed against acceptance criteria and if acceptable, analysis can proceed. In this situation the calibration date is referenced so that the curve can be easily reviewed, if necessary.

15.0 QUALITY AUDITS

System and data quality audits are outlined in the ESC Quality Assurance Manual *Version 8*.0.

1.0 SIGNATORY APPROVALS

Metals Department
QUALITY ASSURANCE MANUAL

APPENDIX V TO THE ESC
QUALITY ASSURANCE MANUAL

for

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NOTE: The QAM has been approved by the following people. A signed cover page is available upon request

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2.0 APPENDIX TABLE OF CONTENTS

3.0 SCOPE AND APPLICATION

This appendix discusses specific QA requirements for general analytical protocols to ensure that data generated from the Metals Laboratory is scientifically valid and is of acceptable quality. Any deviations from these requirements and any deviations that result in nonconforming work must be immediately evaluated and their corrective actions documented.

4.0 LABORATORY ORGANIZATION AND RESPONSIBILITIES

ESC Lab Sciences offers diverse environmental capabilities that enable the laboratory to provide the client with both routine and specialized services, field sampling and broad laboratory expertise. A brief outline of the organization and responsibilities as they apply to the ESC Quality Assurance Program is presented in *Section 4.0 in the ESC Quality Assurance Manual Version 8.0.*

5.0 PERSONNEL AND TRAINING

5.1 PERSONNEL

James Burns, with a B.S. degree in Medical Technology, is the Department Manager of the Metals Laboratory. Mr. Burns reviews and approves all data reduction associated with metals analysis. Scheduling for analyses and personnel are his responsibility and is paramount to achieving success and quality analyses of samples. His responsibilities also include the coordination with clients' analytical needs regarding regulatory compliance. Mr. Burns has previous experience with numerous regulatory agencies including: USACE, DOD, DOE, NAVY, AFCEE and CLP. Additionally, he has also been involved in waste management/disposal and has held the position of Radiation Safety Officer. In his absence, Lisa Taylor or Lakeia Layne assume responsibility for departmental decisions.

5.2 TRAINING

The primary analyst or Manager trains all new analysts to the laboratory according to ESC protocol. Performance is documented using an initial demonstration of capability (IDOCs) and continuing demonstration of capability (CDOC). On-going acceptable capability in metals analysis and preparation is also demonstrated by acceptable participation in multiple proficiency testing programs (PTs) and daily Quality Control sample analyses. Documentation of analyst training is maintained on file within the department.

6.0 FACILITIES AND LABORATORY SAFETY

6.1 FACILITIES

The main area of the analysis laboratory has approximately1200 square feet with roughly 90 square feet of bench area. The main area of the metals prep laboratory has approximately 1200 square feet with 232 square feet of bench area. The main area of the mercury/TCLP laboratory has approximately 1272 square feet with 136 square feet of bench area. The lighting standard in all three labs is fluorescence. The air system is a 15-ton make-up unit plus 15-ton HVAC with electric heat. The laboratory reagent water is provided through the US Filter deionizer system. Waste disposal containers are located in the laboratory and Clean Harbors serves as ESC's waste disposal company. ESC's building information guides and site plan are shown in Appendix I.

6.2 LABORATORY SAFETY

- Laboratory access is limited when work is being performed.
- All procedures where chemicals are prepared or splashes may occur are conducted in laboratory exhaust hoods.
- ESC's laboratory safety guidelines are detailed in *the ESC Chemical Hygiene and Safety Plan.*

7.0 SAMPLING PROCEDURES

7.1 FIELD SAMPLING PROCEDURES, SAMPLE STORAGE, AND HANDLING

- Field Sampling procedure is described in Appendix III of this ESC Quality Assurance Manual. Sample information is recorded and kept on the ESC chain of custody and field logbooks.
- Matrices for metals analysis are as follows: groundwater, wastewater, drinking water, soil, sludge, paint chips, wipes, filters, and leachates.
- Sample containers, preservation methods and holding times:
  - Glass and plastic containers are acceptable for all elements except Boron and Silicon. Plastic must be used for Boron and Silicon.
  - Water Samples that are analyzed for dissolved metals must be filtered using a 0.45µm pore membrane. Water samples for total metals are not filtered. All water samples are acidified with 1+1 nitric acid to a pH<2. Filtered water samples (dissolved metals) are preserved immediately after filtration. All other water samples are preserved immediately after sampling. Water samples are not refrigerated prior to analysis.
  - Paint chips, dust wipes and filters do not require preservation.
  - Soil samples are stored at 4 ± 2^{o}C and do not require acid preservation.
  - Hold times for all metals, except Mercury, are 180 days. Mercury has a hold time of 28 days.

8.0 EQUIPMENT

Instrument Software

- PE ELAN ICPMS - PE - ICP Winlab - Used for calibration, calculation, QC review, diagnostics, data storage
- Perkin Elmer ICP Optima DV - PE - ICP Winlab - Used for calibration, calculation, qc review, diagnostics, data storage

NOTE: All purchased software that is used in conjunction with software specific instruments is guaranteed by the supplier to function as required. The supplier of the software performs all troubleshooting or software upgrades and revisions.

8.1 EQUIPMENT LIST

LABORATORY EQUIPMENT LIST: MAJOR ITEMS - Metals Analysis and Preparation *This table is subject to revision without notice*						
Item	***Manufacturer***	***Model***	***Name***	***#***	***Serial number***	***Location***
Balance - Top Loading	Mettler Toledo	PB3002-5		1	1119070828	Metals Prep Lab
Balance - Top Loading	Mettler Toledo	PB3002-5		1	71242213216	Mercury Lab
Hot Block	CPI	Mod Block	A	1	NA	Metals Prep Lab
Hot Block	Env. Express	SC154	C	1	3994CEC1880	Metals Prep Lab
ICPMS with autosampler	Perkin Elmer	ELAN DRC-e SC Fast	ICPMS4	1	AH13650804	Metals Lab
ICPMS with autosampler	Perkin Elmer	ELAN DRC-e ASX-510	ICPMS3	1	AH00110504H	Metals Lab
ICPMS with autosampler	Perkin Elmer	ELAN 9000	ICPMS5	1	AJ12270805	Metals Lab
ICPMS with autosampler	Perkin Elmer	ELAN DRC II	ICPMS6	1	AI13820805H	Metals Lab
ICP - Simultaneous with autosampler	Perkin Elmer	Optima 4300DV AS 93 Plus	ICP3	1	077NO110301	Metals Lab
ICP - Simultaneous with autosampler	Perkin Elmer	Optima 4300DV ASX-510	ICP4	1	077N2100201	Metals Lab
ICP - Simultaneous with autosampler	Perkin Elmer	Optima 5300DV ASX-510	ICP5	1	077N5041802	Metals Lab
ICP - Simultaneous with autosampler	Perkin Elmer	Optima 5300DV ASX-510	ICP6	1	077N5091002	Metals Lab
ICP - Simultaneous with autosampler	Perkin Elmer	Optima 5300DV ASX-510	ICP7	1	077C6110602	Metals Lab
Hot Block	CPI	Mod Block	NA	1	3256	Mercury Lab
Hot Block	CPI	Mod Block	NA	1	3356	Mercury Lab
Mercury Auto Analyzer	Perkin Elmer	(2) FIMS 400	I	1	4545	Mercury Lab
Mercury Auto Analyzer	Perkin Elmer	(2) FIMS 400	II	1	401S3060101	Mercury Lab
Mercury Auto Analyzer	Perkin Elmer	(1) FIMS 100	III	1	110156051101	Mercury Lab
Mercury Auto Sampler	Perkin Elmer	(2) AS-91, (1) AS-93	NA	3	NA	Mercury Lab

LABORATORY EQUIPMENT LIST: MAJOR ITEMS - Metals Analysis and Preparation ***This table is subject to revision without notice***						
Item	***Manufacturer***	***Model***	***Name***	***#***	***Serial number***	***Location***
Microwave	CEM	MARS 5	NA	1	DS-9071	Metals Prep Lab
Microwave	CEM	MARS 5	NA	1	DS-8025	Metals Prep Lab
Microwave	CEM	MARS 5	NA	1	DS-8177	Metals Prep Lab
Microwave	CEM	MARS Xpress	NA	1	MD-2861	Metals Prep Lab
Microwave	CEM	MARS Xpress	NA	1	MD-9972	Metals Prep Lab
Microwave	CEM	MARS Xpress	NA	1	MD-9640	Metals Prep Lab
Prep Station	Env. Express	Automated prep station	Autoblock 1	1	1243	Metals Prep Lab
Prep Station	Env. Express	Automated prep station	Autoblock 2	2	1783	Metals Prep Lab
TCLP Extraction Unit	Env. Express	6 Position	NA	1	NA	TCLP Lab
TCLP Extraction Unit	Env. Express	12 Position	NA	5	4809-12-542	TCLP Lab
TCLP Extraction Unit	Env. Express	12 Position	NA	5	1918-12-415	TCLP Lab
TCLP Extraction Unit	Env. Express	12 Position	NA	5	1918-12-414	TCLP Lab
TCLP Extraction Unit	Env. Express	12 Position	NA	5	5152-12-548	TCLP Lab
TCLP Extraction Unit	Env. Express	12 Position	NA	5	NA	TCLP Lab
TCLP Extraction Unit	Env. Express	10 Position	NA	1	NA	TCLP Lab
TCLP Extraction Unit	Env. Express	Teflon Vessels	NA	12	NA	TCLP Lab
TCLP Zero Headspace Extractor	Env. Express	Vessels	NA	20	NA	TCLP Lab
Turbidimeter	HACH	2100N		1	05090C020685	Metals Prep Lab
Water Purification - Nanopure	Barnstead	D11911		1	1372051120948	Metals Prep Lab
PH Meter	Orion	410A	NA	1	015683	TCLP Lab
Auto Block	Env. Express		NA	1	1783	Metals Prep Lab
Auto pipetters 1000µl to 20 µl	Oxford	Varies	NA		NA	Metals Lab
Auto pipetters	Eppendorf, Oxford	Varies	NA		NA	Metals Prep Lab
MAX/MIN Thermometer	VWR	MAX/MIN	TCLP #1		NA	TCLP Lab
MAX/MIN Thermometer		MAX/MIN	TCLP #2		NA	TCLP Lab

8.2 EQUIPMENT PREVENTIVE MAINTENANCE, EQUIPMENT CALIBRATION

INSTRUMENT	P. M. DESCRIPTION	FREQUENCY
ICP	•Maintain manufacturer's service contract	Renew annually
ICP and ICPMS	•Pump tubing, torch alignment, o-ring, injector tip and torch	Check daily and adjust/change as needed
ICPMS	•Sampler and Skimmer cones	Clean or replace when needed
ICP and ICPMS	•Pump rollers	Clean and lubricate when needed

INSTRUMENT	P. M. DESCRIPTION	FREQUENCY
ICP and ICPMS	•Nebulizer	As needed
Mercury Analyzer	•Calibrate and check sensitivity with previous data	Daily with use
Mercury Analyzer	•Response factor problems, check tubing for leaks, particularly in pump head, and check cell for fogging	As needed
Mercury Analyzer	•Replace desiccant in tube	With each use
Mercury Analyzer	•Check rotometer for airflow, if inadequate, replace flex tubing in pump lead	As needed
TCLP Apparatus (ZHE)	•Change O-rings	As needed
Thermometer	•All working thermometers are compared to a NIST thermometer.	Semi-annually
pH Meter	•Calibrated according to manufacturers instructions. •The slope is documented and acceptable range 95-105%	Daily
Analytical Balance	•Analytical balances are checked and calibrated by a certified technician semi-annually. •Calibration is checked daily with class S weights. Must be within 0.1% S class weights calibrated annually	Semi-annually Daily
TCLP Tumblers	•Visually timed and confirmed to be 30±2 rpm.	Monthly
Microwaves	•Checked and calibrated by a certified technician	Semi-annually, calibrated weekly by staff
Microwaves	Check cap membranes for leaks	As needed

8.3 STANDARDS AND REAGENTS

All reagents and standards must meet the requirements listed in the analytical methods.

Table 8.3A: Stock Standard sources, receipt, and preparation information.
(subject to revision as needed)

STOCK STANDARD SOURCES
****ICP metals used – Ag, Al, As, B, Ba, Be, Ca, Cd, Co, Cr, Cu, Fe, K, Li, Mg, Mn, Mo, Na, Ni, Pb, Sb, Se, Si, Sn, Sr, Ti, Tl, V, Zn (Sulfur is analyzed individually)***
****ICP/MS metals used – Ag, As, Ba, Be, Cd, Co, Cr, Cu, Mn, Mo, Ni, Pb, Sb, Se, Sn, Tl, V, Zn***

Instrument Group/Standard	*Standard Source**	*How Received**	*Source/ Storage*	*Lab Stock Storage*	*Receipt Frequency*
ICP (single element standards)	Env. Express or High Purity	1000ppm	Room temp.		Annual/Expiration Date
ICP/ICV	High Purity	500ppm – Al. Ca, Fe, Mg, Na, K 5ppm – Ag 50ppm – All others	Room temp.	5% HNO3 w/ Tr HF	As needed
ICP/Calibration Standard and CCV	Env. Express	1000ppm – Al, Ca, Fe, K, Mg, Na 10ppm – Ag 100ppm – All others	Room temp.	5% HNO3 w/ Tr HF	As needed
ICP/LCS water	Ultra Scientific	1000ppm – Ca, Mg, K, Na 100ppm – all others except Li (spiked separately)	Room temp.	5% HNO3	As needed

Table 8.3A: Stock Standard sources, receipt, and preparation information.
(subject to revision as needed)

STOCK STANDARD SOURCES
****ICP metals used – Ag, Al, As, B, Ba, Be, Ca, Cd, Co, Cr, Cu, Fe, K, Li, Mg, Mn, Mo, Na, Ni, Pb, Sb, Se, Si, Sn, Sr, Ti, Tl, V, Zn (Sulfur is analyzed individually)***
****ICP/MS metals used – Ag, As, Ba, Be, Cd, Co, Cr, Cu, Mn, Mo, Ni, Pb, Sb, Se, Sn, Tl, V, Zn***

Instrument Group/Standard	*Standard Source**	*How Received**	*Source/ Storage*	*Lab Stock Storage*	*Receipt Frequency*
ICP/LCS soil	ERA	Varies with Lot #	Room temp.	none	As needed
ICP/ICSA	Env. Express	5000ppm – Al, Ca, Mg, Na 2000ppm – Fe 100ppm – K	Room temp.	10% HNO3	As needed
ICP/ICSB	Env. Express	100ppm – B, Cd, Pb, Ag, Ni, Si, Zn, 50ppm – all others except Sr, Li	Room temp.	4% HNO3 w/ Tr HF	As needed
ICP/Yttrium	Env. Express	10,000 ppm	Room temp.	4% HNO3	As needed
ICPMS/ICV	High Purity	5 ppm	Room temp.	5% HNO3 w/ Tr HF	As needed
ICPMS/ Calibration Standard and CCV	Env. Express	100 ppm	Room temp.	5% HNO3 w/ Tr HF	As needed
ICPMS/LCS water	Ultra Scientific	1000ppm – Ca, Mg, K, Na 100ppm – all others except Li (spiked separately)	Room temp.	5% HNO3	As needed
ICPMS/LCS soil	ERA	Varies with Lot #	Room temp.	none	As needed
ICPMS/ICSA	Env. Express	10000ppm – Cl 2000ppm – C 1000ppm – Al, Ca, Fe, Mg, P, K, Na, S 20ppm – Mo, Ti	Room temp.	10% HNO3	As needed
ICP/ICSB	Env. Express	2ppm – Sb, As, Be, Ca, Cr, Co, Cu, Pb, Ni, Se, Ag, Tl, Sn, Zn	Room temp.	4% HNO3 w/ Tr HF	As needed
Hg/ICV and LCS	Inorganic Ventures	1000ppm – Hg	Room temp.	2% HNO3	As needed
Hg/Calibration Standard and CCV	Env. Express	1000ppm – Hg	Room temp.	2% HNO3	As needed

**Equivalent Providers may be utilized.*

Table 8.3B: Working standard concentration, storage and preparation information.
(subject to revision as needed)

WORKING STANDARD PREPARATION
****ICP metals used – Ag, Al, As, B, Ba, Be, Ca, Cd, Co, Cr, Cu, Fe, K, Li, Mg, Mn, Mo, Na, Ni, Pb, Sb, Se, Si, Sn, Sr, Ti, Tl, V, Zn (Sulfur is analyzed individually)***
****ICP/MS metals used – Ag, As, Ba, Be, Cd, Co, Cr, Cu, Mn, Mo, Ni, Pb, Sb, Se, Sn, Tl, V, Zn***

Instrument Group/Standard	*How Prepared*	*Final Concentration*	*Source/ Storage*	*Expiration*
ICP/ICV	10mL Custom Stock ICV A and B, 0.1 mL stock Sc adjusted to 100mL with 5% HNO3	50ppm – Al, Ca, Fe, K, Mg, Na 0.5ppm – Ag 2ppm - Sr 5ppm – All others	Room temp.	1 month

Table 8.3B: Working standard concentration, storage and preparation information.
(subject to revision as needed)

WORKING STANDARD PREPARATION
****ICP metals used – Ag, Al, As, B, Ba, Be, Ca, Cd, Co, Cr, Cu, Fe, K, Li, Mg, Mn, Mo, Na, Ni, Pb, Sb, Se, Si, Sn, Sr, Ti, Tl, V, Zn (Sulfur is analyzed individually)***
****ICP/MS metals used – Ag, As, Ba, Be, Cd, Co, Cr, Cu, Mn, Mo, Ni, Pb, Sb, Se, Sn, Tl, V, Zn***

Instrument Group/Standard	*How Prepared*	*Final Concentration*	*Source/ Storage*	*Expiration*
ICP/Calibration Standard	Std 6 – 10mL Stock Cal. Std. Std 5 – 1mL Stock Cal. Std. Std 4 – 1mL Std. 6 Std 3 – 1mL Std. 5 Std 2 – 0.5mL Std. 5 Std 1 – 2mL Std. 4 All adjusted to 100 mL with 5%HNO3	Std 6 – 1/10/1000ppm Std 5 – 0.1/1/10ppm Std 4 – 0.01/0.1/1ppm Std 3 – 0.01/0.1ppm Std 2 – 0.005ppm Std 1 – 0.002ppm	Room temp.	1 month
ICP/CCV	50mL Custom Stock CCV adjusted to 1000mL with 5% HNO3	50ppm – Al, Ca, Fe, K, Mg, Na 0.5ppm – Ag 5ppm – All others	Room temp.	1 month
ICP/ICSA	100mL Custom Stock ICSA adjusted to 1000mL with 5% HNO3	500ppm – Al, Ca, Mg, Na 200ppm – Fe 10ppm – K	Room temp.	1 month
ICP/ICSAB	100mL Custom Stock ICSA, 10mL Stock ICSAB adjusted to 1000mL with 5% HNO3	500ppm – Al, Ca, Mg, Na 200ppm – Fe 10ppm – K 1ppm – B, Cd, Pb, Ag, Ni, Si, Zn, 0.5ppm – all others except Sr, Li	Room temp.	1 month
ICP/Yttrium	5mL Stock Yttrium adjusted to 10L with 5% HNO3	5 ppm	Room temp.	1 month
ICPMS/ICV	1.0mL Stock ICV adjusted to 100mL with 5% HNO3	0.05 ppm	Room temp.	1 month
ICPMS/ Calibration Standard	0.1 Stock Cal Std adjusted to 100mL with 5%HNO3. Serial Dilutions are done each calibration from 0.1ppm Std.	Cal 5 – 0.1ppm Cal 4 – 0.05ppm Cal 3 – 0.01ppm Cal 2 – 0.001ppm Cal 1 – 0.0005ppm	Room temp.	1 month
ICPMS/CCV	0.05mL Stock CCV adjusted to 100mL with 5% HNO3.	0.050 ppm	Room temp.	1 month
ICPMS/ICSA	10mL Stock ICSA adjusted to 100mL with 5% HNO3	1000ppm – Cl 200ppm – C 100ppm – Al, Ca, Fe, Mg, P, K, Na, S 2ppm – Mo, Ti	Room temp.	1 month
ICPMS/ICSAB	10mL Stock ICSA, 1mL Stock ICSAB adjusted to 100mL with 5% HNO3	1000ppm – Cl 200ppm – C 100ppm – Al, Ca, Fe, Mg, P, K, Na, S 2ppm – Mo, Ti 0.02ppm – Sb, As, Be, Ca, Cr, Co, Cu, Pb, Ni, Se, Ag, Tl, Sn, Zn	Room temp.	1 Month
Hg/ICV	30µL of 3ppm Intermediate	0.003ppm – Hg	Room temp.	1 Month

Table 8.3B: Working standard concentration, storage and preparation information.
(subject to revision as needed)

WORKING STANDARD PREPARATION
****ICP metals used – Ag, Al, As, B, Ba, Be, Ca, Cd, Co, Cr, Cu, Fe, K, Li, Mg, Mn, Mo, Na, Ni, Pb, Sb, Se, Si, Sn, Sr, Ti, Tl, V, Zn (Sulfur is analyzed individually)***
****ICP/MS metals used – Ag, As, Ba, Be, Cd, Co, Cr, Cu, Mn, Mo, Ni, Pb, Sb, Se, Sn, Tl, V, Zn***

Instrument Group/Standard	*How Prepared*	*Final Concentration*	*Source/ Storage*	*Expiration*
Hg/Calibration Standard	Std 6 - 100µL of 3ppm Intermediate Std 5 - 50µL of 3ppm Intermediate Std 4 - 200µL of 300ppb Intermediate Std 3 - 100µL of 300ppb Intermediate Std 2 - 40µL of 300ppb Intermediate Std 1 - 20µL of 300ppb Intermediate	Std 6 – 0.01ppm Std 5 – 0.005ppm Std 4 – 0.002ppm Std 3 – 0.001ppm Std 2 – 0.0004ppm Std 1 – 0.0002ppm	Room temp.	4 days
Hg/CCV	2.5ppb CCV - 25µL of 3ppm Intermediate	0.0025ppm	Room temp.	1 Month
Hg/LCS	30µL of 3ppm Intermediate	0.003ppm – Hg	Room temp.	1 Month

8.4 INSTRUMENT CALIBRATION

Mercury Analyzer - SOP Numbers 340384A & 340384B

Calibration of the mercury analyzer is achieved using 5 standards. Acceptable calibration is achieved when the correlation coefficient $\geq$0.998. All results are calculated using software based on the peak area of the sample. A second source ICV is analyzed initially and must recover within $\pm$10% for Methods 7470A/7471A/7471B and within $\pm$5% for method 245.1. A primary source CCV is analyzed after every tenth sample and at the conclusion of the analytical sequence. The CCV must recovery within $\pm$10% for all analyses. Duplicate and spike analyses are performed on 5% of the samples analyzed using EPA Method 7470A/7471A/7471B and on 10% of the samples analyzed using EPA Method 245.1.

Inductively Coupled Plasma - SOP Numbers 340386 & 340390

The PE ICP Optima 4300DV, 5300DV and PE ELAN 6100 and DRC-e ICPMS are calibrated using at least 3 standards. A new calibration curve is analyzed daily. All calculations are performed by software using computerized linear regression. The linear regression correlation coefficient for the each analyte in the calibration curve lines must be 0.998 or better for all methods, except methods 6010C and 6020A that must be 0.998 or better. A second source ICV is run initially and a primary source CCV is run after every tenth sample. For method 200.7, the ICV must recover within 5% of the true value and for all other methods, the ICV must recover within 10%. The CCV for all methods must recover within 10% of the true value. Duplicate and spike analyses are performed on 5% of the samples for EPA Methods 6010B, 6010C, 6020, 6020A and on 10% of the samples analyzed using EPA Methods 200.7 & 200.8.

TABLE 8.4: CALIBRATION STANDARD CONCENTRATIONS
This table is subject to revision without notice

HIGH LEVEL	ICP (mg/L)	ICP/MS (mg/L)
Aluminum	0.10 - 100	----------
Antimony	0.01 - 10	0.0005 – 0.05
Arsenic	0.01 - 10	0.0005 – 0.10
Barium	0.005 - 10	0.0005 – 0.10
Beryllium	0.002 – 10	0.0005 – 0.01
Boron	0.10 – 10	----------
Cadmium	0.005 - 10	0.0005 – 0.10
Calcium	0.10 - 100	----------
Chromium	0.01 - 10	0.0005 – 0.10
Cobalt	0.01 - 10	0.0005 – 0.10
Copper	0.01 - 10	0.0005 – 0.10
Iron	0.10- 100	----------
Lead	0.005 - 10	0.0005 – 0.10
Lithium	0.005 - 10	----------
Magnesium	0.10 - 100	----------
Manganese	0.010 - 10	0.0005 – 0.10
Molybdenum	0.002 - 10	0.0005 – 0.10
Nickel	0.01 - 10	0.0005 – 0.10
Potassium	0.50 - 100	----------
Selenium	0.01 - 10	0.0005 – 0.10
Silicon	0.10 - 10	----------
Silver	0.01 – 1.0	0.0005 – 0.05
Sodium	0.50 - 100	----------
Strontium	0.002 – 10	----------
Sulfur	10 - 100	----------
Thallium	0.01 - 10	0.0005 – 0.05
Tin	0.01 - 10	0.0005 – 0.10

TABLE 8.4: CALIBRATION STANDARD CONCENTRATIONS *This table is subject to revision without notice*		
HIGH LEVEL	ICP (mg/L)	ICP/MS (mg/L)
Titanium	0.01 – 10	----------
Vanadium	0.01 - 10	0.0005 – 0.10
Zinc	0.010 - 10	0.001 – 0.10
MERCURY		
Mercury	Blank, 0.2 - 0.010 µg/L	

8.5 ACCEPTANCE/REJECTION OF CALIBRATION

The initial calibration curve is compared with previous curves for the same analyte. All new standard curves are immediately checked with a secondary source or laboratory control standard prepared from a separate source than those used for calibration. All curves are visually reviewed to ensure that acceptable correlation represents linearity. Calibration curves may be rejected for nonlinearity, abnormal sensitivity, or poor response of the laboratory control standard. Specific criteria for each instrument are outlined in Table 8.5.

Continuing calibration verification is performed on each day that initial calibration is not performed and following every tenth sample. If a check standard does not perform within established criteria then the instrument is evaluated to determine the problem. Once the problem is corrected, all samples between the last in control sample and the first out of control check are re-analyzed.

TABLE 8.5 INSTRUMENT CALIBRATION & QC

Instrument (Analysis)	Calibration Type	Number of Standards	Acceptance/ Rejection Criteria	Frequency
ICP & ICPMS	Linear/ Initial	3 - 5	6010B, 6020, 200.7 200.8: Must have a correlation coefficient of at least 0.998 6010C, 6020A: Must have a correlation coefficient of at least 0.998	Daily
ICP & ICPMS	Initial	Secondary source (ICV)	6010B, 6010C, 6020, 6020A, 200.8: ICV must be within +/-10%; 200.7: ICV must be within +/-5%	After initial calibration
ICP & ICPMS	Initial	1 Initial Calibration Blank	< ½ RL, concentrations of common laboratory contaminants shall not exceed the RL	After initial calibration
ICP, ICPMS, Mercury	Continuing	1 mid-level ref. std. (CCV)	Must be within ±10%	Every 10th sample
ICP & ICPMS	Continuing	1 Continuing Calibration Blank	< ½ RL, concentrations of common laboratory contaminants must not exceed the RL	Every 10th sample
ICP & ICPMS	Continuing	1 ICSA 1 ICSAB	Must be within ±20% for ICP, No criteria for ICPMS	After initial calibration, at end and every 8 hours of run time.
ICP, ICPMS, Mercury	Continuing	1 Method Blank	< ½ RL, concentrations of common laboratory contaminants must not exceed the RL	1 per batch
ICP, ICPMS, Mercury	Continuing	1 Laboratory Control Standard	Liquid Samples (all methods) - LCS must be within ±15%. Solid Samples (all methods) - LCS must be within the certified standard value determined by the provider.	1 per batch
ICP, ICPMS, Mercury	Continuing	1 Sample Duplicate	Sample and Duplicate must have an RPD ≤20%	1 per batch
ICP & ICPMS	Continuing	1 Matrix Spike (MS), 1 Matrix Spike Duplicate (MSD)	Spike must be within ±25%, MS and MSD must have an RPD ≤20%	1 of each per batch
Mercury	Linear/ Initial	3 - 5	Must have a correlation coefficient of at least 0.998	Daily
Mercury	Initial	Secondary source (ICV)	7470A, 7471: ICV must be within ±10% 245.1: ICV must be within ±5%	After initial calibration
Mercury	Initial	1 Initial Calibration Blank	< ½ RL	After initial calibration
Mercury	Continuing	1 Continuing Calibration Blank	< ½ RL	Every 10th sample
Mercury	Continuing	1 Matrix Spike (MS), 1 Matrix Spike Duplicate (MSD)	Spike must be within ±30%, MS and MSD must have an RPD ≤20%	1 of each per batch

9.0 LABORATORY PRACTICES

9.1 REAGENT GRADE WATER

ASTM Type I grade water.

9.2 GLASSWARE WASHING AND STERILIZATION PROCEDURES

Glassware involved in metals preparation is washed with soap and water, rinsed in 1+1 nitric acid, and rinsed in DI water. Through digestion blanks, it has been determined that chromic acid washing is unnecessary. Glassware with visible gummy deposits remaining after washing is disposed of properly. All metals glassware is given another DI water rinse immediately prior to use. Metals glassware is segregated from all other glassware.

10.0 ANALYTICAL PROCEDURES

10.1 A list of laboratory SOP's associated with the metals laboratory can be found in the following table.

TABLE 10.1: METALS DEPARTMENT SOP'S

This table is subject to revision without notice

SOP #	Title
TCLP SOP's	
340358	TCLP
340362	SPLP
340363	EP TOX
340364	MEP
340705	California Waste Extraction Test
Mercury SOP's	
340384A	Mercury in Liquid Waste (Cold-Vapor Technique) 7470A/245.1
340384B	Mercury in Solid Waste (Cold-Vapor Technique) 7471A
Metals Prep SOP's	
340389	Acid Digestion of Aqueous Samples and Extracts Method 3005A/3010A/3015/3030C
340380	Digestion of Metals and Trace Elements in DW and Wastes Method 200.2
340388	Acid Digestion of Sediments, Sludge, Soils and Oils Method 3050B/3051
340701	Metals Digestion of personal cassettes Method 7300, 3051
340702	Metals Digestion for Sediments, Soils, and Sludge NIOSH 7300, Method 3051 for ELLAP Paint chips and ELLAP soils
340703	Metals Digestion of Hi-Vol filters and Environmental Lead Wipes 3050B and 3051
340391	Silver (Photographic Waste) Method 7760 and 272.1
340392	Sodium Adsorption Ratio
Metals Analysis SOP's	
340386	Metals by ICP Method 6010, 200.7
340390	Metals by ICP-MS Method 6020, 200.8

11.0 QUALITY CONTROL CHECKS

NOTE: For specific guidance on each determinative method, including required quality control and specific state requirements/modifications, refer to the relevant laboratory standard operating procedure(s).

11.1 ESC participates in proficiency testing (PT's) in support of various laboratory accreditations/recognitions. Environmental samples are purchased from Environmental Resource Associates (ERA). The WS, WP and solid matrix studies are completed every 6 months. For industrial hygiene and environmental lead accreditation, PTs are administered by AIHA. IHPAT samples for metals analysis, including lead in air, by NIOSH 7300 is completed every quarter. Soil, wipes and paint PTs are also completed in conjunction with the AIHA Environmental Lead Laboratory Accreditation Program (ELLAP). AIHA PT samples are received and analyzed by method according to the vendor's instructions and according to ESC SOP.

11.2 Initial Demonstrations of Capability (IDOC's) are performed during new analyst training and/or prior to acceptance and use of any new method/instrumentation. Continuing Demonstration of Capability (CDOC's) must be updated at least annually. The associated data is filed within the department and available for review.

11.3 Sample Duplicates, Matrix Spike and Matrix Spike Duplicates are performed on 5–10% of samples analyzed depending on analytical method requested. For methods 6010, 6020, 7470A and 7471A duplicates, matrix spikes and matrix spike duplicates are performed on 5% of samples. For methods 200.7, 200.8 and 245.1, the same QC is performed on 10% of samples. The RPD must not exceed 20%.

11.4 A laboratory control sample (LCS) is analyzed one per batch of samples. The acceptance criteria for all water samples is ±15%. See certificate of analysis for soil true values. For Industrial Hygiene samples, the LCS is analyzed in duplicate per batch.

11.5 A method preparation blank is performed per batch of samples processed. If one-half the reporting limit [RL] is exceeded, the laboratory evaluates whether reprocessing of the samples is necessary, based on the following criteria:

- The blank contamination exceeds a concentration greater than 1/10 of the measured concentration of any sample in the associated preparation batch or
- The blank contamination is greater than 1/10 of the specified regulatory limit.

The concentrations of common laboratory contaminants must not exceed the reporting limit. Any samples associated with a blank that fail these criteria is re-processed in a subsequent preparation batch, except when the sample analysis resulted in non-detected results for the failing analytes.

12.0 DATA REDUCTION, VALIDATION, AND REPORTING

12.1 DATA REDUCTION

The analyst performs the data calculation and is responsible for the initial examination of the finished data. Data reduction steps applied to the raw data are outlined in ESC SOP #030201, *Data Handling and Reporting.* The Quality Control Department performs the secondary review of the data package using the ESC SOP #030227, *Data Review*. The QC Reviewer verifies that the analysis has performed as required and meets method criteria, all associate data is present and complete, and also ensures that any additional documentation is completed as required (i.e. Ohio VAP checklists, required flags on test reports, etc.)

12.2 VALIDATION

The validation process consists of data generation, reduction review, and reporting results. Once data reduction is complete, validation is conducted by verification that the QC samples are within acceptable QC limits and that all documentation is complete, including the analytical report and associated QC. See Table 12.1 for current QC targets and controls and current reporting limits.

12.3 REPORTING

Reporting procedures are documented in SOP #030201, *Data Handling and Reporting.*

Table 12.3A: QC Targets for Environmental Metals Accuracy (LCS), Precision and RL's
(subject to revision without notice)

Class	Analyte	Prep Method	Analysis Method	Matrix	Accuracy Range (%)	Precision (RPD)	RL (ppb)
(ICP-AES)	Aluminum	3050B (mod.)	6010B/C	Solid	Certified Standard Values	<20	5000
(ICP-AES)	Aluminum	3051 (mod.)	6010B/C	Solid	Certified Standard Values	<20	5000
(ICP-AES)	Aluminum	3015/3010 (mod.)	6010B/C	Liquid/Aqueous	85 - 115	<20	100
(ICP-AES)	Aluminum	200.2 (mod.), NPDES	200.7	Liquid/Aqueous	85 - 115	<20	100
(ICP-MS)	Antimony	3051 (mod.)	6020/A (mod.)	Solid	Certified Standard Values	<20	50
(ICP-AES)	Antimony	3050B (mod.)	6010B/C	Solid	Certified Standard Values	<20	1000
(ICP-AES)	Antimony	3051 (mod.)	6010B/C	Solid	Certified Standard Values	<20	1000
(ICP-MS)	Antimony	200.2 (mod.), NPDES	200.8	Liquid/Aqueous	85 - 115	<20	1
(ICP-MS)	Antimony	3015/3010 (mod.)	6020/A (mod.)	Liquid/Aqueous	85 - 115	<20	1
(ICP-AES)	Antimony	3015/3010 (mod.)	6010B/C	Liquid/Aqueous	85 - 115	<20	20
(ICP-AES)	Antimony	200.2 (mod.), NPDES	200.7	Liquid/Aqueous	85 - 115	<20	20

Table 12.3A: QC Targets for Environmental Metals Accuracy (LCS), Precision and RL's

(subject to revision without notice)

Class	Analyte	Prep Method	Analysis Method	Matrix	Accuracy Range (%)	Precision (RPD)	RL (ppb)
(ICP-MS)	Arsenic	3051 (mod.)	6020/A (mod.)	Solid	Certified Standard Values	<20	50
(ICP-AES)	Arsenic	3050B (mod.)	6010B/C	Solid	Certified Standard Values	<20	1000
(ICP-AES)	Arsenic	3051 (mod.)	6010B/C	Solid	Certified Standard Values	<20	1000
(ICP-AES)	Arsenic	1311, 1312	6010B/C	Leachate	85 - 115	<20	50
(ICP-MS)	Arsenic	200.2 (mod.), NPDES	200.8	Liquid/Aqueous	85 - 115	<20	1
(ICP-MS)	Arsenic	3015/3010 (mod.)	6020/A (mod.)	Liquid/Aqueous	85 - 115	<20	1
(ICP-AES)	Arsenic	3015/3010 (mod.)	6010B/C	Liquid/Aqueous	85 - 115	<20	20
(ICP-AES)	Arsenic	NPDES	200.7	Liquid/Aqueous	85 - 115	<20	20
(ICP-MS)	Barium	3051 (mod.)	6020/A (mod.)	Solid	Certified Standard Values	<20	100
(ICP-AES)	Barium	3050B (mod.)	6010B/C	Solid	Certified Standard Values	<20	250
(ICP-AES)	Barium	3051 (mod.)	6010B/C	Solid	Certified Standard Values	<20	250
(ICP-AES)	Barium	1311-12	6010B/C	Leachate	85 - 115	<20	50
(ICP-AES)	Barium	3015/3010 (mod.)	6010B/C	Liquid/Aqueous	85 - 115	<20	5
(ICP-MS)	Barium	3015/3010 (mod.)	6020/A (mod.)	Liquid/Aqueous	85 - 115	<20	2
(ICP-MS)	Barium	200.2 (mod.), NPDES	200.8	Liquid/Aqueous	85 - 115	<20	2
(ICP-AES)	Barium	200.2 (mod.), NPDES	200.7	Liquid/Aqueous	85 - 115	<20	5
(ICP-MS)	Beryllium	3051 (mod.)	6020/A (mod.)	Solid	Certified Standard Values	<20	50
(ICP-MS)	Beryllium	200.2 (mod.), NPDES	200.8	Liquid/Aqueous	85 - 115	<20	1
(ICP-MS)	Beryllium	3015/3010 (mod.)	6020/A (mod.)	Liquid/Aqueous	85 - 115	<20	1
(ICP-AES)	Beryllium	3050B (mod.)	6010B/C	Solid	Certified Standard Values	<20	100
(ICP-AES)	Beryllium	3051 (mod.)	6010B/C	Solid	Certified Standard Values	<20	100
(ICP-AES)	Beryllium	3015/3010 (mod.)	6010B/C	Liquid/Aqueous	85 - 115	<20	2
(ICP-AES)	Beryllium	200.2 (mod.), NPDES	200.7	Liquid/Aqueous	85 - 115	<20	2
(ICP-AES)	Boron	3050B (mod.)	6010B/C	Solid	Certified Standard Values	<20	10000
(ICP-AES)	Boron	3051 (mod.)	6010B/C	Solid	Certified Standard Values	<20	10000
(ICP-AES)	Boron	3015/3010 (mod.)	6010B/C	Liquid/Aqueous	85 - 115	<20	200
(ICP-AES	Boron	200.2 (mod.), NPDES	200.7	Liquid/Aqueous	85 - 115	<20	200
(ICP-MS)	Cadmium	3051 (mod.)	6020/A (mod.)	Solid	Certified Standard Values	<20	25
(ICP-AES)	Cadmium	3050B (mod.)	6010B/C	Solid	Certified Standard Values	<20	250
(ICP-AES)	Cadmium	3051 (mod.)	6010B/C	Solid	Certified Standard Values	<20	250
(ICP-AES)	Cadmium	1311-1312	6010B/C	Leachate	85 - 115	<20	50

Table 12.3A: QC Targets for Environmental Metals Accuracy (LCS), Precision and RL's

(subject to revision without notice)

Class	Analyte	Prep Method	Analysis Method	Matrix	Accuracy Range (%)	Precision (RPD)	RL (ppb)
(ICP-MS)	Cadmium	200.2 (mod.), NPDES	200.8	Liquid/Aqueous	85 - 115	<20	0.5
(ICP-MS)	Cadmium	3015/3010 (mod.)	6020/A (mod.)	Liquid/Aqueous	85 - 115	<20	0.5
(ICP-AES)	Cadmium	3015/3010 (mod.)	6010B/C	Liquid/Aqueous	85 - 115	<20	5
(ICP-AES)	Cadmium	200.2 (mod.), NPDES	200.7	Liquid/Aqueous	85 - 115	<20	5
(ICP-AES)	Calcium	3050B (mod.)	6010B/C	Solid	Certified Standard Values	<20	25000
(ICP-AES)	Calcium	3051 (mod.)	6010B/C	Solid	Certified Standard Values	<20	25000
(ICP-AES)	Calcium	3015/3010 (mod.)	6010B/C	Liquid/Aqueous	85 - 115	<20	500
(ICP-AES)	Calcium	200.2 (mod.), NPDES	200.7	Liquid/Aqueous	85 - 115	<20	500
(ICP-MS)	Chromium	3051 (mod.)	6020/A (mod.)	Solid	Certified Standard Values	<20	50
(ICP-AES)	Chromium	3050B (mod.)	6010B/C	Solid	Certified Standard Values	<20	500
(ICP-AES)	Chromium	3051 (mod.)	6010B/C	Solid	Certified Standard Values	<20	500
(ICP-AES)	Chromium	1311-12	6010B/C	Leachate	85 - 115	<20	50
(ICP-MS)	Chromium	200.2 (mod.), NPDES	200.8	Liquid/Aqueous	85 - 115	<20	1
(ICP-MS)	Chromium	3015/3010 (mod.)	6020/A (mod.)	Liquid/Aqueous	85 - 115	<20	1
(ICP-AES)	Chromium	3015/3010 (mod.)	6010B/C	Liquid/Aqueous	85 - 115	<20	10
(ICP-AES)	Chromium	200.2 (mod.), NPDES	200.7	Liquid/Aqueous	85 - 115	<20	10
(ICP-MS)	Cobalt	3051 (mod.)	6020/A (mod.)	Solid	Certified Standard Values	<20	50
(ICP-AES)	Cobalt	3050B (mod.)	6010B/C	Solid	Certified Standard Values	<20	500
(ICP-AES)	Cobalt	3051 (mod.)	6010B/C	Solid	Certified Standard Values	<20	500
(ICP-MS)	Cobalt	200.2 (mod.), NPDES	200.8	Liquid/Aqueous	85 - 115	<20	1
(ICP-MS)	Cobalt	3015/3010 (mod.)	6020/A (mod.)	Liquid/Aqueous	85 - 115	<20	1
(ICP-AES)	Cobalt	3015/3010 (mod.)	6010B/C	Liquid/Aqueous	85 - 115	<20	10
(ICP-AES)	Cobalt	200.2 (mod.), NPDES	200.7	Liquid/Aqueous	85 - 115	<20	10
(ICP-MS)	Copper	3051 (mod.)	6020/A (mod.)	Solid	Certified Standard Values	<20	50
(ICP-AES)	Copper	3050B (mod.)	6010B/C	Solid	Certified Standard Values	<20	1000
(ICP-AES)	Copper	3051 (mod.)	6010B/C	Solid	Certified Standard Values	<20	1000
(ICP-AES)	Copper	1311-12	6010B/C	Leachate	85 - 115	<20	50
(ICP-MS)	Copper	3015/3010 (mod.)	6020/A (mod.)	Liquid/Aqueous	85 - 115	<20	1
(ICP-MS)	Copper	200.2 (mod.), NPDES	200.8	Liquid/Aqueous	85 - 115	<20	1
(ICP-AES)	Copper	3015/3010 (mod.)	6010B/C	Liquid/Aqueous	85 - 115	<20	20

Table 12.3A: QC Targets for Environmental Metals Accuracy (LCS), Precision and RL's

(subject to revision without notice)

Class	Analyte	Prep Method	Analysis Method	Matrix	Accuracy Range (%)	Precision (RPD)	RL (ppb)
(ICP-AES)	Copper	200.2 (mod.), NPDES	200.7	Liquid/Aqueous	85 - 115	<20	20
(ICP-AES)	Iron	3050B (mod.)	6010B/C	Solid	Certified Standard Values	<20	5000
(ICP-AES)	Iron	3051 (mod.)	6010B/C	Solid	Certified Standard Values	<20	5000
(ICP-AES)	Iron	3015/3010 (mod.)	6010B/C	Liquid/Aqueous	85 - 115	<20	100
(ICP-AES)	Iron	200.2 (mod.), NPDES	200.7	Liquid/Aqueous	85 - 115	<20	100
(ICP-MS)	Lead	3051 (mod.)	6020/A (mod.)	Solid	Certified Standard Values	<20	50
(ICP-AES)	Lead	3050B (mod.)	6010B/C	Solid	Certified Standard Values	<20	250
(ICP-AES)	Lead	3051 (mod.)	6010B/C	Solid	Certified Standard Values	<20	250
(ICP-AES)	Lead	1311-12	6010B/C	Leachate	85 - 115	<20	50
(ICP-MS)	Lead	3015/3010 (mod.)	6020/A (mod.)	Liquid/Aqueous	85 - 115	<20	1
(ICP-MS)	Lead	200.2 (mod.)	200.8	Liquid/Aqueous	85 - 115	<20	1
(ICP-AES)	Lead	3015/3010 (mod.)	6010B/C	Liquid/Aqueous	85 - 115	<20	5
(ICP-AES)	Lead	NPDES	200.7	Liquid/Aqueous	85 - 115	<20	5
(ICP-AES)	Lithium	3050B (mod.)	6010B/C	Solid	Certified Standard Values	<20	750
(ICP-AES)	Lithium	3051 (mod.)	6010B/C	Solid	Certified Standard Values	<20	750
(ICP-AES)	Lithium	3015/3010 (mod.)	6010B/C	Liquid/Aqueous	85 - 115	<20	15
(ICP-AES)	Lithium	200.2 (mod.), NPDES	200.7	Liquid/Aqueous	85 - 115	<20	15
(ICP-AES)	Magnesium	3050B (mod.)	6010B/C	Solid	Certified Standard Values	<20	5000
(ICP-AES)	Magnesium	3051 (mod.)	6010B/C	Solid	Certified Standard Values	<20	5000
(ICP-AES)	Magnesium	3015/3010 (mod.)	6010B/C	Liquid/Aqueous	85 - 115	<20	100
(ICP-AES)	Magnesium	200.2 (mod.), NPDES	200.7	Liquid/Aqueous	85 - 115	<20	100
(ICP-MS)	Manganese	3051 (mod.)	6020/A (mod.)	Solid	Certified Standard Values	<20	100
(ICP-AES)	Manganese	3050B (mod.)	6010B/C	Solid	Certified Standard Values	<20	500
(ICP-AES)	Manganese	3051 (mod.)	6010B/C	Solid	Certified Standard Values	<20	500
(ICP-MS)	Manganese	3015/3010 (mod.)	6020/A (mod.)	Liquid/Aqueous	85 - 115	<20	2
(ICP-MS)	Manganese	200.2 (mod.), NPDES	200.8	Liquid/Aqueous	85 - 115	<20	2
(ICP-AES)	Manganese	3015/3010 (mod.)	6010B/C	Liquid/Aqueous	85 - 115	<20	10
(ICP-AES)	Manganese	200.2 (mod.), NPDES	200.7	Liquid/Aqueous	85 - 115	<20	10
(CVAA)	Mercury	7471 (mod.)	7471	Solid	Certified Standard Values	<20	20
(CVAA)	Mercury	1311-12	7470A	Leachate	85 - 115	<20	1
(CVAA)	Mercury	245.1 (mod.)/7470A	245.1/7470A	Liquid/Aqueous	85 - 115	<20	0.2

Table 12.3A: QC Targets for Environmental Metals Accuracy (LCS), Precision and RL's

(subject to revision without notice)

Class	Analyte	Prep Method	Analysis Method	Matrix	Accuracy Range (%)	Precision (RPD)	RL (ppb)
(ICP-MS)	Molybdenum	3051 (mod.)	6020/A (mod.)	Solid	Certified Standard Values	<20	100
(ICP-AES)	Molybdenum	3050B (mod.)	6010B/C	Solid	Certified Standard Values	<20	250
(ICP-AES)	Molybdenum	3051 (mod.)	6010B/C	Solid	Certified Standard Values	<20	250
(ICP-MS)	Molybdenum	3015/3010 (mod.)	6020/A (mod.)	Liquid/Aqueous	85 - 115	<20	2
(ICP-MS)	Molybdenum	200.2 (mod.), NPDES	200.8	Liquid/Aqueous	85 - 115	<20	2
(ICP-AES)	Molybdenum	3015/3010 (mod.)	6010B/C	Liquid/Aqueous	85 - 115	<20	5
(ICP-AES)	Molybdenum	200.2 (mod.), NPDES	200.7	Liquid/Aqueous	85 - 115	<20	5
(ICP-MS)	Nickel	3051 (mod.)	6020/A (mod.)	Solid	Certified Standard Values	<20	50
(ICP-AES)	Nickel	3050B (mod.)	6010B/C	Solid	Certified Standard Values	<20	1000
(ICP-AES)	Nickel	3051 (mod.)	6010B/C	Solid	Certified Standard Values	<20	1000
(ICP-AES)	Nickel	1311-12	6010B/C	Leachate	85 - 115	<20	50
(ICP-AES)	Nickel	3015/3010 (mod)	6020/A (mod.)	Liquid/Aqueous	85 - 115	<20	1
(ICP-MS)	Nickel	200.2 (mod.), NPDES	200.8	Liquid/Aqueous	85 - 115	<20	1
(ICP-AES)	Nickel	3015/3010 (mod.)	6010B/C	Liquid/Aqueous	85 - 115	<20	20
(ICP-AES)	Nickel	200.2 (mod.), NPDES	200.7	Liquid/Aqueous	85 - 115	<20	20
(ICP-AES)	Potassium	3050B (mod.)	6010B/C	Solid	Certified Standard Values	<20	25000
(ICP-AES)	Potassium	3051 (mod.)	6010B/C	Solid	Certified Standard Values	<20	25000
(ICP-AES)	Potassium	3015/3010 (mod.)	6010B/C	Liquid/Aqueous	85 - 115	<20	500
(ICP-AES)	Potassium	200.2 (mod.), NPDES	200.7	Liquid/Aqueous	85 - 115	<20	500
(ICP-MS)	Selenium	3051 (mod.)	6020/A (mod.)	Solid	Certified Standard Values	<20	50
(ICP-AES)	Selenium	3050B (mod.)	6010B/C	Solid	Certified Standard Values	<20	1000
(ICP-AES)	Selenium	3051 (mod.),	6010B/C	Solid	Certified Standard Values	<20	1000
(ICP-AES)	Selenium	1311-12	6010B/C	Leachate	85 - 115	<20	50
(ICP-MS)	Selenium	3015/3010 (mod.)	6020/A	Liquid/Aqueous	85 - 115	<20	1
(ICP-MS)	Selenium	200.2 (mod.), NPDES	200.8	Liquid/Aqueous	85 - 115	<20	1
(ICP-AES)	Selenium	3015/3010 (mod.)	6010B/C	Liquid/Aqueous	85 - 115	<20	20
(ICP-AES)	Selenium	200.2 (mod.), NPDES	200.7	Liquid/Aqueous	85 - 115	<20	20
(ICP-AES)	Silicon	3050B (mod.)	6010B/C	Solid	85-115	<20	10000
(ICP-AES)	Silicon	3051 (mod.)	6010B/C	Solid	85-115	<20	10000
(ICP-AES)	Silicon	3015/3010 (mod.)	6010B/C	Liquid/Aqueous	85 - 115	<20	200
(ICP-AES)	Silicon	200.2 (mod.) NPDES	200.7	Liquid/Aqueous	85 - 115	<20	200

Table 12.3A: QC Targets for Environmental Metals Accuracy (LCS), Precision and RL's

(subject to revision without notice)

Class	Analyte	Prep Method	Analysis Method	Matrix	Accuracy Range (%)	Precision (RPD)	RL (ppb)
(ICP-MS)	Silver	3050B (mod.)	6020/A (mod.)	Solid	Certified Standard Values	<20	25
(ICP-AES)	Silver	3050B (mod.)	6010B/C	Solid	Certified Standard Values	<20	500
(ICP-AES)	Silver	1311-12	6010B/C	Leachate	85 - 115	<20	50
(ICP-MS)	Silver	3015/3010 (mod.)	6020/A	Liquid/Aqueous	85 - 115	<20	0.5
(ICP-MS)	Silver	200.2 (mod.), NPDES	200.8	Liquid/Aqueous	85 - 115	<20	0.5
(ICP-AES)	Silver	3015/3010 (mod.)	6010B/C	Liquid/Aqueous	85 - 115	<20	10
(ICP-AES)	Silver	200.2 (mod.), NPDES	200.7	Liquid/Aqueous	85 - 115	<20	10
(ICP-AES)	Sodium	3050B (mod.)	6010B/C	Solid	Certified Standard Values	<20	25000
(ICP-AES)	Sodium	3051 (mod.)	6010B/C	Solid	Certified Standard Values	<20	25000
(ICP-AES)	Sodium	3015/3010 (mod.)	6010B/C	Liquid/Aqueous	85 - 115	<20	500
(ICP-AES)	Sodium	200.2 (mod.) NPDES	200.7	Liquid/Aqueous	85 - 115	<20	500
(ICP-AES)	Strontium	3050B (mod.)	6010B/C	Solid	Certified Standard Values	<20	500
(ICP-AES)	Strontium	3051 (mod.)	6010B/C	Solid	Certified Standard Values	<20	500
(ICP-AES)	Strontium	3015/3010 (mod.)	6010B/C	Liquid/Aqueous	85 - 115	<20	10
(ICP-AES)	Strontium	200.2 (mod.), NPDES	200.7	Liquid/Aqueous	85 - 115	<20	10
(ICP-AES)	Sulfur	3050B (mod.)	6010B/C	Solid	85-115	<20	50000
(ICP-AES)	Sulfur	3051 (mod.)	6010B/C	Solid	85-115	<20	50000
(ICP-AES)	Sulfur	3015/3010 (mod.)	6010B/C	Liquid/Aqueous	85 - 115	<20	1000
(ICP-AES)	Sulfur	200.2 (mod.) NPDES	200.7	Liquid/Aqueous	85 - 115	<20	1000
(ICP-MS)	Thallium	3051 (mod.)	6020/A (mod.)	Solid	Certified Standard Values	<20	50
(ICP-AES)	Thallium	3050B (mod.)	6010B/C	Solid	Certified Standard Values	<20	1000
(ICP-AES)	Thallium	3051 (mod.)	6010B/C	Solid	Certified Standard Values	<20	1000
(ICP-AES)	Thallium	3015/3010 (mod.)	6020/A	Liquid/Aqueous	85 - 115	<20	1
(ICP-MS)	Thallium	200.2 (mod.), NPDES	200.8	Liquid/Aqueous	85 - 115	<20	1
(ICP-AES)	Thallium	3015/3010 (mod.)	6010B/C	Liquid/Aqueous	85 - 115	<20	20
(ICP-AES)	Thallium	NPDES	200.7	Liquid/Aqueous	85 - 115	<20	20
(ICP-MS)	Tin	3051 (mod.)	6020/A (mod.)	Solid	Certified Standard Values	<20	50
(ICP-AES)	Tin	3050B (mod.)	6010B/C	Solid	Certified Standard Values	<20	1000
(ICP-AES)	Tin	3051 (mod.)	6010B/C	Solid	Certified Standard Values	<20	1000
(ICP-MS)	Tin	3015/3010 (mod.)	6020/A	Liquid/Aqueous	85 - 115	<20	1
(ICP-MS)	Tin	200.2 (mod.), NPDES	200.8	Liquid/Aqueous	85 - 115	<20	1

Table 12.3A: QC Targets for Environmental Metals Accuracy (LCS), Precision and RL's

(subject to revision without notice)

Class	Analyte	Prep Method	Analysis Method	Matrix	Accuracy Range (%)	Precision (RPD)	RL (ppb)
(ICP-AES)	Tin	3015/3010 (mod.)	6010B/C	Liquid/Aqueous	85 - 115	<20	20
(ICP-AES)	Tin	200.2 (mod.), NPDES	200.7	Liquid/Aqueous	85 - 115	<20	20
(ICP-AES)	Titanium	3051 (mod.)	6010B/C	Solid	Certified Standard Values	<20	500
(ICP-AES)	Titanium	3050B (mod.)	6010B/C	Solid	Certified Standard Values	<20	500
(ICP-AES)	Titanium	3015/3010 (mod.)	6010B/C	Liquid/Aqueous	85 - 115	<20	10
(ICP-AES)	Titanium	200.2 (mod.), NPDES	200.7	Liquid/Aqueous	85 - 115	<20	10
(ICP-MS)	Vanadium	3051 (mod.)	6020/A (mod.)	Solid	Certified Standard Values	<20	100
(ICP-AES)	Vanadium	3050B (mod.)	6010B/C	Solid	Certified Standard Values	<20	500
(ICP-AES)	Vanadium	3051 (mod.)	6010B/C	Solid	Certified Standard Values	<20	500
(ICP-MS)	Vanadium	3015/3010 (mod.)	6020/A (mod.)	Liquid/Aqueous	85 - 115	<20	2
(ICP-MS)	Vanadium	200.2 (mod.), NPDES	200.8	Liquid/Aqueous	85 - 115	<20	2
(ICP-AES)	Vanadium	3015/3010 (mod.)	6010B/C	Liquid/Aqueous	85 - 115	<20	10
(ICP-AES)	Vanadium	200.2 (mod.), NPDES	200.7	Liquid/Aqueous	85 - 115	<20	10
(ICP-MS)	Zinc	3051 (mod.)	6020/A (mod.)	Solid	Certified Standard Values	<20	500
(ICP-AES)	Zinc	3050B (mod.)	6010B/C	Solid	Certified Standard Values	<20	1500
(ICP-AES)	Zinc	3051 (mod.)	6010B/C	Solid	Certified Standard Values	<20	1500
(ICP-AES)	Zinc	1311-12	6010B/C	Leachate	85 - 115	<20	50
(ICP-MS)	Zinc	3015/3010 (mod.)	6020/A (mod.)	Liquid/Aqueous	85 - 115	<20	10
(ICP-MS)	Zinc	200.2 (mod.), NPDES	200.8	Liquid/Aqueous	85 - 115	<20	10
(ICP-AES)	Zinc	3015/3010 (mod.)	6010B/C	Liquid/Aqueous	85 - 115	<20	30
(ICP-AES)	Zinc	200.2 (mod.), NPDES	200.7	Liquid/Aqueous	85 - 115	<20	30

Table 12.3B: QC Targets for IH Metals Accuracy (LCS), Precision and RL's

(subject to revision without notice)

Class	Analyte	Prep Method	Analysis Method	Matrix	Accuracy Range (%)	Precision (% RPD)	RL
(ICP-AES)	Lead	3050B (mod.)	6010B/C	Filters	85-115	<20	2.5 ug/sample
(ICP-AES)	Lead	3050B (mod.)	6010B/C	Paint Chips	80-120	<20	50. mg/kg
(ICP-AES)	Lead	3050B (mod.)	6010B/C	Wipes	80-120	<20	2.0 ug/sample

13.0 CORRECTIVE ACTIONS

13.1 In the event that a nonconformance occurs in conjunction with the analytical batch, a corrective action response (CAR) form must be completed. The reason for the nonconformance is stated on the form and the measures taken to correct the nonconformance clearly defined. The effectiveness of the corrective action must be assessed and noted. The CAR is kept on file by the QA department. Corrective action procedures are documented in SOP #030208, *Corrective and Preventive Action*

13.2 Required Corrective Action

Control limits have been established for each type of analysis. When these control limits are exceeded, corrective action must be taken. Calculated sample spike control limits are also used.

All samples and procedures are governed by ESC's quality assurance program. General corrective actions are as follows; however additional and more specific direction is provided in the specific determinative procedure. For more information, see the appropriate determinative SOP

13.2.1 Laboratory QC Criteria and Appropriate Corrective Actions

If the analytical method contains acceptance/rejection criteria and it is more stringent than those controls generated by the laboratory the method criteria takes precedence.

13.2.2 Calibration Verification Criteria Are Not Met: Inorganic Analysis

Rejection Criteria - See Table 8.5.

Corrective Action - If a standard curve linearity is not acceptable and/or the absorbance for specific standard(s) is not analogous to historic data, the instrument settings, nebulizer, etc. are examined to ensure that nothing has been altered, clogged, etc. The working standards are made fresh, intermediate dilutions are re-checked and the instrument is re-calibrated. If a problem persists, the Department Manager or QA department is notified for further action.

If the initial reference check sample is out of control, the instrument is re-calibrated and the check sample is rerun. If the problem continues the check sample is re-prepared. If the problem still exists then the standards and reagent blank are re-prepared. If the problem persists, the Department Manager or QA department is notified for further action.

13.2.3 Out Of Control Blanks: Applies to Method, Trip, Rinsate & Instrument Blanks

Rejection Criteria - Blank reading is more than ½ the RL for Method Blanks and/or Instrument Blanks.

Corrective Action - Standard curves and samples are evaluated for any obvious contamination that may be isolated or uniform throughout the sequence. If necessary, reagents, QC samples and field samples are re-prepared and re-analyzed. Re-analyses are not initiated until the cause of the contamination is identified and resolved. If samples have already been partially prepared or analyzed, the group leader or QA department is consulted to determine if data needs to be rejected or if samples need to be reprepped.

13.2.4 Out Of Control Laboratory Control Standards (LCS)

Rejection Criteria - If the performance is outside of lab-generated control (Listed in Table 12.3).

Corrective Action - Instrument settings are checked. The LCS standard is re-analyzed. If the LCS is still out of control, re-calibration is performed, and samples affected since the last in control reference standard are re-analyzed. If the LCS fails again after re-calibration, the entire workgroup must be re-prepped. The group leader, Department Manager, or QA department is consulted for further action.

13.2.5 Out Of Control Matrix Spike Samples

Rejection Criteria - If spike recovery is outside of lab-generated control limits determined from accuracy charts on matrix spike samples from a similar matrix (i.e., water, solid, etc).

Corrective Action - Spiking technique is assessed to ascertain if the sample has been spiked correctly. The spiked sample should be 1 – 5 times the client sample concentration; otherwise, the percent recovery (%R) or relative percent difference (%RPD) of the MS/MSD is flagged as not meaningful or usable per the EPA method. The sample is re-analyzed. If an out of control situation persists, sample matrix interference is suspected and flagged.

13.2.6 Out Of Control Duplicate Samples

Rejection Criteria - Lab-generated maximum RPD limit (as listed under precision in Table 12.3)

Corrective Action - Instrument and samples checked to see if precision variance is likely (i.e., high suspended solids content, high viscosity, etc.). The duplicates are re-analyzed along with the parent sample. If problem persists, matrix interference is suspected and flagged

13.2.7 Out Of Control Matrix Spike Duplicates

These QC samples can be out of control for either accuracy, precision, or both. The appropriate corrective actions listed for either matrix spikes, duplicate samples, or both are followed.

NOTE: Some samples cannot be duplicated. This is the case for wipe samples, filters, and some water samples. When possible, sampling personnel should collect duplicate samples.

Analysis-specific corrective action lists are available for each type of analysis performed by ESC.

13.2.8 Out Of Control Calibration Standards: ICV, CCV, SSCV

Rejection Criteria - If the performance is outside of method requirements.

Corrective Action - Instrument settings are checked, calibration verification standard is rerun. If the standard is still out of control, re-calibration is performed, and samples affected since the last in control reference standard are rerun. The group leader, Department Manager, or QA department is consulted for further action.

13.3 Responsibility - It is the Department Manager's responsibility to evaluate the validity of the corrective action response and submit it to QA department for processing. In addition, the manager is responsible for appointing the appropriate person within the department to be responsible for correcting the nonconformance. When a corrective action warrants a cessation of analysis, the following personnel are responsible for executing the "stop work" order:

- Laboratory Manager
- QA Department
- Department Manager
- Technical Service Representative

14.0 RECORD KEEPING

Record keeping is outlined in SOP #010103, *Document Control and Distribution*, SOP #030203, *Reagent Logs and Records* and SOP #030201, *Data Handling and Reporting*

15.0 QUALITY AUDITS

System and data quality audits are outlined in the ESC Quality Assurance Manual Version 8.0

1.0 SIGNATORY APPROVALS

VOLATILES QUALITY ASSURANCE MANUAL

APPENDIX VI TO THE ESC QUALITY ASSURANCE MANUAL

for

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Prepared by

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NOTE: The QAM has been approved by the following people. A signed cover page is available upon request

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2.0 APPENDIX TABLE OF CONTENTS

3.0 SCOPE AND APPLICATION

This appendix discusses specific QA requirements for general analytical protocols to ensure analytical data generated from the Volatiles (VOC) laboratory are scientifically valid and are of acceptable quality. Any deviations from these requirements and any deviations that result in non-conforming work must be immediately evaluated and their corrective actions documented.

4.0 LABORATORY ORGANIZATION AND RESPONSIBILITIES

ESC Lab Sciences offers diverse environmental capabilities that enable the laboratory to provide the client with both routine and specialized services, field sampling and broad laboratory expertise. A brief outline of the organization and responsibilities as they apply to the ESC Quality Assurance Program is presented in *Section 4.0 in the ESC Quality Assurance Manual Version 8.0.*

5.0 PERSONNEL AND TRAINING

5.1 PERSONNEL

Kenneth W. Buckley, with a B.S. degree in General Science, is the Department Manager of Organics and Wet Chemistry laboratories. Mr. Buckley reviews and approves all data reduction associated with analyses in these areas and is responsible for the overall production of these laboratories; including the management of the staff and scheduling. Mr. Buckley has over 9 years of environmental laboratory experience. In his absence, J. D. Gentry, with a B.S. degree in Chemistry and over 9 years of environmental laboratory experience, assumes responsibility for Volatiles Department decisions.

5.2 TRAINING

5.2.1 All new analysts to the laboratory are trained by a primary analyst or Manager according to ESC protocol. Performance is documented using an initial demonstration of capability (IDOCs) and continuing demonstration of capability (CDOC). On-going acceptable capability in VOC analyses is demonstrated by acceptable participation in multiple proficiency testing programs (PTs) and daily Quality Control sample analyses. Documentation of analyst training is maintained on file within the department.

6.0 FACILITIES AND LABORATORY SAFETY

6.1 FACILITIES

The main area of the instrumentation laboratory in Building #2 has approximately 7000 square feet with 700 square feet of bench area and 300 square feet of preparatory area. The lighting standard is fluorescence. The air handling systems are (1) 60-ton units with gas heating and (1) 25-ton unit. The physical and air-handling separations, between this laboratory and other ESC sections, prevent potential cross-contamination between solvent vapor generation and incompatible analytical processes. The laboratory reagent water is created by reverse osmosis/DI filtration and evaluated to 0.055uS/cm to ensure purity. Waste disposal containers are located in the laboratory and Clean Harbors serves as ESC's waste disposal carrier. Waste handling is discussed in detail in Section 6.0 of the ESC Quality Assurance Manual. ESC's building information guides and site plan are shown in Appendix I.

6.2 LABORATORY SAFETY

- Laboratory access is limited when work is being performed.
- All procedures where chemicals are prepared or splashes may occur are conducted in laboratory exhaust hoods.
- ESC's laboratory safety guidelines are detailed in the *ESC Chemical Hygiene and Safety Plan.*

7.0 SAMPLING PROCEDURES

7.1 FIELD SAMPLING PROCEDURES, SAMPLE STORAGE, AND HANDLING

- Field Sampling procedure is described in Appendix III of this ESC Quality Assurance Manual. Sample information is recorded and kept on the ESC chain of custody and field logbooks.
- Matrices for VOC environmental analyses include groundwater, wastewater, drinking water, soil, and sludge.
- Sample containers, preservation methods and holding times vary depending on analyses requested. Please see determinative procedures for specific directions.
- Plastic containers or lids may NOT be used for the storage of samples due to sample contamination from the phthalate esters and other hydrocarbons in the plastic.
- Environmental sample containers should be filled carefully to prevent any portion of the sample from coming into contact with the sampler's gloves causing possible contamination.
- Containers for VOC samples should be selected carefully to minimize headspace that could lead to a low bias in the analytical results. Headspace is monitored during sample login and is documented on the Sample Receipt Corrective Action form when observed.

8.0 EQUIPMENT

8.1 EQUIPMENT LIST

LABORATORY EQUIPMENT LIST: MAJOR ITEMS - Volatiles Analysis
This table is subject to revision without notice

Item	*Manufacturer*	*Model*	*Instrument Name*	*#*	*Serial #*	*Location*
Gas Chromatograph	Hewlett Packard	5890 Series II	VOCGC	1	3333A31215	Volatiles
Gas Chromatograph	Agilent	6890	VOCGC	2	cn10609095	Volatiles
Gas Chromatograph	Hewlett Packard	5890 Series II	VOCGC	3	2950A26786	Volatiles
Gas Chromatograph	Hewlett Packard	5890 Series II	VOCGC	4	3336A50614	Volatiles
Gas Chromatograph	Hewlett Packard	5890 Series II	VOCGC	5	3027A29678	Volatiles
Gas Chromatograph	Hewlett Packard	5890 Series II	VOCGC	6	2950A27895	Volatiles
Gas Chromatograph	Hewlett Packard	5890 Series II	VOCGC	7	3313A37610	Volatiles
Gas Chromatograph	Hewlett Packard	5890 Series II	VOCGC	8	3033A31856	Volatiles
Gas Chromatograph	Hewlett Packard	5890 Series II	VOCGC	13	2921A23548	Volatiles
Gas Chromatograph	Agilent	6890	VOCGC	10	US00022519	Volatiles
Gas Chromatograph	Agilent	6890	VOCGC	12	US00000410	Volatiles
Gas Chromatograph	Agilent	6890	VOCGC	14	CN10408054	Volatiles
Gas Chromatograph/ Mass Spectrometer	Hewlett Packard	5890 GC/ 5972 MSD	VOCMS	1	GC336A50093 MS3329A00703	Volatiles
Gas Chromatograph/ Mass Spectrometer	Agilent	6890 GC/ 5975 MSD	VOCMS	2	GCCN10641044 MSUS63234371	Volatiles
Gas Chromatograph/ Mass Spectrometer	Hewlett Packard	5890 GC/ 5972 MSD	VOCMS	3	GC3310A48625 MS3435A01982	Volatiles
Gas Chromatograph/ Mass Spectrometer	Hewlett Packard	5890 GC/ 5972 MSD	VOCMS	5	GC3310A48625 MS3341A01200	Volatiles
Gas Chromatograph/ Mass Spectrometer	Hewlett Packard	6890 GC/ 5973 MSD	VOCMS	6	CN10343037 US44647141	Volatiles
Gas Chromatograph/ Mass Spectrometer	Hewlett Packard	5890 GC/ 5972 MSD	VOCMS	9	GC3308A46997 MS3609A03629	Volatiles
Gas Chromatograph/ Mass Spectrometer	Hewlett Packard	5890 GC/ 5972 MSD	VOCMS	10	GC2921A22675 MS3329A00524	Volatiles
Gas Chromatograph/ Mass Spectrometer	Hewlett Packard	5890 GC/ 5972 MSD	VOCMS	12	GC3336A51994 MS3549A03312	Volatiles
Gas Chromatograph/ Mass Spectrometer	Hewlett Packard	5890 GC/ 5971 MSD	VOCMS	11	GC3336A61599 MS3306A04478	Volatiles
Gas Chromatograph/ Mass Spectrometer	Agilent	6890 GC/ 5973MSD	VOCMS	4	GCUS00003465 MSUS82311257	Volatiles
Gas Chromatograph/ Mass Spectrometer	Agilent	6890 GC/ 5973MSD	VOCMS	7	GCUS00040221 MS05040022	Volatiles
Gas Chromatograph/ Mass Spectrometer	Agilent	6890 GC/ 5973MSD	VOCMS	8	GCUS00040221 MS03940725	Volatiles
Gas Chromatograph/ Mass Spectrometer	Agilent	6890 GC/ 5973MSD	VOCMS	13	GCCN103390006 MSUS91911078	Volatiles

LABORATORY EQUIPMENT LIST: MAJOR ITEMS - Volatiles Analysis
This table is subject to revision without notice

Item	*Manufacturer*	*Model*	*Instrument Name*	*#*	*Serial #*	*Location*
Gas Chromatograph/ Mass Spectrometer	Agilent	6890 GC/ 5973MSD	VOCMS	14	GCUS00009794 MSUS63810153	Volatiles
Gas Chromatograph/ Mass Spectrometer	Agilent	6890 GC/ 5973MSD	VOCMS	16	GCUS00006479 MSUS82321899	Volatiles
Gas Chromatograph/ Mass Spectrometer	Agilent	6890 GC/ 5973MSD	VOCMS	17	GCUS10232130 MSUS03940744	Volatiles
Gas Chromatograph/ Mass Spectrometer	Agilent	6890 GC/ 5973MSD	VOCMS	18	GC CN10517046 MSUS03340424	Volatiles
Gas Chromatograph/ Mass Spectrometer	Agilent	6890 GC/ 5973MSD	VOCMS	19	GCCN10611062 MSUS60542638	Volatiles
Gas Chromatograph/ Mass Spectrometer	Agilent	6890 GC/ 5975MSD	VOCMS	20	GCCN621S4367 MSUS469A4832	Volatiles
Gas Chromatograph/ Mass Spectrometer	Agilent	6890 GC/ 5975MSD	VOCMS	21	GCCN621S4368 MSUS469A4833	Volatiles
Gas Chromatograph/ Mass Spectrometer	Agilent	7890 GC/ 5975MSD	VOCMS	22	GCCN10728074 MSUS71236615	Volatiles
Gas Chromatograph/ Mass Spectrometer	Agilent	6890 GC/ 5975MSD	VOCMS	23	GCCN10728068 MS71236616	Volatiles
Gas Chromatograph/ Mass Spectrometer	Agilent	7890 GC/ 5975MSD	VOCMS	24	GCCN10151020 MSUS10223406	Volatiles
Gas Chromatograph/ Mass Spectrometer	Agilent	7890 GC/ 5975MSD	VOCMS	25	GCCN99205324 MSUS98003634	Volatiles
Gas Chromatograph/ Mass Spectrometer	Agilent	7890 GC/ 5975MSD	VOCMS	26	GCCN10301152 MSUS10313616	Volatiles
Gas Chromatograph/ Mass Spectrometer	Agilent	7890 GC/ 5975MSD	VOCMS	27	GCCN10301155 MSUS10313619	Volatiles
Centurion Autosampler	(8) PTS/EST	Centurion				Volatiles
Autosampler	(27) Varian	Archon				Volatiles
Purge and Trap	(16) OI Analytical	Eclipse				Volatiles
Purge and Trap	(14) PTS/EST	Encon				Volatiles

8.2 EQUIPMENT PREVENTIVE MAINTENANCE, EQUIPMENT CALIBRATION

INSTRUMENT	P. M. DESCRIPTION	FREQUENCY
Analytical Balances	•Check with Class "I" weights	Daily; tolerance ±0.1%
Analytical Balances	•Service/Calibration (semiannual contract maintenance and calibration check)	Semiannually
Refrigerators & Incubators	•Maintenance service	As needed - determined by daily temperature performance checks
Gas Chromatograph Detectors: FID	Change Quartz jet; clean; replace flame tip	As needed - when deterioration is noticeable
Gas Chromatograph Detectors: PID	Change or clean lamp	As needed - when deterioration is noticeable
Gas Chromatograph/Mass Spectrometer	•Autotune Report	Inspected daily

8.2 EQUIPMENT PREVENTIVE MAINTENANCE, EQUIPMENT CALIBRATION		
INSTRUMENT	P. M. DESCRIPTION	FREQUENCY
Gas Chromatograph/Mass Spectrometer	•Clean ion source	As needed to maintain high mass resolution
Gas Chromatograph/Mass Spectrometer & Gas Chromatographs	•Replace septum and liner	As needed to maintain injection port inert
Gas Chromatograph/Mass Spectrometer	•Replace vacuum pump oil	Every 6 months
Gas Chromatograph/Mass Spectrometer & Gas Chromatographs	•Replace column	When separation begins to degrade
Archon/ Centurion Autosampler	•Monitor the Daily QC, including internal standards for changes or failure.	Daily with use

8.3 STANDARDS AND REAGENTS

Table 8.3A: Standard stock sources, description and calibration information.
This table is subject to revision without notice

Method	Vendor*	Description	Calibration	Storage Req.	Expiration
8260	NSI	Gases Mix	Primary	-10°C to –20°C	1 week
	NSI	Custom VOC Mix1	Primary	-10°C to –20°C	6 months
	NSI	Mix 2	Primary	4° ± 2°C	6 months
	Absolute Stds	n-Hexane	Primary	-10°C to –20°C	6 months
	Restek	TX TPH Mix (GRO)	Primary	4° ± 2°C	6 months
	Ultra	CUS-5661	Primary	-10°C to –20°C	6 months
	NSI	Custom Std	Primary	4° ± 2°C	6 months
	Absolute Std	Acrolein	Primary	4° ± 2°C	3 months
	NSI	2-CEVE	Secondary	4° ± 2°C	6 months
	Restek	Vinyl Acetate	Secondary	-10°C to –20°C	6 months
	Restek	Custom LCS Additions	Secondary	-10°C to –20°C	6 months
	Restek	Custome Voa LCS Mix 1	Secondary	-10°C to –20°C	6 months
	Absolute Stds	n-Hexane	Secondary	-10°C to –20°C	6 months
	Restek	Acrolein	Secondary	4° ± 2°C	3 months
8015 (GRO)	Restek	Certified BTEX in Unleaded Gas Composite Standard	Primary	4° ± 2°C	6 months
	NSI	Gas Composite	Secondary	4° ± 2°C	6 months
8021	Restek	WISC PVOC/GRO Mix	Primary	-10°C to –20°C	6 months
	NSI	PVOC/GRO Mix	Secondary	4° ± 2°C	6 months
VPH	NSI	VPH ICV MIX	Primary	4° ± 2°C	6 months
	NSI	VPH LSC MIX	Secondary	4° ± 2°C	6 months

Equivalent Providers may be utilized.

TABLE 8.3B: Working Standard Concentrations
This table is subject to revision without notice

ORGANIC COMPOUNDS	Method #	GC/MS	GC
VOC's by GC/MS	524.2, 624, SM6200B 20th, 8260B	GW/WW 0.5, 1, 2, 5, 10, 25, 40, 50, 100 µg/L DW 0.5, 1, 2, 5, 10, 25, 50, 100, 150 µg/L GRO 0.4, 1, 2, 4, 5, 7, 10, 20ug/mL	
BTEX/GRO, 8015MOD, WI GRO, LA TPH G, OHIO GRO, WI PVOC	BTEX 8021 GRO 8015 or state specific		BTEX 0.5, 1, 5,10, 25,50,100,150,200, 250ug/L (m,p-Xylene is doubled) GRO 0.055, 0.11, 0.55, 1.1. 2.75, 5.5, 11

TABLE 8.3B: Working Standard Concentrations
This table is subject to revision without notice

ORGANIC COMPOUNDS	Method #	GC/MS	GC
			mg/L
MADEP VPH	MADEP VPH		Aromatic C9-C10: 0.001, 0.002, 0.01, 0.02, 0.05, 0.1, 0.2, 0.4, 1.0, 2.0 mg/L Aliphatic C5-C8: 0.006, 0.012, 0.06, 0.12, 0.3, 0.6, 1.2, 2.4, 6.0, 12.0 mg/L Aliphatic C9-C12: 0.007, 0.014, 0.07, 0.14, 0.36, 0.7, 1.4, 2.8, 7.0, 14.0 mg/L
BTEX/OA1	BTEX OA1		BTEX 0.5, 1, 5,10, 25,50,100,150,200, 250ug/L (m,p-Xylene is doubled) GRO 0.055, 0.11, 0.55, 1.1. 2.75, 5.5, 11 mg/L

8.4 INSTRUMENT CALIBRATION

602 - BTEX - SOP Number 330351

The gas chromatograph is calibrated using the internal standard procedure. A standard curve is prepared using a minimum of three concentration levels for each compound of interest. The calibration standards are tabulated according to peak height or area responses against concentration for each compound and response factors are calculated. If the response factors are <10 % RSD over the working range, the average RF can be used for calculations. Alternatively, when the response factor criteria is exceeded, the analyst may utilize a linear calibration model of response ratios (i.e. Area/Ref. Area or Amt./Ref Amt.) for quantitation providing that the correlation coefficient is at least 0.990 (0.995 for USACE DOD Projects). An independent, or second source, calibration verification standard (SSCV) is analyzed after each initial calibration and should recover within ±20% of the expected concentration for each analyte.

During the analytical sequence, the stability of the initial calibration is verified, following every 10th sample and at the end of the sequence, by the analysis of continuing calibration verification (CCV) standards. The CCV must recovery within 15% of the expected concentration for each analyte.

At daily instrument startup and in lieu of performing an entire initial calibration, the working calibration curve or response factors are verified on each working day by the analysis of a Quality Control Check Standard. The responses must meet the criteria found in Table 2 of the 602 Method. If the responses do not meet these criteria, the analysis must be repeated. If the standard still does not meet the criteria, a new calibration curve is prepared.

8021B - BTEX - SOP Number 330351

The gas chromatograph is calibrated using the internal standard procedure. A standard curve is prepared using a minimum of five concentration levels for each compound of interest.

The calibration standards are tabulated according to peak height or area responses against concentration for each compound and response factors are calculated. If the response factors are <20 % RSD over the working range, the average RF can be used for calculations. Alternatively, the results can be used to plot a calibration curve of response ratios (Area/Ref. Area) vs (Amt./Ref Amt). If the response factors of the initial calibration are <20 % RSD over the calibration range, the average RF can be used for calculations. Alternatively, when the response factor criteria is exceeded, the analyst may utilize a linear calibration model of response ratios (i.e. Area/Ref. Area or Amt./Ref Amt.) for quantitation providing that the correlation coefficient is at least 0.990 (0.995 for USACE DOD Projects). An independent, or second source, calibration verification standard (SSCV) is analyzed after each initial calibration and should recover within ±20% of the expected concentration for each analyte.

At daily instrument startup and in lieu of performing an entire initial calibration, the most recent calibration curve may be verified by the analysis of check calibration verification standard (CCV). If the response for any analyte in this check varies from the predicted response by more than ±15%, the analysis must be repeated using fresh standard. If the standard still does not meet the acceptance criteria, a new initial calibration curve must be generated.

8015B/C/D & State Methods - Gasoline Range Organics - SOP Number 330351

Certain state accreditation/registration programs may have specific requirements for calibration and analysis that must be met. Those requirements supersede the general guidance provided in this section and are addressed in the determinative SOP. 8015GRO analysis, the gas chromatograph is calibrated using the internal standard procedure. A standard curve is prepared using a minimum of five concentration levels for each analyte of interest. The calibration range must represent the typical environmental sample concentration and include the RL as the lowest calibration point. The linear range of the instrument must also be monitored to ensure that the maximum calibration point is within detection range. The calibration standards are tabulated according to peak height or area responses against concentration for each compound and response factors are calculated. If the response factors of the initial calibration are <20 % RSD over the calibration range, the average RF can be used for calculations. Alternatively, when the response factor criteria is exceeded, the analyst may utilize a linear calibration model of response ratios (i.e. Area/Ref. Area or Amt./Ref Amt.) for quantitation providing that the correlation coefficient is at least 0.990 (0.995 for USACE DOD Projects). An independent, or second source, calibration verification standard (SSCV) is analyzed after each initial calibration and should meet criteria of ±20% of the expected concentration for each analyte.

The working calibration curve or response factors are verified on each working day by the analysis of one or more calibration standards. If the response of any analyte varies from the predicted response by more than 15% RSD, the analysis must be repeated using a new calibration standard. If the standard still does not meet the criteria, a new calibration curve is prepared.

8260B/C, 624, SM6200B, 524.2 - Gas Chromatography/Mass Spectrometry (GC/MS): Volatile Organics - SOP Numbers 330363 & 330364

Detector mass calibration is performed daily using the autotune function of the GC/MS analytical system and PFTBA (Perfluorotributylamine). Following verification of the appropriate masses, the instrument sensitivity is verified by injecting a tuning solution containing Bromofluorobenzene (BFB). The BFB spectra must meet the following ion abundance criteria:

Mass	Ion Abundance Criteria
50	15 to 40% of mass 95
75	30 to 60% of mass 95
95	base peak, 100% relative abundance
96	5 to 9% of mass 95
173	0% to less than 2% of mass 174
174	greater than 50% of mass 95
175	5 to 9% of mass 174
176	greater than 95% but less than 101% of mass 174
177	5 to 9% of mass 176

Successful tuning must occur every 12 hours for method 524.2, 8260B/C & SM6200B and every 24 hours for method 624.

Following successful tuning, the GC/MS is calibrated using the internal standard procedure. A standard curve is prepared using a minimum of three standards for method 624, 524.2 and five standards for method 8260B and SM6200B. The calibration standards are tabulated according to peak height or area against concentration and the concentrations and responses of the internal standard analytes. The results are used to determine a response factor for each analyte in each standard injected. A calibration curve is constructed and is determined to be acceptable if each target analyte is found to be constant over the working range as defined as:

≤15% RSD for methods 8260B/C and SM6200B,
≤20% RSD for method 524.2, and
≤35% RSD for method 624.

The calibration checks compounds (CCCs) for method 8260 must be ≤30% RSD. When these conditions are met, linearity through the origin can be assumed and the average RF can be used in place of a calibration curve. Per the analytical method, specific target analytes are defined as calibration check compounds (CCCs) or system performance check compounds (SPCCs).

Linear regression can be used for any target compound exceeding the 15% RSD criteria but less than 40% (poor performers <50%), if the correlation coefficient is 0.990 or better. For USACE projects the correlation coefficient must meet 0.995 or better. The same is true for the CCC's as long as the RSD does not exceed 30%. A second source calibration verification standard is analyzed after each calibration and should meet the criteria of ± 20%. For 524.2 the second source calibration verification standard must be within ± 30%.

SPCCs:	
Analyte	**Minimum Average Response Factor**
Chloromethane	0.10
1,1-Dichloroethane	0.10
Bromoform	0.10
Chlorobenzene	0.30
1,1,2,2-Tetrachloroethane	0.30

CCCs:	
1,1-Dicholoethene	Toluene
Chloroform	Ethylbenzene
1,2-Dichloropropane	Vinyl Chloride

The initial calibration range must represent the typical environmental sample and include the RL as the lowest calibration point. The linear range of the instrument must be monitored to ensure that the maximum calibration point is within the range. A second source calibration verification standard is analyzed after each calibration. The second source should recover within 20% for all CCC compounds and within 40% for other analytes of interest, with the exception of analytes known to perform poorly (i.e. low purging efficiency, etc.) that will meet historical limits. Following successful calibration, the analysis of field and QC samples may begin. Analysis may be performed only during the timeframe of a valid tuning cycle (12 hours for 8260B, 524.2 & SM6200B and 24 hours for 624). Following the expiration of the tuning clock, the instrument must be retuned and either recalibrated or existing calibration may be re-verified.

For 8260B, 524.2 & SM6200B analyses, daily calibration verification includes successful demonstration of BFB sensitivity and the injection of a mid-level CCV standard containing all the target analytes of interest, the CCC, and SPCC compounds. The BFB tune must meet the ion abundance criteria (see table above). Each SPCC in the calibration verification standard must meet the minimum response factors listed above. The CCC must achieve the criteria of +/- 20% RSD. Each internal standard in the CCV must recover between -50% to + 100%, when compared to the same internal standard compound in the mid-point standard of the initial calibration curve. Additionally, if the retention time of an internal standard changes by more than 30 seconds from the retention time of the same internal standard in the mid-level standard of the most recent initial calibration, the system must be evaluated, corrected, and possibly re-calibrated.

Daily calibration is accomplished for method 624 by a BFB tuning and analysis of a QC check standard. The BFB tune must meet EPA ion abundance criteria. The QC check standard must meet the criteria found in table 5 of the method.

Poor performing compounds for 8260B/524.2/SM6200B/624:

Dichlorofluoromethane	Vinyl acetate
Bromomethane	trans-1,4-Dichloro-2-butene
Chloroethane.	Alcohols (Ethanol, TBA, TAA, ETBA, TBF, Butanol)
2,2-Dichloropropane.	Iodomethane.
1,2-Dibromo-3-chloropropane	Naphthalene
2-Chloroethylvinylether (2-CEVE)	2- Methylnaphthalene
Acrolein	1- Methylnaphthalene
Acetone	4-Methyl-2-pentanone
2-Butanone	2-Hexanone

8.5 ACCEPTANCE/REJECTION OF CALIBRATION

Organic Chemistry

The initial calibration curve is compared with previous curves for the same analyte. All new standard curves are immediately checked with a secondary source or laboratory control standard prepared from a separate source than those used for calibration. All curves are visually reviewed to ensure that acceptable correlation represents linearity. Calibration curves may be rejected for nonlinearity, abnormal sensitivity, or poor response of the laboratory control standard.

Continuing calibration verification is performed on each day that initial calibration is not performed and following every tenth sample for GC analyses and once per 12 hour shift for GCMS analyses. If a check standard does not perform within established criteria, the instrument is evaluated to determine the cause. Once the issue is corrected, all samples between the last in control sample and the first out of control check is re-analyzed.

TABLE 8.5: INSTRUMENT CALIBRATION

Instrument (Analysis)	Calibration Type	Minimum Number of Standards	Type of Curve	Acceptance/ Rejection Criteria	Frequency
GC (VOC)	Initial	3 –600 series 5 –All others	Avg. RF	Must be ≤10% RSD for 601/602, ≤20%RSD for 8021B, and ≤20% difference for 8015B	As needed
	Second Source	1 Second Source		+/- 20% of true value	With each calibration
	Daily / Cont.	1/10	External	Must be within 15% of the initial calibration curve	Beginning, every 10 and ending
		1	Internal	Must be within 15% of the initial calibration curve	Every 12 hours
GC/MS VOC 8260	Initial	5 –8000 series	Avg. RF	8260B - Must be ≤15 %RSD for all target analytes and ≤30% for CCC's	As needed
	Second Source	1 Second Source		Should recover within 20% for all CCC compounds and within 40% for other analytes of interest, with the exception of analytes known to perform poorly	With each calibration
	Daily / Cont.	Tune & CCV every 12 hours		Must pass established method tuning criteria; 8260B - CCV must be ≤20% difference for CCC compounds, RF criteria for SPCC compounds must meet method criteria. Targets must meet ESC %drift criteria.	Every 12 hours
GC/MS VOC 624	Initial	3 –600 series	Avg. RF	624 - Must be ≤35 %RSD for all target analytes and ≤30% for CCC's	As needed
	Second Source	1 Second Source		Should recover within 20% for all CCC compounds and within 40% for other analytes of interest, with the exception of analytes known to perform poorly	With each calibration
	Daily / Cont.	Tune & CCV every 12 hours		Must pass established method tuning criteria; 624 - CCV must be ≤20% difference for CCC, RF for SPCC compounds must meet method criteria. Targets must meet ESC %drift criteria.	Every 12 hours

9.0 LABORATORY PRACTICES

9.1 REAGENT GRADE WATER

ASTM Type I grade water.

9.2 GLASSWARE WASHING PROCEDURE

All VOA sampling vials are purchased specifically for volatiles analysis and only used once. They are stored in a contaminant-free environment in the original carton with screw cap lids tightly fastened. All glassware used for volatiles analysis (volumetric flasks, syringes, etc.) is segregated from other laboratory glassware. Standard cleaning procedures involve rinsing three times with methanol. Volatiles spargers are kept on the autosampler at all times. Between runs, spargers are cleaned with a distilled water rinse. When a highly contaminated sample is purged, a blank is analyzed in the sparger before another sample can be purged in it. If the sparger is contaminated, it is removed from the autosampler and cleaned with soap and water then a methanol rinse followed by heating to drive off any remaining volatile contaminants. The sparger is then returned to its position and a blank analysis is performed. If the blank proves to be contaminant free, the system is then ready for further field sample analysis.

10.0 ANALYTICAL PROCEDURES

10.1 A list of laboratory SOP's associated with the volatiles laboratory can be found in the following table:

TABLE 10.1: VOLATILE DEPARTMENT SOP'S

This table is subject to revision without notice

SOP #	Title
330351	BTEX and Gasoline Range Organics by Gas Chromatography (8015B)
330351A	TNGRO
330351B	BTEXM (8021B)
330354	NC - Volatile Petroleum Hydrocarbons
330357	Volatile Organic Compounds (GRO by GCMS)
330362	8021B (601/602) Volatile Organic Compounds by Gas Chromatography
330363	Volatile Organic Compounds by Gas Chromatography/Mass Spectrometry
330364	DW Volatile Organic Compounds by GC/MS (524.2)
330365	VOC Screen using RAE Systems PID ppbRAE
330751	5035 Closed System Purge and Trap and Extraction for VOC's in Soil and Waste
330752	5030B Purge and Trap for Aqueos Samples

11.0 QUALITY CONTROL CHECKS

NOTE: For specific guidance on each determinative method, including required quality control and specific state requirements/modifications, refer to the relevant laboratory standard operating procedure(s).

11.1 ESC participates in proficiency testing (PT's) in support of various laboratory accreditations/recognitions. Environmental samples are purchased from Environmental Resource Associates (ERA). The WS, WP and solid matrix studies are completed every 6 months. PT samples are received and analyzed by method according to the vendor's instructions and according to ESC SOP.

11.2 Initial Demonstrations of Capability (IDOC's) are performed during new analyst training and/or prior to acceptance and use of any new method/instrumentation. Continuing Demonstration of Capability (CDOC's) must be updated at least annually. The associated data is filed within the department and available for review.

11.3 Matrix Spike and Matrix Spike Duplicates are performed on each batch of samples analyzed depending on analytical method requested.

11.4 A Laboratory Control Sample (LCS) and LCS Duplicate are analyzed one per batch of samples.

11.5 A method preparation blank is performed per batch of samples processed. If one-half the reporting limit [RL] is exceeded, the laboratory shall evaluate whether reprocessing of the samples is necessary, based on the following criteria:

- The blank contamination exceeds a concentration greater than 1/10 of the measured concentration of any sample in the associated preparation batch or
- The blank contamination is greater than 1/10 of the specified regulatory limit. The concentrations of common laboratory contaminants shall not exceed the reporting limit. Any samples associated with a blank that fail these criteria shall be reprocessed in a subsequent preparation batch, except where the sample analysis resulted in non-detected results for the failing analytes.

12.0 DATA REDUCTION, VALIDATION AND REPORTING

12.1 DATA REDUCTION

The analyst performs the data calculation functions and is responsible for the initial examination of the finished data. Data reduction steps applied to the raw data are outlined in SOP #030201, *Data Handling and Reporting.* The Quality Control Department performs the secondary review of the data package using the ESC SOP #030227, *Data Review*. The QC Reviewer verifies that the analysis has performed as required and meets method criteria, all associate data is present and complete, and also ensures that any additional documentation is completed as required (i.e. Ohio VAP checklists, required flags on test reports, etc.)

TABLE 12.1 Data Reduction Formulas

PARAMETER	FORMULA
GC	response of sample analyte {*area*} x final extract volume {*mL*} x dilution response factor {*area/(mg/L)*} x initial extract volume-mass {*mL or g*} *Calculations performed by HP Enviroquant Software*
GC/MS	response of analyte {*area*} x extract volume {*mL*} x dilution x int. std amt. {*area*} response factor {*area/(mg/mL)*} x initial volume-mass {*mL or g*} x int. std cal. {area} *Calculations performed by HP Enviroquant Software*

12.2 VALIDATION

The validation process consists of data generation, reduction review, and reporting results. Once data reduction is complete, validation is conducted by verification that the QC samples are within acceptable QC limits and that all documentation is complete, including the analytical report and associated QC. See Table 12.3 by method for current QC targets and controls and current reporting limits.

Marginal Excedence – When a large number of analytes exist in the LCS, it is statistically possible for a few analytes to be outside established control limits while the analytical system remains in control. These excursions must be random in nature and, if not, a review of the control limits or analytical process is necessary.

Upper and lower marginal excedence (ME) limits are established as the mean of at least 20 data points ± four times their standard deviations. The number of allowable marginal excedences per event is based on the number of analytes spiked in the LCS.

Allowable Marginal Excedence per Event	
Analytes in LCS:	**ME Allowable**
>90	5
71-90	4
51-70	3
31-50	2
11-30	1
<11	0

Organic Control Limits - The organic QC targets are statutory in nature; warning and control limits for organic analyses are initially set for groups of compounds based on preliminary method validation data. When additional data becomes available, the QC targets are reviewed. All QC targets are routinely re-evaluated at least annually (and updated, if necessary) against laboratory historical data to insure that the limits continue to reflect realistic, method achievable goals.

12.3 REPORTING

Reporting procedures are documented in SOP #030201, *Data Handling and Reporting.*

Table 12.3: QC Targets for Volatiles Accuracy (LCS), Precision and RL's
This table is subject to revision without notice

Class	Analyte	Method	Matrix	Accuracy (%)**	Prec.** (RPD)	RL	Unit
Volatiles	Dichlorodifluoromethane	8260B/C, 624, 6200B	GW, WW	39-189	24	0.001	mg/L
Volatiles	Chloromethane	8260B/C, 624, 6200B	GW, WW	45-152	20	0.001	mg/L
Volatiles	Vinyl Chloride	8260B/C, 624, 6200B	GW, WW	55-153	20	0.001	mg/L
Volatiles	Bromomethane	8260B/C, 624, 6200B	GW, WW	45-175	20	0.001	mg/L
Volatiles	Chloroethane	8260B/C, 624, 6200B	GW, WW	49-155	20	0.001	mg/L
Volatiles	Trichlorofluoromethane	8260B/C, 624, 6200B	GW, WW	54-156	20	0.001	mg/L
Volatiles	Ethyl Ether	8260B/C, 624, 6200B	GW, WW	60-142	20	0.001	mg/L
Volatiles	Acrolein	8260B/C, 624, 6200B	GW, WW	6-182	39	0.050	mg/L
Volatiles	1,1-Dichloroethene	8260B/C, 624, 6200B	GW, WW	60-130	20	0.001	mg/L
Volatiles	1,1,2-Trichloro-1,2,2-trifluoroethane	8260B/C, 624, 6200B	GW, WW	51-149	20	0.001	mg/L
Volatiles	Acetone	8260B/C, 624, 6200B	GW, WW	48-134	20	0.050	mg/L
Volatiles	Iodomethane	8260B/C, 624, 6200B	GW, WW	61-148	20	0.050	mg/L
Volatiles	Carbon Disulfide	8260B/C, 624, 6200B	GW, WW	41-148	20	0.001	mg/L
Volatiles	Methylene Chloride	8260B/C, 624, 6200B	GW, WW	64-125	20	0.005	mg/L
Volatiles	Acrylonitrile	8260B/C, 624, 6200B	GW, WW	60-140	20	0.050	mg/L
Volatiles	trans-1,2-Dichloroethene	8260B/C, 624, 6200B	GW, WW	67-129	20	0.001	mg/L
Volatiles	Methyl Tert Butyl Ether	8260B/C, 624, 6200B	GW, WW	51-142	20	0.001	mg/L

Table 12.3: QC Targets for Volatiles Accuracy (LCS), Precision and RL's
This table is subject to revision without notice

Class	Analyte	Method	Matrix	Accuracy (%)**	Prec.** (RPD)	RL	Unit
Volatiles	1,1-Dichloroethane	8260B/C, 624, 6200B	GW, WW	67-133	20	0.001	mg/L
Volatiles	Vinyl Acetate	8260B/C, 624, 6200B	GW, WW	34-178	26	0.050	mg/L
Volatiles	Di Isopropyl Ether	8260B/C, 624, 6200B	GW, WW	63-139	20	0.001	mg/L
Volatiles	2,2-Dichloropropane	8260B/C, 624, 6200B	GW, WW	46-151	20	0.001	mg/L
Volatiles	cis-1,2-Dichloroethene	8260B/C, 624, 6200B	GW, WW	72-128	20	0.001	mg/L
Volatiles	2-Butanone (MEK)	8260B/C, 624, 6200B	GW, WW	53-132	20	0.050	mg/L
Volatiles	Bromochloromethane	8260B/C, 624, 6200B	GW, WW	75-128	20	0.001	mg/L
Volatiles	Tetrahydrofuran	8260B/C, 624, 6200B	GW, WW	50-140	20	0.001	mg/L
Volatiles	Chloroform	8260B/C, 624, 6200B	GW, WW	66-126	20	0.005	mg/L
Volatiles	1,1,1-Trichloroethane	8260B/C, 624, 6200B	GW, WW	67-137	20	0.001	mg/kg
Volatiles	Carbon Tetrachloride	8260B/C, 624, 6200B	GW, WW	64-141	20	0.001	mg/kg
Volatiles	1,1-Dichloropropene	8260B/C, 624, 6200B	GW, WW	68-132	20	0.001	mg/kg
Volatiles	Benzene	8260B/C, 624, 6200B	GW, WW	67-126	20	0.001	mg/kg
Volatiles	1,2-Dichloroethane	8260B/C, 624, 6200B	GW, WW	67-133	20	0.001	mg/kg
Volatiles	Trichloroethene	8260B/C, 624, 6200B	GW, WW	74-126	20	0.001	mg/kg
Volatiles	1,2-Dichloropropane	8260B/C, 624, 6200B	GW, WW	74-122	20	0.001	mg/kg
Volatiles	Dibromomethane	8260B/C, 624, 6200B	GW, WW	73-125	20	0.001	mg/kg
Volatiles	Bromodichloromethane	8260B/C, 624, 6200B	GW, WW	68-133	20	0.001	mg/kg
Volatiles	2-Chloroethylvinyl Ether	8260B/C, 624, 6200B	GW, WW	0-171	27	0.050	mg/kg
Volatiles	cis-1,3-Dichloropropene	8260B/C, 624, 6200B	GW, WW	73-131	20	0.001	mg/kg
Volatiles	4-Methyl-2-Pentanone (MIBK)	8260B/C, 624, 6200B	GW, WW	60-142	20	0.050	mg/kg
Volatiles	Toluene	8260B/C, 624, 6200B	GW, WW	72-122	20	0.005	mg/kg
Volatiles	trans-1,3-Dichloropropene	8260B/C, 624, 6200B	GW, WW	66-137	20	0.001	mg/kg
Volatiles	1,1,2-Trichloroethane	8260B/C, 624, 6200B	GW, WW	79-123	20	0.001	mg/kg
Volatiles	Tetrachloroethene	8260B/C, 624, 6200B	GW, WW	67-135	20	0.001	mg/kg
Volatiles	1,3-Dichloropropane	8260B/C, 624, 6200B	GW, WW	77-119	20	0.001	mg/kg
Volatiles	2-Hexanone	8260B/C, 624, 6200B	GW, WW	56-147	20	0.050	mg/kg
Volatiles	Chlorodibromomethane	8260B/C, 624, 6200B	GW, WW	73-138	20	0.001	mg/kg
Volatiles	1,2-Dibromoethane	8260B/C, 624, 6200B	GW, WW	75-126	20	0.001	mg/kg
Volatiles	Chlorobenzene	8260B/C, 624, 6200B	GW, WW	77-125	20	0.001	mg/kg
Volatiles	1,1,1,2-Tetrachloroethane	8260B/C, 624, 6200B	GW, WW	75-134	20	0.001	mg/kg
Volatiles	Ethylbenzene	8260B/C, 624, 6200B	GW, WW	76-129	20	0.001	mg/kg
Volatiles	Total-Xylene	8260B/C, 624, 6200B	GW, WW	75-128	20	0.003	mg/kg
Volatiles	Styrene	8260B/C, 624, 6200B	GW, WW	78-130	20	0.001	mg/kg
Volatiles	Bromoform	8260B/C, 624, 6200B	GW, WW	60-139	20	0.001	mg/L
Volatiles	Isopropylbenzene	8260B/C, 624, 6200B	GW, WW	73-132	20	0.001	mg/L

Table 12.3: QC Targets for Volatiles Accuracy (LCS), Precision and RL's
This table is subject to revision without notice

Class	Analyte	Method	Matrix	Accuracy (%)**	Prec.** (RPD)	RL	Unit
Volatiles	Bromobenzene	8260B/C, 624, 6200B	GW, WW	76-123	20	0.001	mg/L
Volatiles	1,1,2,2-Tetrachloroethane	8260B/C, 624, 6200B	GW, WW	72-128	20	0.001	mg/L
Volatiles	1,2,3-Trichloropropane	8260B/C, 624, 6200B	GW, WW	68-130	20	0.001	mg/L
Volatiles	trans-1,4-Dichloro-2-Butene	8260B/C, 624, 6200B	GW, WW	48-139	20	0.001	mg/L
Volatiles	n-Propylbenzene	8260B/C, 624, 6200B	GW, WW	71-132	20	0.001	mg/L
Volatiles	2-Chlorotoluene	8260B/C, 624, 6200B	GW, WW	74-128	20	0.001	mg/L
Volatiles	4-Chlorotoluene	8260B/C, 624, 6200B	GW, WW	74-130	20	0.001	mg/L
Volatiles	1,3,5-Trimethylbenzene	8260B/C, 624, 6200B	GW, WW	73-134	20	0.001	mg/L
Volatiles	tert-Butylbenzene	8260B/C, 624, 6200B	GW, WW	72-134	20	0.001	mg/L
Volatiles	1,2,4-Trimethylbenzene	8260B/C, 624, 6200B	GW, WW	72-135	20	0.001	mg/L
Volatiles	sec-Butylbenzene	8260B/C, 624, 6200B	GW, WW	70-135	20	0.001	mg/L
Volatiles	1,3-Dichlorobenzene	8260B/C, 624, 6200B	GW, WW	70-121	20	0.001	mg/L
Volatiles	p-Isopropyltoluene	8260B/C, 624, 6200B	GW, WW	68-138	20	0.001	mg/L
Volatiles	1,4-Dichlorobenzene	8260B/C, 624, 6200B	GW, WW	70-121	20	0.001	mg/L
Volatiles	1,2,3-Trimethylbenzene	8260B/C, 624, 6200B	GW, WW	70-127	20	0.001	mg/L
Volatiles	1,2-Dichlorobenzene	8260B/C, 624, 6200B	GW, WW	75-122	20	0.001	mg/L
Volatiles	n-Butylbenzene	8260B/C, 624, 6200B	GW, WW	63-142	20	0.001	mg/L
Volatiles	1,2-Dibromo-3-Chloropropane	8260B/C, 624, 6200B	GW, WW	55-134	20	0.001	mg/L
Volatiles	1,2,4-Trichlorobenzene	8260B/C, 624, 6200B	GW, WW	65-137	20	0.001	mg/L
Volatiles	Hexachlorobutadiene	8260B/C, 624, 6200B	GW, WW	67-135	20	0.001	mg/L
Volatiles	Naphthalene	8260B/C, 624, 6200B	GW, WW	56-145	20	0.005	mg/L
Volatiles	1,2,3-Trichlorobenzene	8260B/C, 624, 6200B	GW, WW	63-138	20	0.001	mg/L
Volatiles	Hexane	8260B/C, 624, 6200B	GW, WW	33-167	20	0.010	mg/L
Volatiles	Acetonitrile	8260B/C, 624, 6200B	GW, WW	61.3-134-.7	25	0.050	mg/L
Volatiles	Allyl Chloride	8260B/C, 624, 6200B	GW, WW	77.9-127-.7	25	0.005	mg/L
Volatiles	Chloroprene	8260B/C, 624, 6200B	GW, WW	49.4-142.3	25	0.050	mg/L
Volatiles	Isobutanol	8260B/C, 624, 6200B	GW, WW	59.3-137.6	25	0.100	mg/L
Volatiles	1,4-Dioxane	8260B/C, 624, 6200B	GW, WW	76.2-132.3	25	0.100	mg/L
Volatiles	Methacrylonitrile	8260B/C, 624, 6200B	GW, WW	74.7-126.1	25	0.050	mg/L
Volatiles	Methyl Methacrylate	8260B/C, 624, 6200B	GW, WW	62-142.2	25	0.005	mg/L
Volatiles	Ethyl methacrylate	8260B/C, 624, 6200B	GW, WW	55.4-126.3	25	0.005	mg/L
Volatiles	Propionitrile	8260B/C, 624, 6200B	GW, WW	53.7-143.7	25	0.050	mg/L
Volatiles	Pentachloroethane	8260B/C, 624, 6200B	GW, WW	10-200	25	0.005	mg/L
Volatiles	Cyclohexanone	8260B/C, 624, 6200B	GW, WW	36.5-138.1	25	0.010	mg/L
Volatiles	Bromoethane	8260B/C, 624, 6200B	GW, WW	74.3-136.2	25	0.001	mg/L

Table 12.3: QC Targets for Volatiles Accuracy (LCS), Precision and RL's
This table is subject to revision without notice

Class	Analyte	Method	Matrix	Accuracy (%)**	Prec.** (RPD)	RL	Unit
Volatiles	2Butanol	8260B/C, 624, 6200B	GW, WW	64.8-140.6	25	0.050	mg/L
Volatiles	Ethanol	8260B/C, 624, 6200B	GW, WW	51.8-153.6	25	0.050	mg/L
Volatiles	Di-isopropyl ether	8260B/C, 624, 6200B	GW, WW	63-139	20	0.001	mg/L
Volatiles	Ethyl tert-butyl ether	8260B/C, 624, 6200B	GW, WW	63.5-131.4	25	0.001	mg/L
Volatiles	Methyl-tert-butyl ether	8260B/C, 624, 6200B	GW, WW	51-142	20	0.001	mg/L
Volatiles	Tert-Butyl alcohol	8260B/C, 624, 6200B	GW, WW	44.2-173.9	25	0.050	mg/L
Volatiles	Tert-Amyl Methyl Ether	8260B/C, 624, 6200B	GW, WW	69.3-125.1	25	0.001	mg/L
Volatiles	Dichlorodifluoromethane	8260B/C	Solid	26-186	22	0.001	mg/kg
Volatiles	Chloromethane	8260B/C	Solid	42-149	20	0.001	mg/kg
Volatiles	Vinyl Chloride	8260B/C	Solid	50-151	20	0.001	mg/kg
Volatiles	Bromomethane	8260B/C	Solid	41-175	20	0.001	mg/kg
Volatiles	Chloroethane	8260B/C	Solid	44-159	20	0.001	mg/kg
Volatiles	Trichlorofluoromethane	8260B/C	Solid	52-147	20	0.001	mg/kg
Volatiles	Ethyl Ether	8260B/C	Solid	56-147	20	0.001	mg/kg
Volatiles	Acrolein	8260B/C	Solid	3-181	31	0.050	mg/kg
Volatiles	1,1-Dichloroethene	8260B/C	Solid	53-136	20	0.001	mg/kg
Volatiles	1,1,2-Trichloro-1,2,2-trifluoroethane	8260B/C	Solid	49-155	20	0.001	mg/kg
Volatiles	Acetone	8260B/C	Solid	44-140	25	0.050	mg/kg
Volatiles	Iodomethane	8260B/C	Solid	55-156	20	0.050	mg/kg
Volatiles	Carbon Disulfide	8260B/C	Solid	36-161	20	0.001	mg/kg
Volatiles	Methylene Chloride	8260B/C	Solid	57-129	20	0.005	mg/kg
Volatiles	Acrylonitrile	8260B/C	Solid	55-143	20	0.050	mg/kg
Volatiles	trans-1,2-Dichloroethene	8260B/C	Solid	61-133	20	0.001	mg/kg
Volatiles	Methyl Tert Butyl Ether	8260B/C	Solid	44-148	20	0.001	mg/kg
Volatiles	1,1-Dichloroethane	8260B/C	Solid	61-134	20	0.001	mg/kg
Volatiles	Vinyl Acetate	8260B/C	Solid	45-163	20	0.050	mg/kg
Volatiles	Di Isopropyl Ether	8260B/C	Solid	59-143	20	0.001	mg/kg
Volatiles	2,2-Dichloropropane	8260B/C	Solid	50-147	20	0.001	mg/kg
Volatiles	cis-1,2-Dichloroethene	8260B/C	Solid	71-129	20	0.001	mg/kg
Volatiles	2-Butanone (MEK)	8260B/C	Solid	51-131	25	0.050	mg/kg
Volatiles	Bromochloromethane	8260B/C	Solid	73-130	20	0.001	mg/kg
Volatiles	Tetrahydrofuran	8260B/C	Solid	44-144	25	0.001	mg/kg
Volatiles	Chloroform	8260B/C	Solid	63-123	20	0.005	mg/kg
Volatiles	1,1,1-Trichloroethane	8260B/C	Solid	62-135	20	0.001	mg/kg
Volatiles	Carbon Tetrachloride	8260B/C	Solid	60-140	20	0.001	mg/kg
Volatiles	1,1-Dichloropropene	8260B/C	Solid	63-132	20	0.001	mg/kg

Table 12.3: QC Targets for Volatiles Accuracy (LCS), Precision and RL's
This table is subject to revision without notice

Class	Analyte	Method	Matrix	Accuracy (%)**	Prec.** (RPD)	RL	Unit
Volatiles	Benzene	8260B/C	Solid	65-128	20	0.001	mg/kg
Volatiles	1,2-Dichloroethane	8260B/C	Solid	58-141	20	0.001	mg/kg
Volatiles	Trichloroethene	8260B/C	Solid	71-126	20	0.001	mg/kg
Volatiles	1,2-Dichloropropane	8260B/C	Solid	71-128	20	0.001	mg/kg
Volatiles	Dibromomethane	8260B/C	Solid	70-130	20	0.001	mg/kg
Volatiles	Bromodichloromethane	8260B/C	Solid	66-126	20	0.001	mg/kg
Volatiles	2-Chloroethylvinyl Ether	8260B/C	Solid	0-188	39	0.050	mg/kg
Volatiles	cis-1,3-Dichloropropene	8260B/C	Solid	73-132	20	0.001	mg/kg
Volatiles	4-Methyl-2-Pentanone (MIBK)	8260B/C	Solid	61-143	23	0.050	mg/kg
Volatiles	Toluene	8260B/C	Solid	70-120	20	0.005	mg/kg
Volatiles	trans-1,3-Dichloropropene	8260B/C	Solid	70-135	20	0.001	mg/kg
Volatiles	1,1,2-Trichloroethane	8260B/C	Solid	77-124	20	0.001	mg/kg
Volatiles	Tetrachloroethene	8260B/C	Solid	65-135	20	0.001	mg/kg
Volatiles	1,3-Dichloropropane	8260B/C	Solid	76-120	20	0.001	mg/kg
Volatiles	2-Hexanone	8260B/C	Solid	62-145	23	0.050	mg/kg
Volatiles	Chlorodibromomethane	8260B/C	Solid	72-137	20	0.001	mg/kg
Volatiles	1,2-Dibromoethane	8260B/C	Solid	76-127	20	0.001	mg/kg
Volatiles	Chlorobenzene	8260B/C	Solid	75-125	20	0.001	mg/kg
Volatiles	1,1,1,2-Tetrachloroethane	8260B/C	Solid	73-134	20	0.001	mg/kg
Volatiles	Ethylbenzene	8260B/C	Solid	74-128	20	0.001	mg/kg
Volatiles	Total-Xylene	8260B/C	Solid	74-127	20	0.003	mg/kg
Volatiles	Styrene	8260B/C	Solid	76-133	20	0.001	mg/kg
Volatiles	Bromoform	8260B/C	Solid	64-139	20	0.001	mg/kg
Volatiles	Isopropylbenzene	8260B/C	Solid	73-130	20	0.001	mg/kg
Volatiles	Bromobenzene	8260B/C	Solid	75-123	20	0.001	mg/kg
Volatiles	1,1,2,2-Tetrachloroethane	8260B/C	Solid	74-129	20	0.001	mg/kg
Volatiles	1,2,3-Trichloropropane	8260B/C	Solid	70-133	20	0.001	mg/kg
Volatiles	trans-1,4-Dichloro-2-Butene	8260B/C	Solid	52-143	20	0.001	mg/kg
Volatiles	n-Propylbenzene	8260B/C	Solid	71-132	20	0.001	mg/kg
Volatiles	2-Chlorotoluene	8260B/C	Solid	73-128	20	0.001	mg/kg
Volatiles	4-Chlorotoluene	8260B/C	Solid	72-129	20	0.001	mg/kg
Volatiles	1,3,5-Trimethylbenzene	8260B/C	Solid	71-133	20	0.001	mg/kg
Volatiles	tert-Butylbenzene	8260B/C	Solid	72-132	20	0.001	mg/kg
Volatiles	1,2,4-Trimethylbenzene	8260B/C	Solid	68-135	20	0.001	mg/kg
Volatiles	sec-Butylbenzene	8260B/C	Solid	71-134	20	0.001	mg/kg
Volatiles	1,3-Dichlorobenzene	8260B/C	Solid	71-132	20	0.001	mg/kg

Table 12.3: QC Targets for Volatiles Accuracy (LCS), Precision and RL's
This table is subject to revision without notice

Class	Analyte	Method	Matrix	Accuracy (%)**	Prec.** (RPD)	RL	Unit
Volatiles	p-Isopropyltoluene	8260B/C	Solid	67-138	20	0.001	mg/kg
Volatiles	1,4-Dichlorobenzene	8260B/C	Solid	72-123	20	0.001	mg/kg
Volatiles	1,2,3-Trimethylbenzene	8260B/C	Solid	73-126	20	0.001	mg/kg
Volatiles	1,2-Dichlorobenzene	8260B/C	Solid	77-123	20	0.001	mg/kg
Volatiles	n-Butylbenzene	8260B/C	Solid	60-145	20	0.001	mg/kg
Volatiles	1,2-Dibromo-3-Chloropropane	8260B/C	Solid	61-134	21	0.001	mg/kg
Volatiles	1,2,4-Trichlorobenzene	8260B/C	Solid	61-148	20	0.001	mg/kg
Volatiles	Hexachlorobutadiene	8260B/C	Solid	65-137	20	0.001	mg/kg
Volatiles	Naphthalene	8260B/C	Solid	61-142	20	0.005	mg/kg
Volatiles	1,2,3-Trichlorobenzene	8260B/C	Solid	62-146	20	0.001	mg/kg
Volatiles	Hexane	8260B/C	Solid	28-169	20	0.010	mg/kg
Volatiles	Acetonitrile	8260B/C	Solid	59.6-170.4	25	0.050	mg/kg
Volatiles	Allyl Chloride	8260B/C	Solid	66.7-106.4	25	0.005	mg/kg
Volatiles	Chloroprene	8260B/C	Solid	61-114.3	25	0.050	mg/kg
Volatiles	Isobutanol	8260B/C	Solid	80.4-130.2	25	0.100	mg/kg
Volatiles	1,4-Dioxane	8260B/C	Solid	78.4-148.5	25	0.100	mg/kg
Volatiles	Methacrylonitrile	8260B/C	Solid	87.1-108.6	25	0.050	mg/kg
Volatiles	Methyl Methacrylate	8260B/C	Solid	90.4-141.9	25	0.005	mg/kg
Volatiles	Ethyl methacrylate	8260B/C	Solid	41.6-159	25	0.005	mg/kg
Volatiles	Propionitrile	8260B/C	Solid	77.8-136	25	0.050	mg/kg
Volatiles	Pentachloroethane	8260B/C	Solid	63.5-179.2	25	0.005	mg/kg
Volatiles	Cyclohexanone	8260B/C	Solid	21.3-170	25	0.010	mg/kg
Volatiles	Bromoethane	8260B/C	Solid	61.7-123.8	25	0.001	mg/kg
Volatiles	2Butanol	8260B/C	Solid	82.5-138.5	25	0.050	mg/kg
Volatiles	Ethanol	8260B/C	Solid	65.6-136.3	25	0.050	mg/kg
Volatiles	Di-isopropyl ether	8260B/C	Solid	59-143	20	0.001	mg/kg
Volatiles	Ethyl tert-butyl ether	8260B/C	Solid	81.4-110.9	25	0.001	mg/kg
Volatiles	Methyl-tert-butyl ether	8260B/C	Solid	44-148	20	0.001	mg/kg
Volatiles	Tert-Butyl alcohol	8260B/C	Solid	59.5-170.4	25	0.050	mg/kg
Volatiles	Tert-Amyl Methyl Ether	8260B/C	Solid	82-115.5	25	0.001	mg/kg
Volatiles	GRO	8015B/C/D	GW, WW	70-124	20	0.100	mg/L
Volatiles	Benzene	8021B, 602, 6200C	GW, WW	79 - 131	20	0.0005	mg/L
Volatiles	Toluene	8021B, 602, 6200C	GW, WW	68 - 114	20	0.005	mg/L
Volatiles	Ethylbenzene	8021B, 602, 6200C	GW, WW	68 - 125	20	0.0005	mg/L
Volatiles	m&p-Xylene	8021B, 602, 6200C	GW, WW	67 - 113	20	0.001	mg/L

Table 12.3: QC Targets for Volatiles Accuracy (LCS), Precision and RL's
This table is subject to revision without notice

Class	Analyte	Method	Matrix	Accuracy (%)**	Prec.** (RPD)	RL	Unit
Volatiles	o-Xylene	8021B, 602, 6200C	GW, WW	72 - 114	20	0.0005	mg/L
Volatiles	MTBE	8021B, 602, 6200C	GW, WW	60 - 133	20	0.001	mg/L
Volatiles	Benzene	502.2	DW	70 - 130	25	0.0005	mg/L
Volatiles	Toluene	502.2	DW	70 - 130	25	0.005	mg/L
Volatiles	Ethylbenzene	502.2	DW	70 - 130	25	0.0005	mg/L
Volatiles	m&p-Xylene	502.2	DW	70 - 130	25	0.001	mg/L
Volatiles	o-Xylene	502.2	DW	70 - 130	25	0.0005	mg/L
Volatiles	MTBE	502.2	DW	70 - 130	25	0.001	mg/L
Volatiles	GRO	8015B	Solid	67 - 135	20	0.500	mg/kg
Volatiles	Benzene	8021B	Solid	78 - 141	20	0.0025	mg/kg
Volatiles	Toluene	8021B	Solid	65 - 117	20	0.025	mg/kg
Volatiles	Ethylbenzene	8021B	Solid	69 - 133	20	0.0025	mg/kg
Volatiles	m&p-Xylene	8021B	Solid	61 - 121	20	0.005	mg/kg
Volatiles	o-Xylene	8021B	Solid	71 - 121	20	0.0025	mg/kg
Volatiles	MTBE	8021B	Solid	54 - 129	20	0.005	mg/kg
Volatiles	1,1,1,2-Tetrachloroethane	524.2	DW	70 - 130	25	0.0005	mg/L
Volatiles	1,1,1-Trichloroethane	524.2	DW	70 - 130	25	0.0005	mg/L
Volatiles	1,1,2,2-Tetrachloroethane	524.2	DW	70 - 130	25	0.0005	mg/L
Volatiles	1,1,2-Trichloroethane	524.2	DW	70 - 130	25	0.0005	mg/L
Volatiles	1,1-Dichloroethane	524.2	DW	70 - 130	25	0.0005	mg/L
Volatiles	1,1-Dichloroethene	524.2	DW	70 - 130	25	0.0005	mg/L
Volatiles	1,1-Dichloropropanone	524.2	DW	70 - 130	25		mg/L
Volatiles	1,1-Dichloropropene	524.2	DW	70 - 130	25	0.0005	mg/L
Volatiles	1,2,3-Trichlorobenzene	524.2	DW	70 - 130	25	0.0005	mg/L
Volatiles	1,2,3-Trichloropropane	524.2	DW	70 - 130	25	0.0005	mg/L
Volatiles	1,2,4-Trichlorobenzene	524.2	DW	70 - 130	25	0.0005	mg/L
Volatiles	1,2,4-Trimethylbenzene	524.2	DW	70 - 130	25	0.0005	mg/L
Volatiles	1,2-Dibromo-3-chloropropane	524.2	DW	70 - 130	25	0.0010	mg/L
Volatiles	1,2-Dibromoethane	524.2	DW	70 - 130	25	0.0010	mg/L
Volatiles	1,2-Dichlorobenzene	524.2	DW	70 - 130	25	0.0005	mg/L
Volatiles	1,2-Dichloroethane	524.2	DW	70 - 130	25	0.0005	mg/L
Volatiles	1,2-Dichloropropane	524.2	DW	70 - 130	25	0.0005	mg/L
Volatiles	1,3,5-Trimethylbenzene	524.2	DW	70 - 130	25	0.0005	mg/L
Volatiles	1,3-Dichlorobenzene	524.2	DW	70 - 130	25	0.0005	mg/L
Volatiles	1,3-Dichloropropane	524.2	DW	70 - 130	25	0.0005	mg/L

Table 12.3: QC Targets for Volatiles Accuracy (LCS), Precision and RL's
This table is subject to revision without notice

Class	Analyte	Method	Matrix	Accuracy (%)**	Prec.** (RPD)	RL	Unit
Volatiles	1,4-Dichlorobenzene	524.2	DW	70 - 130	25	0.0005	mg/L
Volatiles	1-Chlorobutane	524.2	DW	70 - 130	25		mg/L
Volatiles	2,2-Dichloropropane	524.2	DW	70 - 130	25	0.0005	mg/L
Volatiles	2-Butanone	524.2	DW	70 - 130	25		mg/L
Volatiles	2-Chlorotoluene	524.2	DW	70 - 130	25	0.0005	mg/L
Volatiles	2-Hexanone	524.2	DW	70 - 130	25		mg/L
Volatiles	2-Nitropropane	524.2	DW	70 - 130	25		mg/L
Volatiles	4-Chlorotoluene	524.2	DW	70 - 130	25	0.0005	mg/L
Volatiles	4-Isopropyltoluene	524.2	DW	70 - 130	25	0.0005	mg/L
Volatiles	4-Methyl-2-pentanone	524.2	DW	70 - 130	25		mg/L
Volatiles	Acetone	524.2	DW	70 - 130	25	0.01	mg/L
Volatiles	Acrylonitrile	524.2	DW	70 - 130	25		mg/L
Volatiles	Allyl Chloride	524.2	DW	70 - 130	25		mg/L
Volatiles	Benzene	524.2	DW	70 - 130	25	0.0005	mg/L
Volatiles	Bromobenzene	524.2	DW	70 - 130	25	0.0005	mg/L
Volatiles	Bromochloromethane	524.2	DW	70 - 130	25	0.0005	mg/L
Volatiles	Bromodichloromethane	524.2	DW	70 - 130	25	0.0005	mg/L
Volatiles	Bromoform	524.2	DW	70 - 130	25	0.0005	mg/L
Volatiles	Bromomethane	524.2	DW	70 - 130	25	0.0005	mg/L
Volatiles	Carbon Disulfide	524.2	DW	70 - 130	25		mg/L
Volatiles	Carbon Tetrachloride	524.2	DW	70 - 130	25	0.0005	mg/L
Volatiles	Chloroacetonitrile	524.2	DW	70 - 130	25		mg/L
Volatiles	THMs	524.2	DW	70 - 130	25		mg/L
Volatiles	Chlorobenzene	524.2	DW	70 - 130	25	0.0005	mg/L
Volatiles	Chloroethane	524.2	DW	70 - 130	25	0.0005	mg/L
Volatiles	Chloroform	524.2	DW	70 - 130	25	0.0005	mg/L
Volatiles	Chloromethane	524.2	DW	70 - 130	25	0.0005	mg/L
Volatiles	Cis-1,2-dichloroethene	524.2	DW	70 - 130	25	0.0005	mg/L
Volatiles	Cis-1,3-dichloropropene	524.2	DW	70 - 130	25	0.0010	mg/L
Volatiles	Dibromochloromethane	524.2	DW	70 - 130	25		mg/L
Volatiles	Dibromomethane	524.2	DW	70 - 130	25	0.0005	mg/L
Volatiles	Dichlorodifluoromethane	524.2	DW	70 - 130	25	0.0005	mg/L
Volatiles	Diethyl ether	524.2	DW	70 - 130	25		mg/L
Volatiles	Ethyl Methacrylate	524.2	DW	70 - 130	25		mg/L
Volatiles	Ethylbenzene	524.2	DW	70 - 130	25	0.0005	mg/L

Table 12.3: QC Targets for Volatiles Accuracy (LCS), Precision and RL's
This table is subject to revision without notice

Class	Analyte	Method	Matrix	Accuracy (%)**	Prec.** (RPD)	RL	Unit
Volatiles	Hexachlorobutadiene	524.2	DW	70 - 130	25	0.0005	mg/L
Volatiles	Hexachloroethane	524.2	DW	70 - 130	25		mg/L
Volatiles	Isopropylbenzene	524.2	DW	70 - 130	25	0.0005	mg/L
Volatiles	Meta-xylene	524.2	DW	70 - 130	25		mg/L
Volatiles	Methacrylonitrile	524.2	DW	70 - 130	25		mg/L
Volatiles	Methyl Iodide	524.2	DW	70 - 130	25		mg/L
Volatiles	Methylacrylate	524.2	DW	70 - 130	25		mg/L
Volatiles	Methylene Chloride	524.2	DW	70 - 130	25	0.0005	mg/L
Volatiles	Methylmethacrylate	524.2	DW	70 - 130	25		mg/L
Volatiles	Methyl-t-butyl ether	524.2	DW	70 - 130	25	0.0005	mg/L
Volatiles	Naphthalene	524.2	DW	70 - 130	25	0.0050	mg/L
Volatiles	N-butylbenzene	524.2	DW	70 - 130	25	0.0005	mg/L
Volatiles	Nitrobenzene	524.2	DW	70 - 130	25		mg/L
Volatiles	N-propylbenzene	524.2	DW	70 - 130	25	0.0005	mg/L
Volatiles	Ortho-xylene	524.2	DW	70 - 130	25		mg/L
Volatiles	Para-xylene	524.2	DW	70 - 130	25		mg/L
Volatiles	Pentachloroethane	524.2	DW	70 - 130	25		mg/L
Volatiles	Propionitrile	524.2	DW	70 - 130	25		mg/L
Volatiles	Sec-butylbenzene	524.2	DW	70 - 130	25	0.0005	mg/L
Volatiles	Styrene	524.2	DW	70 - 130	25	0.0005	mg/L
Volatiles	Tert-butylbenzene	524.2	DW	70 - 130	25	0.0005	mg/L
Volatiles	Tetrachloroethene	524.2	DW	70 - 130	25	0.0005	mg/L
Volatiles	Tetrahydrofuran	524.2	DW	70 - 130	25		mg/L
Volatiles	Toluene	524.2	DW	70 - 130	25	0.0005	mg/L
Volatiles	Trans-1,2-dichloroethene	524.2	DW	70 - 130	25	0.0005	mg/L
Volatiles	Trans-1,3-dichloropropene	524.2	DW	70 - 130	25	0.0010	mg/L
Volatiles	Trans-1,4-dichloro-2-butene	524.2	DW	70 - 130	25		mg/L
Volatiles	Trichloroethene	524.2	DW	70 - 130	25	0.0005	mg/L
Volatiles	Trichlorofluoromethane	524.2	DW	70 - 130	25	0.0005	mg/L
Volatiles	Vinyl Chloride	524.2	DW	70 - 130	25	0.0005	mg/L
Volatiles	Xylenes – total	524.2	DW	70 - 130	25		mg/L

*** Specific organizations may require limits that supersede values listed.*

13.0 CORRECTIVE ACTION

13.1 In the event that a nonconformance occurs in conjunction with the analytical batch, a corrective action response (CAR) form must be completed. The cause of the event is stated on the form and the measures taken to correct the nonconformance clearly defined. The effectiveness of the corrective action must be assessed and noted. The CAR are kept on file by the QA Department. Corrective action procedures are documented in SOP #030208, *Corrective and Preventive Action*

13.2 Required Corrective Action

Control limits have been established for each type of analysis. When these limits are exceeded, corrective action must be taken. Calculated sample spike control limits are also used.

All samples and procedures are governed by ESC's quality assurance program. General corrective actions are as follows; however additional and more specific direction is provided in the specific determinative procedure. For more information, see the appropriate determinative SOP.

13.2.1 Laboratory QC Criteria and Appropriate Corrective Actions

If the analytical method contains acceptance/rejection criteria and it is more stringent than those controls generated by the laboratory the method criteria take precedence.

13.2.2 Out Of Control Blanks: Applies to Method, Trip, Rinsate & Instrument Blanks

Rejection Criteria - Blank reading is more than twice the background absorbance or more than 1/2 RL.

Corrective Action - Blanks are reanalyzed and the response is assessed. Standard curves and samples are evaluated for any obvious contamination that is isolated or uniform throughout the run. If necessary, reagents are re-prepared. Analyses are not initiated until the problem is identified and solved. If samples have already been prepared or analyzed, the Department Manager or QA Department is consulted to determine if data needs to be rejected or if samples need to be re-prepared.

13.2.3 Out Of Control Laboratory Control Standards (LCS & LCSD)

Rejection Criteria - If the performance is outside of lab-generated control limits which are calculated as the mean of at least 20 data points +/- 3 times the standard deviation of those points (Listed in Section 12) and the marginal excedence allowance is surpassed (see section 12.2).

Corrective Action - Instrument settings are checked and the LCS standard is re-analyzed. If the LCS is still out of control, instrumentation is checked for systemic problems and repaired (if necessary). Re-calibration is performed and the samples affected since the last in control reference standard are rerun. The group leader, Department Manager, or QA Department is consulted for further action.

13.2.4 Out Of Control Matrix Spike Samples

Rejection Criteria - If sample is outside of lab-generated control limits from accuracy charts on matrix spike samples from a similar matrix (i.e., water, solid, etc). Limits are calculated as the mean of at least 20 data points +/- 3 times the standard deviation of those points.

Corrective Action - Spiking technique is assessed to ascertain if the sample has been spiked correctly. The spiked sample should be 1 – 5 times the client sample concentration; otherwise, the percent recovery (%R) or relative percent difference (%RPD) of the MS/MSD is flagged as not meaningful or usable. The sample is re-spiked and re-analyzed, along with several other similar samples in subset. If an out of control situation persists, sample matrix interference is indicated. Samples to be analyzed by standard additions are prepared (where appropriate), and the group leader, Department Manager, or QA Department is notified.

13.2.5 Out Of Control Duplicate Samples

Rejection Criteria - Lab-generated maximum RPD limit (as listed under precision in Section 12)

Corrective Action - Instrument and samples are checked to see if precision variance is likely (i.e., high suspended solids content, high viscosity, etc.). They are re-analyzed in duplicate and samples just before and just after the duplicated sample are re-checked. If problem still exists, Department Manager, or QA Department is notified to review the analytical techniques.

13.2.6 Out Of Control Matrix Spike Duplicates

Rejection Criteria - These QC samples can be out of control for accuracy, precision, or both.

Corrective Action - The appropriate corrective actions listed for either matrix spikes, duplicate samples, or both are followed.

NOTE: Some samples cannot be duplicated. This is the case for wipe samples, filters, and some water samples. When possible, sampling personnel should collect duplicate samples.

13.2.7 Out Of Control Calibration Standards: ICV, CCV, SSCV

Rejection Criteria - If the performance is outside of method requirements.

Corrective Action - Instrument settings are checked, calibration verification standard is re-analyzed. If the standard is still out of control, re-calibration is performed, and samples affected since the last in control reference standard are rerun. The group leader, Department Manager, or QA Department is consulted for further action.

14.0 RECORD KEEPING

Record keeping is outlined in SOP #010103, *Document Control and Distribution*, SOP #030203, *Reagent Logs and Records* and SOP #030201, *Data Handling and Reporting.* Volatile organics calibration data are recorded and integrated using HP Enviroquant software. Calibration data from the volatile analyses, in addition to the initial and daily calibration, includes GC/MS autotunes, DFTPP reports and surrogate recovery reports. Hard copy records of initial calibration and daily calibration are stored with chromatograms and integrated with sample data by date analyzed.

15.0 QUALITY AUDITS

System and data quality audits are outlined in the ESC Quality Assurance Manual Version 8.0.

1.0 SIGNATORY APPROVALS

Semi-Volatile
QUALITY ASSURANCE MANUAL

APPENDIX VII TO THE ESC
QUALITY ASSURANCE MANUAL

for

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Prepared by

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NOTE: The QAM has been approved by the following people. A signed cover page is available upon request

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2.0 APPENDIX TABLE OF CONTENTS

3.0 SCOPE AND APPLICATION

This appendix discusses specific QA requirements for general analytical protocols to ensure that analytical data generated from the Semi-Volatile (SVOC) laboratory are scientifically valid and are of acceptable quality. Any deviations from these requirements and any deviations that result in non-conforming work must be immediately evaluated and their corrective actions documented.

4.0 LABORATORY ORGANIZATION AND RESPONSIBILITIES

ESC Lab Sciences offers diverse environmental capabilities that enable the laboratory to provide the client with both routine and specialized services, field sampling and broad laboratory expertise. A brief outline of the organization and responsibilities as they apply to the ESC Quality Assurance Program is presented in *Section 4.0 in the ESC Quality Assurance Manual Version 8.0.*

5.0 PERSONNEL AND TRAINING

5.1 PERSONNEL

Kenneth W. Buckley, with a B.S. degree in General Science, is the Department Manager of Organics and Wet Chemistry laboratories. Mr. Buckley reviews and approves all data reduction associated with analyses in these areas and is responsible for the overall production of these laboratories; including the management of the staff and scheduling. Mr. Buckley has over 9 years of environmental laboratory experience. In his absence, Chris Johnson assumes responsibility for departmental decisions. Mr. Johnson has a B.S. degree in Biology and over 9 years of environmental laboratory experience.

5.2 TRAINING

5.2.1 All new analysts to the laboratory are trained by the primary analyst or Manager according to ESC protocol. Performance is documented using an initial demonstration of capability (IDOCs) and continuing demonstration of capability (CDOC). On-going acceptable capability in SVOC analyses and preparation is also demonstrated by acceptable participation in multiple proficiency testing programs (PTs) and daily Quality Control sample analyses. Documentation of analyst training is maintained on file within the department.

6.0 FACILITIES AND LABORATORY SAFETY

6.1 FACILITIES

The main area of the instrumentation laboratory in Building #1 has nearly 4500 square feet with approximately 220 square feet of bench area and an additional storage area of 210 square feet. The air handling system in this area is a 100-ton Trane split unit with natural gas for heating. The 4000 square feet of area in the extraction laboratory, contained in Building 5, includes roughly 330 square feet of bench area with 245 square feet of hood space. There is an additional 2000 square feet of storage for this laboratory. The air system is a 15-ton make-up unit plus 15-ton HVAC with electric heat. The physical and air-handling separations, between this laboratory and other ESC sections, prevent potential cross-contamination between solvent vapor generation and incompatible analytical processes. The laboratory reagent water is provided through the US Filter deionizer system. Waste disposal containers are located in the laboratory and Clean Harbors serves as ESC's waste disposal carrier as discussed in detail in Section 6.0 of the ESC Quality Assurance Manual. ESC's building information guides and site plan are shown in Appendix I.

6.2 LABORATORY SAFETY

- Laboratory access is limited when work is being performed.
- All procedures where chemicals are prepared or splashes may occur are conducted in laboratory exhaust hoods.
- ESC's laboratory safety guidelines are detailed in the *ESC Chemical Hygiene and Safety Plan.*

7.0 SAMPLING PROCEDURES

7.1 FIELD SAMPLING PROCEDURES, SAMPLE STORAGE, AND HANDLING

- Field Sampling procedure is described in Appendix III of this ESC Quality Assurance Manual. Sample information is recorded and kept on the ESC chain of custody and field logbooks.
- Matrices for SVOC environmental analyses include groundwater, wastewater, drinking water, soil, and sludge. Matrices for Industrial Hygiene analyses include: sorbent tubes, filters, or Organic Vapor Monitor (OVM) Badges.
- Sample containers, preservation methods and holding times vary depending on analyses requested. Please see determinative procedures for specific directions.
- Plastic containers or lids may NOT be used for the storage of samples due to possible contamination from the phthalate esters and other hydrocarbons.
- Environmental sample containers should be filled carefully to prevent any portion of the sample from coming into contact with the sampler's gloves causing possible phthalate contamination.

8.0 EQUIPMENT

8.1 EQUIPMENT LIST

LABORATORY EQUIPMENT LIST: MAJOR ITEMS - Semi-Volatiles Analysis
This table is subject to revision without notice

Item	*Manufacturer*	*Model*	*Instrument Name*	*#*	*Serial #*	*Location*
Gas Chromatograph 2	HP	6890	svcompa	2	US00004397	SVOC
Gas Chromatograph 7	Agilent	6890	svcompe	7	US10350064	SVOC
Gas Chromatograph 8	Agilent	6890	svcompp	8	DE00022534	SVOC
Gas Chromatograph 9	HP	6890	svcompj	9	US00029095	SVOC
Gas Chromatograph 10	Agilent	6890	svcompk	10	US00039655	SVOC
Gas Chromatograph 11	Agilent	6890	svcompn	11	US00040550	SVOC
Gas Chromatograph 12	Agilent	6890	svcompo	12	US00034155	SVOC
Gas Chromatograph 13	HP	6890	svcomps	13	US00010364	SVOC
Gas Chromatograph 14	HP	6890	svcompt	14	US00020581	SVOC
Gas Chromatograph 16	Agilent	6890	svcompv	16	US10212071	SVOC
Gas Chromatograph 17	Agilent	6890	svcompw	17	US10344078	SVOC
Gas Chromatograph 18	Agilent	6890	svcompd	18	US10351038	SVOC
Gas Chromatograph 19	Agilent	6890	svcompaa	19	CN10516070	SVOC
Gas Chromatograph 20	Agilent	6890	svcompab	20	CN10543031	SVOC
Gas Chromatograph 21	Agilent	7890	svcompae	21	CN 10730070	SVOC
Gas Chromatograph 22	Agilent	7890	svcompaf	22	CN 10730081	SVOC
Gas Chromatograph 23	Agilent	6890	svcompag	23	CN 92174366	SVOC
Gas Chromatograph 24	Agilent	6890	svcompah	24	CN 92174369	SVOC
Gas Chromatograph 25	Agilent	7890	svcompaj	25	CN 10091009	SVOC
Gas Chromatograph Detectors 3	Detectors	NPD/NPD	svcompo	3	N/A	SVOC
Gas Chromatograph Detectors 7	Detectors	FID	svcompe	7	N/A	SVOC
Gas Chromatograph Detectors 8	Detectors	FID	svcompp	8	N/A	SVOC
Gas Chromatograph Detectors 9	Detectors	FID	svcompj	9	N/A	SVOC
Gas Chromatograph Detectors 10	Detectors	ECD/ECD	svcompk	10	F) U11751 B) U11135	SVOC
Gas Chromatograph Detectors 11	Detectors	ECD/ECD	svcompn	11	F) U12482 B) U12481	SVOC
Gas Chromatograph Detectors 12	Detectors	FPD/FPD	svcompo	12	N/A	SVOC
Gas Chromatograph Detectors 13	Detectors	FID	svcomps	13	N/A	SVOC
Gas Chromatograph Detectors 14	Detectors	ECD/ECD	svcompt	14	F) U0418 B) U6632	SVOC

LABORATORY EQUIPMENT LIST: MAJOR ITEMS - Semi-Volatiles Analysis
This table is subject to revision without notice

Item	*Manufacturer*	*Model*	*Instrument Name*	*#*	*Serial #*	*Location*
Gas Chromatograph Detectors 16	Detectors	FID	svcompu	16	N/A	SVOC
Gas Chromatograph Detectors 17	Detectors	FID	svcompv	17	N/A	SVOC
Gas Chromatograph Detectors 18	Detectors	ECD/ECD	svcompd	18	F) U8422 B) U11613	SVOC
Gas Chromatograph Detectors 19	Detectors	ECD/ECD	svcompaa	19	F) U2620 B) U11614	SVOC
Gas Chromatograph Detectors 20	Detectors	ECD/ECD	svcompab	20	F) U8422 B) U8423	SVOC
Gas Chromatograph Detectors 21	Detectors	FID	svcompae	21	N/A	SVOC
Gas Chromatograph Detectors 22	Detectors	ECD/ECD	svcompaf	22	N/A	SVOC
Gas Chromatograph Detectors 23	Detectors	ECD/ECD	svcompag	23	F) U11733 B) U11734	SVOC
Gas Chromatograph Detectors 24	Detectors	ECD/ECD	svcompah	24	F) U13989 B) U13988	SVOC
Gas Chromatograph/Mass Spectrometer 1	Agilent	6890 GC/5973MSD	svcompf	1	GC CN10335001 MS US33220022	SVOC
Gas Chromatograph/Mass Spectrometer 2	Agilent	6890 GC/5973MSD	svcompc	2	GC US10409048 MS US35120400	SVOC
Gas Chromatograph/Mass Spectrometer 3	Agilent	6890 GC/5973MSD	svcompz	3	GC US00039611 MS US03940681	SVOC
Gas Chromatograph/Mass Spectrometer 4	Agilent	6890 GC/5973MSD	svcomph	4	GC CN10403067 MS US35120308	SVOC
Gas Chromatograph/Mass Spectrometer 5	Agilent	6890 GC/5973MSD	svcompi	5	GC US00024766 MS US91911297	SVOC
Gas Chromatograph/Mass Spectrometer 6	Agilent	6890 GC/5973MSD	svcompl	6	GC US00039647 MS US05040021	SVOC
Gas Chromatograph/Mass Spectrometer 7	Agilent	6890 GC/5973MSD	svcompm	7	GC ---------- MS US03940745	SVOC
Gas Chromatograph/Mass Spectrometer 9	Agilent	6890 GC/5973MSD	svcompx	9	GC CN10344042 MS US33220158	SVOC
Gas Chromatograph/Mass Spectrometer 10	Agilent	6890 GC/5973MSD	svcompy	10	GC CN10340045 MS US33220183	SVOC
Gas Chromatograph/Mass Spectrometer 11	Agilent	6890 GC/5975MSD		11	GC CN10509031 MS US60532657	SVOC
Gas Chromatograph/Mass Spectrometer 12	Agilent	7890 GC/5975MSD	svcompai	12	GC CN10728074/ MS 12-0706-1325	SVOC
Gas Chromatograph/Mass Spectrometer 13	Agilent	7890 GC/5975MSD	svcompak	13	GC CN10301081/ MS US10313621	SVOC
High Performance Liquid Chromatography	Agilent	1100 Series DAD/FLD	hplc1	1	DAD de01608402 FLD de23094489	SVOC
High Performance Liquid Chromatography	Agilent	1100 Series DAD/FLD	hplc2	2	DAD de30518420 FLD de11103457	SVOC
High Performance Liquid Chromatography	Agilent	1100 Series DAD	hplc3	3	DAD us64400711	SVOC
High Performance Liquid Chromatography	Agilent	1100 Series DAD/FLD	hplc4	4	DAD de43623013 FLD de92001880	SVOC

LABORATORY EQUIPMENT LIST: MAJOR ITEMS - Semi-Volatiles Analysis
This table is subject to revision without notice

Item	*Manufacturer*	*Model*	*Instrument Name*	*#*	*Serial #*	*Location*
Analytical Balance	Mettler Toledo	XS204			1122411619	Ext. Lab
Automatic Concentrators	Buchi	Syncore	Buchi	3	1461	Ext. Lab
Automatic Concentrators	Buchi	Syncore	Buchi	7	1462	Ext. Lab
Automatic Concentrators	Buchi	Syncore	Buchi	6	1463	Ext. Lab
Automatic Concentrators	Buchi	Syncore	Buchi	8	1464	Ext. Lab
Automatic Concentrators	Buchi	Syncore	Buchi	10	1466	Ext. Lab
Automatic Concentrators	Buchi	Syncore	Buchi	02	1468	Ext. Lab
Automatic Concentrators	Buchi	Syncore	Buchi	04	1469	Ext. Lab
Automatic Concentrators	Buchi	Syncore	Buchi	9	416870050003	Ext. Lab
Automatic Concentrators	Buchi	Syncore	Buchi	5	406583020005	Ext. Lab
Capping station	Horizon	MARS X			snxc2225	Ext. Lab
Capping station	Horizon	MARS X			snxc2215	Ext. Lab
Centrifuge	Labnet	Z-400			2158	Ext. Lab
Concentration Chiller	Lauda	WKL 3200			2031	Ext. Lab
Concentration Chiller	Lauda	WKL 3200			2039	Ext. Lab
Furnace	Thermo Scientific				1882	Ext. Lab
HAA Shaker	Eberbach	6010-04			1834	Ext. Lab
HAA water Bath	Thermo Scientific	280 series			2033602-102	Ext. Lab
High Intensity Ultrasonic Processor	Misonix				1379	Ext. Lab
High Intensity Ultrasonic Processor	Misonix				1382	Ext. Lab
High Intensity Ultrasonic Processor	Misonix				1888	Ext. Lab
High Intensity Ultrasonic Processor	Misonix				1381	Ext. Lab
High Intensity Ultrasonic Processor	Misonix				1640	Ext. Lab
Microwave	CEM	MARS X			1507	Ext. Lab
Microwave	CEM	MARS X			1518	Ext. Lab
OG concentrator	Horizon	SpeedVap III			1534	Ext. Lab
OG concentrator	Horizon	SpeedVap III			SN04-2020	Ext. Lab
OG SPE extractor	Horizon	SPE-DEX 3000			1481	Ext. Lab
OG SPE extractor	Horizon	SPE-DEX 3000			1482	Ext. Lab
OG SPE extractor	Horizon	SPE-DEX 3000			1483	Ext. Lab
OG SPE extractor	Horizon	SPE-DEX 3000			1484	Ext. Lab
Separatory funnel rotators	ATR				1510	Ext. Lab
Separatory funnel rotators	ATR				1511	Ext. Lab
Separatory funnel rotators	ATR				1512	Ext. Lab
Separatory funnel rotators	ATR				1513	Ext. Lab
Separatory funnel rotators	ATR				1514	Ext. Lab

LABORATORY EQUIPMENT LIST: MAJOR ITEMS - Semi-Volatiles Analysis ***This table is subject to revision without notice***						
Item	***Manufacturer***	***Model***	***Instrument Name***	***#***	***Serial #***	***Location***
Separatory funnel rotators	ATR				1515	Ext. Lab
Separatory funnel rotators	ATR				1516	Ext. Lab
Separatory funnel rotators	ATR				2055	Ext. Lab
Separatory funnel rotators	ATR				2056	Ext. Lab
Separatory funnel rotators	ATR				2057	Ext. Lab
SPE Water Extractor	UCT				1944	Ext. Lab
SPE Water Extractor	UCT				1945	Ext. Lab
Water Bath Sonicator	Branson	8510			RPA040384175E	Ext. Lab
Vacuum Pump	Gast				0908605639	Ext. Lab
Vacuum Pump	Gast				0908605639	Ext. Lab

8.2 EQUIPMENT PREVENTIVE MAINTENANCE, EQUIPMENT CALIBRATION

INSTRUMENT	**P. M. DESCRIPTION**	**FREQUENCY**
Analytical Balances	•Check with Class "I" weights	Daily-tolerance $\pm$0.1%
Analytical Balances	•Service/Calibration (semiannual contract maintenance and calibration check)	Semiannually
Refrigerators & Incubators	•Maintenance service	As needed - determined by daily temperature performance checks
Gas Chromatograph Detectors: ECD	•Bake off or Replace •Perform wipe leakage test	As needed - when deterioration is noticeable Annually
Gas Chromatograph Detectors: FID	•Change Quartz jet; clean; replace flame tip	As needed - when deterioration is noticeable
Gas Chromatograph/Mass Spectrometer	•Autotune Report	Inspected daily
Gas Chromatograph/Mass Spectrometer	•Clean ion source	As needed to maintain high mass resolution
Gas Chromatograph/Mass Spectrometer	•Replace vacuum pump oil	Every 6 months
Gas Chromatographs/Mass Spectrometer & Gas Chromatographs	•Replace septa and liner	As needed to maintain injection port inert
Gas Chromatographs/Mass Spectrometer & Gas Chromatographs	•Replace column	When separation begins to degrade
High Intensity Ultrasonic Processor - Misonix	•Check tuning criteria	Daily with use
Infrared Spectrophotometer - Foxboro Miran 1A	•Optics alignment or replacement	As needed when response begins to deteriorate

8.3 STANDARDS AND REAGENTS

Table 8.3A: Standard stock sources, description and calibration information.
This table is subject to revision without notice

Method	Vendor*	Description	Calibration	Storage Req.	Expiration
8310	Ultra	Aromatic Hydrocarbon	Primary	4° ± 2°C	6 months
	NSI	8310/610 Spike	Second Source	4° ± 2°C	6 months
DRO	NSI	DRO #2 Cal Mix	Primary	-10°C to –20°C	6 months
	NSI	DRO #2 Spike	Second Source	-10°C to –20°C	6 months
EPH TN DRO	NSI	TN-EPH Calibration Mix	Primary	-10°C to –20°C	6 months
	NSI	EPH-TN Spike	Second Source	-10°C to –20°C	6 months
RRO	NSI	30W Oil	Primary	-10°C to –20°C	6 months
PCB	Accustd	Aroclor PCB Kit	Primary	4° ± 2°C	6 months
	NSI	1260 Spike	Second Source	4° ± 2°C	6 months
Chlordane	Restek	Chlordane Mix	Primary	4° ± 2°C	6 months
Toxaphene	Restek	Toxaphene	Primary	4° ± 2°C	6 months
Pesticides	Ultra	Pest Mix	Primary	4° ± 2°C	6 months
	NSI	Pest Spike Mix	Second Source	4° ± 2°C	6 months
Herbicides	NSI	Custom Herbicide Mis	Primary	4° ± 2°C	6 months
	NSI	Herb Spike Mix	Second Source	4° ± 2°C	6 months
	Ultra/NSI	OP Cal Mix A, B	Primary	4° ± 2°C	6 months
	NSI	OP Spike Mix A, B	Second Source	4° ± 2°C	6 months
507 NP Pest	Ultra/NSI	507 Cal Mix	Primary	4° ± 2°C	2 months
	NSI	NP Pest Spike	Second Source	4° ± 2°C	2 months
THAA	Ultra/Accustd	HAA Cal Mix	Primary	-10°C to –20°C	6 months
	Accustd/NSI	HAA Spike	Second Source	-10°C to –20°C	6 months
8270	Ultra	Custom Std Mega Mix	Primary	4° ± 2°C	6 months
	Restek	Spike Mix	Second Source	4° ± 2°C	6 months
8330	Restek	Mix1, Mix2, PETN	Primary	4° ± 2°C	6 months
	Ultra, Chemservice	Mix1, Mix2, PETN	Second Source	4° ± 2°C	6 months
8011, 504.1	Accustd	504.1 Cal Mix	Primary	4° ± 2°C	1 month
	NSI	Spike Mix	Second Source	4° ± 2°C	1 month
Industrial Hygiene	Chemservice	Neat	Primary & Secondary	4° ± 2°C	6 months

**Equivalent Providers may be utilized.*

TABLE 8.3B: Working Standard Concentrations

This table is subject to revision without notice

Organic Compounds	Method #	Standard Concentrations	Storage Requirements	Expiration
Semi-Volatiles	625, SM6410B 20th, 8270C	1,2,4,8,12,16,20,30,40,50,80 (low level and regular)	4° ± 2°C	6 months
PCB's	608, SM6431B 20th, 8082	0.05, 0.1, 0.25, 0.5, 0.75, 1.0 µg/mL	4° ± 2°C	6 months
Pesticides	608, SM 6630C, 8081A, 508	0.05, 0.10, 0.20, 0.40, 0.60, 0.80 µg/mL	4° ± 2°C	6 months
Chlordane and/or Toxaphene	608, SM 6630C, 8081A, 508	0.1, 0.5, 1.0, 2.5, 5.0, 10.0 µg/mL	4° ± 2°C	6 months
PCB Arochlor 1016/1260	8082	0.05, 0.10, 0.25, 0.50, 0.75, 1.0, µg/mL	4° ± 2°C	6 months
PCB Arochlor's 1221, 1232, 1242, 1248, 1254	8082	0.5 µg/mL	4° ± 2°C	6 months
Herbicides	515.2, 8151A, SM6640C 20th	0.02, 0.05, 0.1, 0.2, 0.5, 1.0 mg/L	4° ± 2°C	6 months
OP and NP Pesticides	507 by dual-NPD, 1657A, 8141A by dual-FPD	1.0, 2.0, 5.0, 10.0, 15.0, 20.0 ug/L	4° ± 2°C	6 months
PAHs	8310, 610, SM6440B 20th 8270C SIM	0.04, 0.20,1.0,5.0,8.0,20.0,30.0,40.0 ug/L 0.025, 0.05, 0.10, 0.50, 2.0, 4.0, 10.0, 20.0 ug/L	4° ± 2°C	6 months
Nitroaromatics & Nitramines	8330	.05, 0.1, 0.25, 0.5, 2.0, 5.0, 10.0, 25.0 mg/L	NA*	NA*
EPHTN	EPH TN	10000, 6000, 4000, 2000, 1000, 400, 200, 100 mg/L	NA*	NA*
DRO	OA2 , 8015Mod, LA TPH D, LA TPH O, OHIO DRO	10000, 5000, 3000, 2000, 1000, 400, 200, 100 mg/L	NA*	NA*
MADEP EPH	MADEP EPH	Aromatics C11-C22: 17, 85, 425, 850, 1700, 3400, 6800 mg/L Aliphatic C9 - C18: 6, 30, 150, 300, 600, 1200, 2400 mg/L Aliphatic C19 - C36: 8, 40, 200, 800, 1600, 3200 mg/L	NA*	NA*
EDB, DBCP, TCP	8011, 504.1	0.01, 0.02, 0.05, 0.10, 0.25, 0.5 ug/L	NA*	NA*
THAA's	552.2	1, 2, 4, 10, 20, 30, 40, 50 ug/L	NA*	NA*
FL PRO	FL PRO	85, 850, 2550, 4250, 5950, 8500 mg/l	NA*	NA*
TX TPH	TX1005	Individual Ranges- 4.5, 10, 25, 50, 125, 250, 500, 1250, 2500 ppm. Total Range- 9.0, 20, 50, 100, 250, 500, 1000, 2500, 5000 ppm.	NA*	NA*
IH - Aromatics	NIOSH/OSHA.	10-10000 ug/sample	NA*	NA*
DROMO PAHMO	MO DRO/PAH by 8270	300, 500, 1000, 2000, 4000, 6000, 8000, 10000 mg/L 1.0, 5.0, 10, 20, 40, 60, 80, 100 ug/L	4° ± 2°C	6 months

* indicates solutions are prepared fresh daily as needed.

8.4 INSTRUMENT CALIBRATION

608/8081A or B/SM6630C - Chlorinated Pesticides – SOP Number 330344

The gas chromatograph is calibrated using either the internal or external standard calibration model. A standard curve is prepared using a minimum of three concentration levels for each compound of interest for method 608. A minimum of five concentration levels is necessary for methods 8081A/B and SM6630C. The calibration range must represent the typical environmental sample concentration and include the RL as the lowest calibration point. The linear range of the instrument must also be monitored to ensure that the maximum calibration point is within detection range. The calibration standards are tabulated according to peak height or area responses against concentration or ISTD response for each compound and calibration/response factors are calculated. If performing analysis by method 608 and the response factors of the initial calibration are < 10 % RSD for method 608 and 20% RSD for methods 8081A/B and 6630C over the calibration range, the average RF can be used for calculations. Alternatively, when the response factor criteria is exceeded, the analyst may utilize a linear calibration model of response ratios (i.e. Area/Ref. Area or Amt./Ref Amt.) for quantitation providing that the correlation coefficient is at least 0.990.

During the analytical sequence, the stability of the initial calibration curve is verified, following every 20th sample, by the analysis of a continuing calibration verification (CCV) standard. The CCV must recover within 15% of the expected concentration for each analyte. The concentration of the continuing check standard must be routinely varied to verify the entire calibration range.

At daily instrument startup and in lieu of performing an entire initial calibration, the most recent calibration curve may be verified by the analysis of initial calibration verification standard (ICV). If the response for any analyte in this check varies from the predicted response by more than $\pm$15%, the analysis must be repeated using fresh standard. If the standard still does not meet the acceptance criteria, a new initial calibration curve must be generated.

An independent, or second source, calibration verification standard (SSCV) is analyzed after each initial calibration and should recover within $\pm$20% of the expected concentration for each analyte. When analyte responses in field samples exceed the calibration range, the sample is diluted and re-analyzed.

507 - Nitrogen/Phosphorus Pesticides - SOP Number 330348

The gas chromatograph is calibrated using the external standard procedure. A standard curve is prepared using a minimum of three concentration levels for each compound of interest for method 507. The calibration range must represent the typical environmental sample concentration and include the RL as the lowest calibration point. The linear range of the instrument must also be monitored to ensure that the maximum calibration point is within detection range. The calibration standards are tabulated according to peak height or area responses against concentration for each compound and response factors are calculated. If the response factors of the initial calibration are ≤20 % RSD over the calibration range, the average RF can be used for calculations. Alternatively, when the response factor criteria is exceeded, the analyst may utilize a linear calibration model of response ratios (i.e. Area/Ref. Area or Amt./Ref Amt.) for quantitation providing that the correlation coefficient is at least 0.990.

During the analytical sequence the stability of the initial calibration is verified, following every 10th sample and at the end of the sequence, by the analysis of a continuing calibration verification (CCV) standard. The CCV must recovery within 20% of the expected concentration for each analyte. The concentration of the continuing check standard must be routinely varied to verify the entire calibration range.

At daily instrument startup and in lieu of performing an entire initial calibration, the most recent calibration curve may be verified by the analysis of check calibration verification standard (CCV). If the response for any analyte in this check varies by more than ±20% from the initial calibration, the analysis must be repeated using fresh standard. If the standard still does not meet the criteria, a new initial calibration curve must be generated.

A Quality Control Sample (QCS) is analyzed at minimum quarterly to verify calibration standards.

552.2 - HAA - SOP Number 330319

The gas chromatograph is calibrated using the internal standard procedure. A standard curve is prepared using a minimum of five concentration levels for each compound of interest. The calibration range must represent the typical environmental sample concentration and include the RL as the lowest calibration point. The linear range of the instrument must also be monitored to ensure that the maximum calibration point is within detection range. The calibration standards are tabulated according to peak height or area responses against concentration for each compound and response factors are calculated. If the response factors of the initial calibration are ≤20 % RSD over the calibration range, the average RF can be used for calculations. Alternatively, when the response factor criteria is exceeded, the analyst may utilize a linear calibration model of response ratios (i.e. Area/Ref. Area or Amt./Ref Amt.) for quantitation providing that the correlation coefficient is at least 0.990.

During the analytical sequence the stability of the initial calibration is verified, following every 10th sample and at the end of the sequence, by the analysis of a continuing calibration verification (CCV) standard. The response of the analytes in the CCV must not vary more than 30% from the initial calibration.

At daily instrument startup and in lieu of performing an entire initial calibration, the most recent calibration curve may be verified by the analysis of check calibration verification standard (CCV). If the response for any analyte in this check varies by more than ±30% from the initial calibration, the analysis must be repeated using fresh standard. If the standard still does not meet the criteria, a new initial calibration curve must be analyzed.

A Quality Control Sample (QCS) is analyzed at minimum quarterly to verify calibration standards.

515.1, 8151A, SM6640B – Herbicides - SOP Number 330320

The gas chromatograph is calibrated using the external standard procedure. A standard curve is prepared using a minimum of five concentration levels for each analyte of interest. The calibration range must represent the typical environmental sample concentration and include the RL as the lowest calibration point. The linear range of the instrument must also be monitored to ensure that the maximum calibration point is within detection range. The calibration standards are tabulated according to peak height or area responses against concentration for each compound and response factors are calculated. If the response factors of the initial calibration are ≤20 % RSD over the calibration range, the average RF can be used for calculations. Alternatively, when the response factor criteria is exceeded, the analyst may utilize a linear calibration model of response ratios (i.e. Area/Ref. Area or Amt./Ref Amt.) for quantitation providing that the correlation coefficient is at least 0.990.

During the analytical sequence, the stability of the initial calibration is verified following every 10th sample and at the end of the sequence by the analysis of a continuing calibration verification (CCV) standard. The CCV must recovery within 15% of the expected concentration for each analyte for method 8151A and within 20% for method 6640C. The value of the CCV can exceed the criteria for a single compound provided that all samples in the analytical batch are BDL (below detection limit). The concentration of the continuing check standard must be routinely varied to verify the entire calibration range.

At daily instrument startup and in lieu of performing an entire initial calibration, the most recent calibration curve may be verified by the analysis of check calibration verification standard (CCV). If the response for any analyte in this check varies from the predicted response by more than ±15%, the analysis must be repeated using fresh standard. If the standard still does not meet the criteria, a new initial calibration curve must be generated.

An independent, or second source, calibration verification standard (SSCV) is analyzed after each initial calibration and should recover within ±20% of the expected concentration for each analyte. When sample responses exceed the calibration range, the sample is diluted and re-analyzed.

8141A, 1657A – Organophosphorus Pesticides - SOP Number 330318

The gas chromatograph is calibrated using the external standard procedure. A standard curve is prepared using a minimum of five concentration levels for each analyte of interest. The calibration range must represent the typical environmental sample concentration and include the RL as the lowest calibration point. The linear range of the instrument must also be monitored to ensure that the maximum calibration point is within detection range. The calibration standards are tabulated according to peak height or area responses against concentration for each compound and response factors are calculated. If the response factors of the initial calibration are ≤20 % RSD over the calibration range, the average RF can be used for calculations. Alternatively, when the response factor criteria is exceeded, the analyst may utilize a linear calibration model of response ratios (i.e. Area/Ref. Area or Amt./Ref Amt.) for quantitation providing that the correlation coefficient is at least 0.990.

During the analytical sequence, the stability of the initial calibration is verified following every 10^{th} sample and at the end of the sequence by the analysis of a continuing calibration verification (CCV) standard. The CCV must recovery within 15% of the expected concentration for each analyte. The concentration of the continuing check standard must be routinely varied to verify the entire calibration range.

At daily instrument startup and in lieu of performing an entire initial calibration, the most recent calibration curve may be verified by the analysis of check calibration verification standard (CCV). If the response for any analyte in this check varies from the predicted response by more than ±15%, the analysis must be repeated using fresh standard. If the standard still does not meet the criteria, a new initial calibration curve must be generated.

An independent, or second source, calibration verification standard (SSCV) is analyzed after each initial calibration and should recover within ±20% of the expected concentration for each analyte. When sample responses exceed the calibration range, the sample is diluted and re-analyzed.

625, 8270C or D, SM6410B - Base/Neutrals/Acids by GC/MS: Semivolatile Organics – SOP Number 330345

Detector mass calibration is performed using the autotune function of the GC/MS analytical system and PFTBA (Perfluorotributylamine). Following verification of the appropriate masses, the instrument sensitivity is verified by injecting a tuning solution containing decafluorotriphenylphosphine (DFTPP), benzidine, pentachlorophenol and DDT. The DFTPP must meet the ion abundance criteria specified by the EPA published method.

Benzidine and pentachlorophenol are reviewed for tailing and DDT is reviewed for breakdown to DDE and DDD. Successful tuning must occur every 12 hours for method 8270C/D and every 24 hours for method 625, except where noted in the determinative SOP.

Following successful tuning, the GC/MS is calibrated using the internal standard procedure. A standard curve is prepared using a minimum of three standards for method 625 and five standards for method 8270C/D and SM6410B. The calibration standards are tabulated according to peak height or area against concentration and the concentrations and responses of the internal standard analytes. The results are used to determine a response factor for each analyte in each standard injected. A calibration curve is the constructed and is determined to be acceptable if each analyte meets the criteria specified in the determinative method. When this condition is met, linearity through the origin can be assumed and the average RF can be used in place of a calibration curve. Initial calibration that does not meet these requirements will not be accepted and recalibration must be performed. Linear regression can be used for target compounds exceeding the 15% criteria, providing that the correlation coefficient is 0.990 or better. USACE projects must meet a correlation coefficient of 0.995 or better. The initial calibration range must represent the typical environmental sample and include the RL as the lowest calibration point. The linear range of the instrument must be monitored to ensure that the maximum calibration point is within the range.

A second source calibration verification standard is analyzed after each calibration and should recover within 20% for all CCC compounds and within 40% for other analytes of interest, with the exception of analytes known to perform poorly that will meet historical limits. Following successful calibration, the analysis of field and QC samples may begin. Analysis may be performed only during the timeframe of a valid tuning cycle (12 hours for 8270C/D and 24 hours for 625). Following the expiration of the tuning clock, the instrument must be retuned and either re-calibrated or existing calibration may be re-verified.

For 8270C/D analyses, daily calibration verification includes successful demonstration of DFTPP sensitivity and the injection of a mid-level CCV standard containing all the target analytes of interest. The DFTPP tune must meet the ion abundance criteria specified within the published method. The CCC must achieve the criteria of ±20% RSD. Each internal standard in the CCV must recover between -50% to + 100%, when compared to the same internal standard compound in the mid-point standard of the initial calibration curve. Additionally, if the retention time of an internal standard changes by more than 30 seconds from the retention time of the same internal standard in the mid-level standard of the most recent initial calibration, the system must be evaluated, corrected, and possibly re-calibrated.

For 625 analyses, daily calibration verification is accomplished by a successful demonstration of DFTPP sensitivity and the injection of a mid-level CCV standard containing all the target analytes of interest. The DFTPP tune must meet the same ion abundance criteria as the 8270C analysis and the CCV standard must recover within 20 % of predicted response for all analytes of interest.

8310, 610, SM6640B - PAH's by HPLC - SOP Number 330322

610: A standard curve is prepared using a minimum of three concentration levels for each compound of interest. If the response factors are < 10 % RSD over the working range, the average RF can be used for calculations

8310 & SM6640B: Perform calibration using a minimum of 5 points. If the response factors are < 20 % RSD over the working range, the average RF can be used for calculations or linear regression may be used providing that the correlation coefficient for each analyte of interest is 0.990 or better. USACE projects must meet a correlation coefficient of 0.995 or better. The regression line must never be forced through the origin.

The initial calibration standards are tabulated according to peak height or area responses against concentration for each compound and response factors are calculated. Alternatively, the results can be used to plot a calibration curve of response ratios (Area/Ref. Area) vs (Amt./Ref Amt.). The calibration range must represent the typical environmental sample and include the RL as the lowest calibration point. The linear range of the instrument must be monitored to ensure that the maximum calibration point is within the range. A second source calibration verification standard is analyzed after each calibration and should meet criteria of ±20%.

A continuing calibration verification (CCV) must be run at the beginning of each run and every 10 samples thereafter. The continuing calibration standard is prepared from the same source as the calibration curve and must perform within ±15% of the actual value. The CCV must represent the midpoint of the calibration range.

8330A/B/C – Nitroaromatics/Nitrosamines - SOP Number 330323

A standard curve is prepared using a minimum of five concentration levels for each compound of interest. Experience indicates that a linear calibration curve with zero intercept is appropriate for each analyte. Therefore, a response factor for each analyte can be taken as the slope of the best-fit regression line. The correlation coefficient for each analyte of interest is 0.990 or better. The calibration range must represent the typical environmental sample and include the RL as the lowest calibration point. The linear range of the instrument must be monitored to ensure that the maximum calibration point is within the range. A second source calibration verification standard is analyzed after each calibration and should meet the criteria of $\pm$20%.

Daily calibration is accomplished through the analysis of midpoint calibration standards, at a minimum, at the beginning of the day, and singly after the last sample of the day (assuming a sample group of 10 samples or less). Obtain the response factor for each analyte from the mean peak heights or peak areas and compare it with the response factor obtained for the initial calibration. The mean response factor for the daily calibration must agree within ±20% of the response factor of the initial calibration. If this requirement is not met, a new initial calibration must be obtained.

8015B/C/D or State Specific Method - DRO/RRO - Various SOPs

Certain state accreditation/registration programs may have specific requirements for calibration and analysis that must be met. Those requirements supersede the general guidance provided in this section and are addressed in the determinative SOP. Generally, for 8015B/C/D analysis, the gas chromatograph is calibrated using the external standard procedure. A standard curve is prepared using a minimum of five concentration levels for each analyte of interest. The calibration range must represent the typical environmental sample concentration and include the RL as the lowest calibration point. The linear range of the instrument must also be monitored to ensure that the maximum calibration point is within detection range. The calibration standards are tabulated according to peak height or area responses against concentration for each compound and response factors are calculated. If the response factors of the initial calibration are $\leq$20 % RSD over the calibration range, the average RF can be used for calculations. Alternatively, when the response factor criteria is exceeded, the analyst may utilize a linear calibration model of response ratios (i.e. Area/Ref. Area or Amt./Ref Amt.) for quantitation providing that the correlation coefficient is at least 0.990. USACE projects must meet a correlation coefficient of 0.995 or better.

During the analytical sequence, the stability of the initial calibration is verified following every 10th sample and at the end of the sequence by the analysis of a continuing calibration verification (CCV) standard. Typically, the CCV must recovery within 15% of the expected concentration for each analyte for method 8015B/C/D; however state specific limits for the CCV may vary. See the specific SOP or published method for more guidance. The concentration of the continuing check standard must be routinely varied to verify the entire calibration range.

At daily instrument startup and in lieu of performing an entire initial calibration, the most recent calibration curve may be verified by the analysis of check calibration verification standard (CCV). If the response for any analyte in this check varies from the predicted response by more than ±15% of the expected concentration for each analyte for method 8015B/C/D or more than state specified limits, the analysis must be repeated using fresh standard. If the standard still does not meet the criteria, a new initial calibration curve must be generated.

An independent, or second source, calibration verification standard (SSCV) is analyzed after each initial calibration and should meet criteria of ±20% of the expected concentration for each analyte. When sample responses exceed the range of the standard curve, the sample is diluted to a concentration suspected to be within the calibration range and re-analyzed.

NIOSH 1501 modified – Aromatic Hydrocarbons in Air - SOP Number 330303

The gas chromatograph is calibrated using the external standard procedure. A standard curve is prepared using a minimum of six concentration levels for each analyte of interest. The calibration range must represent the typical sample concentration. The linear range of the instrument must be monitored to ensure that the maximum calibration point is within detection range. The calibration standards are tabulated according to peak height or area responses against concentration for each compound and response factors are calculated. If the response factors of the initial calibration are ≤15% RSD over the calibration range, the average RF can be used for calculations. Alternatively, when the response factor criteria is exceeded, the analyst may utilize a linear calibration model of response ratios (i.e. Area/Ref. Area or Amt./Ref Amt.) for quantitation providing that the correlation coefficient is at least 0.990. When sample responses exceed the range of the standard curve, the sample is diluted and re-analyzed. A mid-level independently prepared calibration verification standard (ICV) is analyzed following each initial calibration and should meet criteria of ±15% of the expected concentration for each analyte. Following each 10 samples and at the end of the analytical sequence, a continuing calibration verification standard is analyzed to demonstrate the continued stability of the analytical sequence. This standard should meet criteria of ±15% of the expected concentration for each analyte.

An independent, or second source, calibration verification standard (SSCV) is analyzed after each initial calibration and should meet criteria of ±20% of the expected concentration for each analyte. When sample responses exceed the range of the standard curve, the sample is diluted to a concentration suspected to be within the calibration range and re-analyzed.

Desorption Efficiency for each lot of sorbent media is determined for each analyte of interest. Desorption Efficiency for analytes on badges has been determined and is available from the manufacturer. The reporting limit from media must be verified with each batch of samples analyzed. Additionally, a Laboratory Control Sample pair (LCS & LCSD) is prepared on media for each batch of samples analyzed.

8.5 ACCEPTANCE/REJECTION OF CALIBRATION

Organic Chemistry

The initial calibration curve is compared with previous curves for the same analyte. All new standard curves are immediately checked with a secondary source or laboratory control standard prepared from a separate source than those used for calibration. All curves are visually reviewed to ensure that acceptable correlation represents linearity. Calibration curves may be rejected for nonlinearity, abnormal sensitivity, or poor response of the laboratory control standard.

Continuing calibration verification is performed on each day that initial calibration is not performed and following every tenth sample. If a check standard does not perform within established criteria then the instrument will undergo an evaluation to determine the cause. Once the issue is corrected, all samples between the last in control standard and the first out of control check will be re-analyzed.

TABLE 8.5: INSTRUMENT CALIBRATION

Instrument (Analysis)	Calibration Type	Minimum Number of Standards	Type of Curve	Acceptance/ Rejection Criteria	Frequency
Gas Chromatography	Initial	3 (600 series methods) - 5 (other) cal.stds	Avg. RF or Linear	8081A, 8151A, 6640C, 8141A, 657A: Must be ≤20% RSD 608 - ≤10% RSD	As needed
(Pest/PCB, Herbicides, Organophos/ Organonitrogen Pesticides)	Second Source	1 Second Source		+/- 20% of true value	With each calibration
	Daily / Continuing	1/10		Must be within 15% of the initial calibration curve, 20% for 6640C.	Beginning, every 10 and ending
HPLC (PAH and Explosive)	Initial	3 (600 series methods) 5 (other) cal.stds	Avg. RF or Linear	8310, 8330: Must be ≤20% RSD 610 - ≤10% RSD	As needed
	Second Source	1 Second Source		+/- 20% of true value	With each calibration
	Daily / Continuing	1/10		Must be within 15% of the initial calibration curve.	Beginning, every 10 and ending.
GC/MS Semi-volatiles 8270C/D	Initial	At least 5 cal. stds	Avg. RF or Linear	8270C - Must be ≤15% RSD, CCCs must be ≤ 30% RSD, Linear regression: 0.990 per method or 0.995 for USACE 8270D - Must be ≤20% RSD for target analytes, Linear regression: 0.990 per method or 0.995 for USACE	As needed
	Second Source	1 Second Source		Should recover within 20% for all CCC compounds and within 40% for other analytes of interest, with the exception of analytes known to perform poorly	With each calibration
	Daily / Continuing	Tune & CCV		Must pass established method criteria. See SOP.	Every 12 hours per method
GC/MS Semi-volatiles 625	Initial	3 cal.stds	Avg. RF or Linear	625 - ≤35% RSD all compounds	As needed
	Second Source	1 Second Source		Should recover within 20% for all CCC compounds and within 40% for other analytes of interest, with the exception of analytes known to perform poorly	With each calibration
	Daily / Continuing	Tune & CCV every 24 hours		Must pass established method tuning criteria; 625: CCV must be ≤20% difference for all compounds,	Every 24 hours
HAA 552.2	Initial	5 cal.stds	Avg. RF or Linear	≤30% RSD all compounds	As needed
	Second Source(QCS)	1 Second Source		±30% of true value	Quarterly
	Daily / Continuing	1/10		CCV must be ≤30% difference for all compounds,	Beginning, every 10 and ending

TABLE 8.5: INSTRUMENT CALIBRATION					
Instrument (Analysis)	**Calibration Type**	**Minimum Number of Standards**	**Type of Curve**	**Acceptance/ Rejection Criteria**	**Frequency**
Pesticides 507	Initial Second Source(QCS) Daily / Continuing	5 cal.stds 1 Second Source 1/10	Avg. RF or Linear	≤20% RSD all compounds ±20% of true value CCV must be ≤20% difference for all compounds,	As needed Quarterly Beginning, every 10 and ending
DRO –8015, State Programs* * Or per state requirement	Initial Second Source Daily / Continuing	5 cal.stds 1 Second Source 1/10	Avg. RF or Linear	8015B/C/D - ≤20% RSD all compounds ±20% of true value CCV must be ≤15% difference for all compounds,	As needed With each calibration Beginning, every 10 and ending
NIOSH 1501 mod.	Initial ICV Continuing	6 cal.stds 1 Independent Prep. 1/10	Avg. RF or Linear	≤15% RSD all compounds ±15% of true value ±15% of true value	Daily With each calibration Beginning, every 10 and ending

9.0 LABORATORY PRACTICES

9.1 REAGENT GRADE WATER

ASTM Type I grade water.

9.2 GLASSWARE WASHING AND STERILIZATION PROCEDURES

Organic laboratory glassware is washed in a non-phosphate detergent and warm tap water. Before washing, all writing and large deposits of grease are removed with acetone. Glassware is then rinsed with: tap water, "No Chromix" solution, tap water, and deionized (DI) water. It is then solvent rinsed in the following order: methanol, acetone, and then methylene chloride. Glassware is stored in designated drawers or on shelves, inverted if possible. All glassware is rinsed with the required solvent for the particular extraction protocol prior to use.

10.0 ANALYTICAL PROCEDURES

10.1 A list of laboratory SOP's associated with the semi-volatile laboratory can be found in the following table:

TABLE 10.1: SEMI-VOLATILE DEPARTMENT SOP'S

This table is subject to revision without notice

SOP #	Title
	Preparatory SOP's
330702	Separatory Funnel Liquid-Liquid Extraction 3510C
330702A	Separatory Funnel Liquid-Liquid Extraction 3510C for Minnesota Samples
330705	Ultrasonic Extraction 3550B
330707	Microwave Extraction 3546
330708	Buchi Syncore Concentration System
330743	Solid Phase Extraction
330754	Waste Dilution for SVOC's 3580A
330755	PCB in Oil Waste Dilution
	Extract Cleanup SOP's
330739	Silica Gel Cleanup 3630C
330740	Acid Cleanup 3665A
330741	Sulfur Cleanup 3660C
330742	Florisil Cleanup 3620B
	Semi-Volatiles Analysis SOPs
330303	Organics on Charcoal Tubes (includes badges)
330318	Organophosphorus Pesticides 8141A/ 1657A/ 614/ 622
330319	THAA's 552.2
330320	Chlorinated Herbicides by Gas Chromatography 8151A/ SM6640B
330322	PAH's by HPLC 8310/ 610/ SM6440B
330323	Explosives by HPLC 8330
330324	Carbamates by HPLC 531.1/ SM6610B
330343	PCB's 8082 & A
330344	Pesticides and PCBS by Gas Chromatography 8081A&B/ 608/ SM6630C
330345	Semi-volatile Organics by GC/MS using Capillary Column 8270C & D/ 625/ SM6410B
330346	EDB in Drinking Water by GC ECD 8011/ 504.1
330348	NP Pesticides in Drinking Water by GC NPD 507
330349	Chlorinated Pesticides in Drinking Water by GC ECD 508
330352	Method for Determination of Extractable Petroleum Hydrocarbons by GC/FID – DRO-KY, TN EPH, TPH-AZ, DRO CA and OH by Modified Method 8015. Includes Wyoming LAUST Requirements
330353	NC - Extractable Petroleum Hydrocarbons
330355	Florida PRO, WI DRO and CT ETPH
330356	TX TPH 1005/1006
330358	OA2 & NWTPH-Dx
330359	AK 102/103
330360	DRO Wisconsin/Minnesota

11.0 QUALITY CONTROL CHECKS

NOTE: For specific guidance on each determinative method, including required quality control and specific state requirements/modifications, refer to the relevant laboratory standard operating procedure(s).

11.1 ESC participates in proficiency testing (PT's) in support of various laboratory accreditations/recognitions. Environmental samples are purchased from Environmental Resource Associates (ERA). The WS, WP and solid matrix studies are completed every 6 months. For industrial hygiene accreditation, PTs are administered by AIHA. PT samples are received and analyzed by method according to the vendor's instructions and according to ESC SOP.

11.2 Initial Demonstrations of Capability (IDOC's) are performed during new analyst training and/or prior to acceptance and use of any new method/instrumentation. Continuing Demonstration of Capability (CDOC's) must be updated at least annually. The associated data is filed within the department and available for review.

11.3 Matrix Spike and Matrix Spike Duplicates are performed on each batch of samples analyzed depending on analytical method requested.

11.4 A Laboratory Control Sample (LCS) and LCS Duplicate are analyzed one per batch of samples.

11.5 A method preparation blank is performed per batch of samples processed. If one-half the reporting limit [RL] is exceeded, the laboratory shall evaluate whether re-processing of the samples is necessary, based on the following criteria:

- The blank contamination exceeds a concentration greater than 1/10 of the measured concentration of any sample in the associated preparation batch or
- The blank contamination is greater than 1/10 of the specified regulatory limit.

The concentrations of common laboratory contaminants shall not exceed the reporting limit. Any samples associated with a blank that fail these criteria shall be reprocessed in a subsequent preparation batch, except when the sample analysis resulted in non-detected results for the failing analytes.

11.6 For Industrial Hygiene analyses (sorbent tubes and badges), a media blank will be prepared with each batch of samples. In addition, a media reporting limit verification will be prepared with each batch of samples. For accuracy and precision determinations, a LCS/LCSD pair will be spiked on media then desorbed and analyzed concurrently with every batch of field samples.

12.0 DATA REDUCTION, VALIDATION AND REPORTING

12.1 DATA REDUCTION

The analyst performs the data calculation functions and is responsible for the initial examination of the finished data. Data reduction steps applied to the raw data are outlined in *SOP 030201 Data Handling and Reporting.* The Quality Control Department performs the secondary review of the data package using the ESC SOP #030227, *Data Review*. The QC Reviewer verifies that the analysis has performed as required and meets method criteria, all associate data is present and complete, and also ensures that any additional documentation is completed as required (i.e. Ohio VAP checklists, required flags on test reports, etc.)

TABLE 12.1 Data Reduction Formulas

PARAMETER	FORMULA
GC and HPLC	response of sample analyte {*area*} x final extract volume {*mL*} x dilution response factor {*area/(mg/mL)*} x initial extract volume-mass {*mL or g*} *Calculations performed by HP Enviroquant Software*
GC/MS	response of analyte {*area*} x extract volume {*mL*} x dilution x int. std amt. {*area*} response factor {*area/(mg/mL)*} x initial volume-mass {*mL or g*} x int. std cal. {area} *Calculations performed by HP Enviroquant Software*
GC - IH	Sample conc. (front tube + back tube) (*ug*) – blank conc. (front tube + back tube) (*ug*) Volume of air sampled (*L*)

12.2 VALIDATION

The validation process consists of data generation, reduction review, and reporting results. Once data reduction is complete, validation is conducted by verification that the QC samples are within acceptable QC limits and that all documentation is complete, including the analytical report and associated QC. See Table 12.3 by method for current QC targets and controls and current reporting limits.

Marginal Excedence – When a large number of analytes exist in the LCS, it is statistically possible for a few analytes to be outside established control limits while the analytical system remains in control. These excursions must be random in nature and, if not, a review of the control limits or analytical process is necessary.

Upper and lower marginal excedence (ME) limits are established as the mean of at least 20 data points ± four times their standard deviations. The number of allowable marginal excedences per event is based on the number of analytes spiked in the LCS.

Allowable Marginal Excedence per Event	
Analytes in LCS:	**ME Allowable**
>90	5
71-90	4
51-70	3
31-50	2
11-30	1
<11	0

Organic Control Limits - The organic QC targets are statutory in nature; warning and control limits for organic analyses are initially set for groups of compounds based on preliminary method validation data. When additional data becomes available, the QC targets are reviewed. All QC targets are routinely re-evaluated at least annually (and updated, if necessary) against laboratory historical data to insure that the limits continue to reflect realistic, method achievable goals.

12.3 REPORTING

Reporting procedures are documented in *SOP 030201 Data Handling and Reporting.*

Table 12.3: QC Targets for Semi-Volatiles Accuracy (LCS), Precision and RL's
This table is subject to revision without notice

Class	Analyte	Method	Matrix	Accuracy (%)	Prec. (RPD)	RL	Unit
Pesticides	Azinphos-Methyl	8141A, 1657A	GW	31-146	31	0.001	mg/L
Pesticides	Bolstar (Sulprofos)	8141A, 1657A	GW	46-126	27	0.001	mg/L
Pesticides	Chlorpyrifos	8141A, 1657A	GW	48-123	28	0.001	mg/L
Pesticides	Coumaphos	8141A, 1657A	GW	37-142	31	0.001	mg/L
Pesticides	Demeton,-O And -S	8141A, 1657A	GW	35-119	27	0.002	mg/L
Pesticides	Diazinon	8141A, 1657A	GW	49-118	26	0.001	mg/L
Pesticides	Dichlorvos	8141A, 1657A	GW	22-106	39	0.002	mg/L
Pesticides	Dimethoate	8141A, 1657A	GW	10-130	25	0.001	mg/L
Pesticides	Disulfoton	8141A, 1657A	GW	34-122	28	0.001	mg/L
Pesticides	Epn	8141A, 1657A	GW	36-134	29	0.001	mg/L
Pesticides	Ethoprop	8141A, 1657A	GW	44-114	28	0.001	mg/L
Pesticides	Ethyl Parathion	8141A, 1657A	GW	41-128	28	0.001	mg/L
Pesticides	Fensulfothion	8141A, 1657A	GW	40-131	28	0.001	mg/L
Pesticides	Fenthion	8141A, 1657A	GW	40-127	26	0.001	mg/L
Pesticides	Malathion	8141A, 1657A	GW	40-129	28	0.001	mg/L

Table 12.3: QC Targets for Semi-Volatiles Accuracy (LCS), Precision and RL's
This table is subject to revision without notice

Class	Analyte	Method	Matrix	Accuracy (%)	Prec. (RPD)	RL	Unit
Pesticides	Merphos	8141A, 1657A	GW	51-147	28	0.001	mg/L
Pesticides	Methyl Parathion	8141A, 1657A	GW	46-117	28	0.001	mg/L
Pesticides	Mevinphos	8141A, 1657A	GW	37-109	32	0.001	mg/L
Pesticides	Naled	8141A, 1657A	GW	13-97	32	0.001	mg/L
Pesticides	Phorate	8141A, 1657A	GW	31-125	27	0.001	mg/L
Pesticides	Ronnel	8141A, 1657A	GW	34-118	26	0.001	mg/L
Pesticides	Stirophos	8141A, 1657A	GW	45-130	28	0.001	mg/L
Pesticides	Sulfotep	8141A, 1657A	GW	33-124	31	0.001	mg/L
Pesticides	Tepp	8141A, 1657A	GW	10-107	64	0.0083	mg/L
Pesticides	Tokuthion (Prothiofos)	8141A, 1657A	GW	47-122	28	0.001	mg/L
Pesticides	Trichloronate	8141A, 1657A	GW	45-125	28	0.001	mg/L
Pesticides	Azinphos-Methyl	8141A	SS	56-123	30	0.1	mg/Kg
Pesticides	Bolstar (Sulprofos)	8141A	SS	58-113	23	0.1	mg/Kg
Pesticides	Chlorpyrifos	8141A	SS	59-106	24	0.1	mg/Kg
Pesticides	Coumaphos	8141A	SS	54-124	32	0.1	mg/Kg
Pesticides	Demeton,-O And -S	8141A	SS	50-104	24	0.1	mg/Kg
Pesticides	Diazinon	8141A	SS	55-104	20	0.1	mg/Kg
Pesticides	Dichlorvos	8141A	SS	27-90	34	0.1	mg/Kg
Pesticides	Dimethoate	8141A	SS	37-114	30	0.1	mg/Kg
Pesticides	Disulfoton	8141A	SS	50-100	22	0.1	mg/Kg
Pesticides	Epn	8141A	SS	55-116	27	0.1	mg/Kg
Pesticides	Ethoprop	8141A	SS	53-97	26	0.1	mg/Kg
Pesticides	Ethyl Parathion	8141A	SS	57-110	21	0.1	mg/Kg
Pesticides	Fensulfothion	8141A	SS	23-170	28	0.1	mg/Kg
Pesticides	Fenthion	8141A	SS	58-105	22	0.1	mg/Kg
Pesticides	Malathion	8141A	SS	57-108	21	0.1	mg/Kg
Pesticides	Merphos	8141A	SS	61-133	23	0.1	mg/Kg
Pesticides	Methyl Parathion	8141A	SS	58-106	22	0.1	mg/Kg
Pesticides	Mevinphos	8141A	SS	37-102	29	0.1	mg/Kg
Pesticides	Naled	8141A	SS	14-85	41	0.1	mg/Kg
Pesticides	Phorate	8141A	SS	46-105	24	0.1	mg/Kg
Pesticides	Ronnel	8141A	SS	44-99	26	0.1	mg/Kg
Pesticides	Stirophos	8141A	SS	58-114	22	0.1	mg/Kg
Pesticides	Sulfotep	8141A	SS	51-102	24	0.1	mg/Kg
Pesticides	Tepp	8141A	SS	10-96	41	0.1	mg/Kg
Pesticides	Tokuthion (Prothiofos)	8141A	SS	60-109	23	0.1	mg/Kg
Pesticides	Trichloronate	8141A	SS	58-105	23	0.1	mg/Kg

Table 12.3: QC Targets for Semi-Volatiles Accuracy (LCS), Precision and RL's
This table is subject to revision without notice

Class	Analyte	Method	Matrix	Accuracy (%)	Prec. (RPD)	RL	Unit
Pesticides	Alachlor	507	DW	70-130	25	0.0002	mg/L
Pesticides	Atrazine	507	DW	70-130	25	0.0001	mg/L
Pesticides	Butachlor	507	DW	70-130	25	0.0001	mg/L
Pesticides	Metolachlor	507	DW	70-130	25	0.0002	mg/L
Pesticides	Metribuzin	507	DW	70-130	25	0.0002	mg/L
Pesticides	Simazine	507	DW	70-130	25	7.00E-05	mg/L
Pesticides	Aldrin	508	DW	70-130	25	0.0005	mg/L
Pesticides	Dieldrin	508	DW	70-130	25	0.0005	mg/L
Pesticides	Endrin	508	DW	70-130	25	0.0005	mg/L
Pesticides	Gamma BHC	508	DW	70-130	25	0.0005	mg/L
Pesticides	Heptachlor	508	DW	70-130	25	0.0005	mg/L
Pesticides	Heptachlor Epoxide	508	DW	70-130	25	0.0005	mg/L
Pesticides	Hexachlorobenzene	508	DW	70-130	25	0.0005	mg/L
Pesticides	Methoxychlor	508	DW	70-130	25	0.0005	mg/L
Pesticides	4,4-DDD	608/8081A/B, 6630C	GW, WW	37-142	39	0.00005	mg/L
Pesticides	4,4-DDE	608/8081A/B, 6630C	GW, WW	33-124	37	0.00005	mg/L
Pesticides	4,4-DDT	608/8081A/B, 6630C	GW, WW	32-143	42	0.00005	mg/L
Pesticides	Aldrin	608/8081A/B, 6630C	GW, WW	25-115	45	0.00005	mg/L
Pesticides	Alpha BHC	608/8081A/B, 6630C	GW, WW	38-119	30	0.00005	mg/L
Pesticides	Beta BHC	608/8081A/B, 6630C	GW, WW	42-126	31	0.00005	mg/L
Pesticides	Chlordane	608/8081A/B, 6630C	GW, WW	-	-	0.005	mg/L
Pesticides	Delta BHC	608/8081A/B, 6630C	GW, WW	24-141	41	0.00005	mg/L
Pesticides	Dieldrin	608/8081A/B, 6630C	GW, WW	37-130	36	0.00005	mg/L
Pesticides	Endosulfan I	608/8081A/B, 6630C	GW, WW	37-125	35	0.00005	mg/L
Pesticides	Endosulfan II	608/8081A/B, 6630C	GW, WW	38-131	36	0.00005	mg/L
Pesticides	Endosulfan Sulfate	608/8081A/B, 6630C	GW, WW	38-131	37	0.00005	mg/L
Pesticides	Endrin	608/8081A/B, 6630C	GW, WW	37-126	37	0.00005	mg/L
Pesticides	Endrin Aldehyde	608/8081A/B, 6630C	GW, WW	24-154	36	0.00005	mg/L
Pesticides	Endrin Ketone	608/8081A/B, 6630C	GW, WW	37-139	36	0.00005	mg/L
Pesticides	Gamma BHC	608/8081A/B, 6630C	GW, WW	35-114	30	0.00005	mg/L
Pesticides	Heptachlor	608/8081A/B, 6630C	GW, WW	21-123	38	0.00005	mg/L
Pesticides	Heptachlor Epoxide	608/8081A/B, 6630C	GW, WW	38-121	33	0.00005	mg/L
Pesticides	Hexachlorobenzene	608/8081A/B, 6630C	GW, WW	28-115	29	0.00005	mg/L
Pesticides	Methoxychlor	608/8081A/B, 6630C	GW, WW	55-150	40	0.00005	mg/L
Pesticides	Toxaphene	608/8081A/B, 6630C	GW, WW	-	-	0.0005	mg/L

Table 12.3: QC Targets for Semi-Volatiles Accuracy (LCS), Precision and RL's
This table is subject to revision without notice

Class	Analyte	Method	Matrix	Accuracy (%)	Prec. (RPD)	RL	Unit
PCBs	PCB 1016	608, 6431B, 8082/A	GW, WW	46-126	34	0.0005	mg/L
PCBs	PCB 1221	608, 6431B, 8082/A	GW, WW	-	-	0.0005	mg/L
PCBs	PCB 1232	608, 6431B, 8082/A	GW, WW	-	-	0.0005	mg/L
PCBs	PCB 1242	608, 6431B, 8082/A	GW, WW	-	-	0.0005	mg/L
PCBs	PCB 1248	608, 6431B, 8082/A	GW, WW	-	-	0.0005	mg/L
PCBs	PCB 1254	608, 6431B, 8082/A	GW, WW	-	-	0.0005	mg/L
PCBs	PCB 1260	608, 6431B, 8082/A	GW, WW	46-126	34	0.0005	mg/L
PCBs	PCB 1016	8082/A	SS	62-131	22	0.017	mg/Kg
PCBs	PCB 1221	8082/A	SS	-	-	0.017	mg/Kg
PCBs	PCB 1232	8082/A	SS	-	-	0.017	mg/Kg
PCBs	PCB 1242	8082/A	SS	-	-	0.017	mg/Kg
PCBs	PCB 1248	8082/A	SS	-	-	0.017	mg/Kg
PCBs	PCB 1254	8082/A	SS	-	-	0.017	mg/Kg
PCBs	PCB 1260	8082/A	SS	62-131	22	0.017	mg/Kg
PCBs	PCB 1260	8082/A	SS	-	-	0.017	mg/Kg
Pesticides	4,4-DDD	8081A/B	SS	62-133	28	0.02	mg/Kg
Pesticides	4,4-DDE	8081A/B	SS	61-122	28	0.02	mg/Kg
Pesticides	4,4-DDT	8081A/B	SS	54-138	31	0.02	mg/Kg
Pesticides	Aldrin	8081A/B	SS	57-114	27	0.02	mg/Kg
Pesticides	Alpha BHC	8081A/B	SS	56-122	30	0.02	mg/Kg
Pesticides	Beta BHC	8081A/B	SS	67-122	23	0.02	mg/Kg
Pesticides	Delta BHC	8081A/B	SS	63-120	26	0.02	mg/Kg
Pesticides	Dieldrin	8081A/B	SS	63-124	26	0.02	mg/Kg
Pesticides	Endosulfan I	8081A/B	SS	63-119	26	0.02	mg/Kg
Pesticides	Endosulfan II	8081A/B	SS	63-123	28	0.02	mg/Kg
Pesticides	Endosulfan Sulfate	8081A/B	SS	60-124	30	0.02	mg/Kg
Pesticides	Endrin	8081A/B	SS	58-118	27	0.02	mg/Kg
Pesticides	Endrin Aldehyde	8081A/B	SS	50-136	32	0.02	mg/Kg
Pesticides	Endrin Ketone	8081A/B	SS	57-127	30	0.02	mg/Kg
Pesticides	Gamma BHC	8081A/B	SS	58-113	27	0.02	mg/Kg
Pesticides	Heptachlor	8081A/B	SS	56-116	29	0.02	mg/Kg
Pesticides	Heptachlor Epoxide	8081A/B	SS	63-118	26	0.02	mg/Kg

Table 12.3: QC Targets for Semi-Volatiles Accuracy (LCS), Precision and RL's
This table is subject to revision without notice

Class	Analyte	Method	Matrix	Accuracy (%)	Prec. (RPD)	RL	Unit
Pesticides	Hexachlorobenzene	8081A/B	SS	51-118	30	0.02	mg/Kg
Pesticides	Methoxychlor	8081A/B	SS	50-152	32	0.02	mg/Kg
Pesticides	Chlordane	8081A/B	SS	-	-	0.2	mg/Kg
Pesticides	Toxaphene	8081A/B	SS	-	-	0.4	mg/Kg
Herbicides	2,4,5-TP (SILVEX)	515.1	DW	70-130	25	0.0001	mg/L
Herbicides	2,4-D	515.1	DW	70-130	25	0.0001	mg/L
Herbicides	Dalapon	515.1	DW	70-130	25	0.001	mg/L
Herbicides	Dicamba	515.1	DW	70-130	25	0.0001	mg/L
Herbicides	Dinoseb	515.1	DW	70-130	25	0.0001	mg/L
Herbicides	Pentachlorophenol	515.1	DW	70-130	25	0.00004	mg/L
Herbicides	Picloram	515.1	DW	70-130	25	0.0001	mg/L
Herbicides	2,4,5-T	1658, 8151A, 6640C	GW, WW	30-136	31	0.002	mg/L
Herbicides	2,4,5-TP (SILVEX)	1658, 8151A, 6640C	GW, WW	33-134	30	0.002	mg/L
Herbicides	2,4-D	1658, 8151A, 6640C	GW, WW	24-127	27	0.002	mg/L
Herbicides	2,4-DB	1658, 8151A, 6640C	GW, WW	22-198	33	0.002	mg/L
Herbicides	Dalapon	1658, 8151A, 6640C	GW, WW	14-121	31	0.002	mg/L
Herbicides	Dicamba	1658, 8151A, 6640C	GW, WW	31-135	25	0.002	mg/L
Herbicides	Dichloroprop	1658, 8151A, 6640C	GW, WW	30-122	26	0.002	mg/L
Herbicides	Dinoseb	1658, 8151A, 6640C	GW, WW	28-183	38	0.002	mg/L
Herbicides	MCPA	1658, 8151A, 6640C	GW, WW	32-153	31	0.1	mg/L
Herbicides	MCPP	1658, 8151A, 6640C	GW, WW	42-133	29	0.1	mg/L
Herbicides	2,4,5-T	8151A	SS	40-122	27	0.07	mg/Kg
Herbicides	2,4,5-TP (SILVEX)	8151A	SS	36-125	26	0.07	mg/Kg
Herbicides	2,4-D	8151A	SS	33-119	28	0.07	mg/Kg
Herbicides	2,4-DB	8151A	SS	20-155	28	0.07	mg/Kg
Herbicides	Dalapon	8151A	SS	10-127	44	0.07	mg/Kg
Herbicides	Dicamba	8151A	SS	37-122	25	0.07	mg/Kg
Herbicides	Dichloroprop	8151A	SS	40-110	23	0.07	mg/Kg
Herbicides	Dinoseb	8151A	SS	10-155	50	0.07	mg/Kg
Herbicides	MCPA	8151A	SS	23-161	27	6.5	mg/Kg
Herbicides	MCPP	8151A	SS	30-148	24	6.5	mg/Kg
PAH	1-Methylnaphthalene	8310, 610, 6440B	GW, WW	31-95	32	0.0001	mg/L
PAH	2-Methylnaphthalene	8310, 610, 6440B	GW, WW	30-97	34	0.0001	mg/L
PAH	Acenaphthene	8310, 610, 6440B	GW, WW	32-120	29	0.0001	mg/L
PAH	Acenaphthylene	8310, 610, 6440B	GW, WW	41-112	30	0.0001	mg/L

Table 12.3: QC Targets for Semi-Volatiles Accuracy (LCS), Precision and RL's
This table is subject to revision without notice

Class	Analyte	Method	Matrix	Accuracy (%)	Prec. (RPD)	RL	Unit
PAH	Anthracene	8310, 610, 6440B	GW, WW	48-122	26	0.0001	mg/L
PAH	Benzo(a)Anthracene	8310, 610, 6440B	GW, WW	52-122	22	0.0001	mg/L
PAH	Benzo(a)Pyrene	8310, 610, 6440B	GW, WW	45-120	24	0.0001	mg/L
PAH	Benzo(b)Fluoranthene	8310, 610, 6440B	GW, WW	46-118	24	0.0001	mg/L
PAH	Benzo(g,h,i)Perylene	8310, 610, 6440B	GW, WW	31-110	31	0.0001	mg/L
PAH	Benzo(k)Fluoranthene	8310, 610, 6440B	GW, WW	45-112	24	0.0001	mg/L
PAH	Chrysene	8310, 610, 6440B	GW, WW	53-126	23	0.0001	mg/L
PAH	Dibenz(a,h)Anthracene	8310, 610, 6440B	GW, WW	26-113	35	0.0001	mg/L
PAH	Fluoranthene	8310, 610, 6440B	GW, WW	52-125	23	0.0001	mg/L
PAH	Fluorene	8310, 610, 6440B	GW, WW	45-117	27	0.0001	mg/L
PAH	Indeno(1,2,3-cd)Pyrene	8310, 610, 6440B	GW, WW	40-113	29	0.0001	mg/L
PAH	Naphthalene	8310, 610, 6440B	GW, WW	22-105	37	0.0001	mg/L
PAH	Phenanthrene	8310, 610, 6440B	GW, WW	48-122	26	0.0001	mg/L
PAH	Pyrene	8310, 610, 6440B	GW, WW	53-128	24	0.0001	mg/L
PAH	1-Methylnaphthalene	8310	SS	18-102	42	0.02	mg/Kg
PAH	2-Methylnaphthalene	8310	SS	18-107	43	0.02	mg/Kg
PAH	Acenaphthene	8310	SS	22-139	36	0.02	mg/Kg
PAH	Acenaphthylene	8310	SS	33-118	35	0.02	mg/Kg
PAH	Anthracene	8310	SS	65-119	20	0.02	mg/Kg
PAH	Benzo(a)Anthracene	8310	SS	77-123	20	0.02	mg/Kg
PAH	Benzo(a)Pyrene	8310	SS	68-118	20	0.02	mg/Kg
PAH	Benzo(b)Fluoranthene	8310	SS	68-110	20	0.02	mg/Kg
PAH	Benzo(g,h,i)Perylene	8310	SS	57-118	28	0.02	mg/Kg
PAH	Benzo(k)Fluoranthene	8310	SS	70-124	20	0.02	mg/Kg
PAH	Chrysene	8310	SS	79-125	20	0.02	mg/Kg
PAH	Dibenz(a,h)Anthracene	8310	SS	64-121	25	0.02	mg/Kg
PAH	Fluoranthene	8310	SS	76-121	20	0.02	mg/Kg
PAH	Fluorene	8310	SS	47-126	28	0.02	mg/Kg
PAH	Indeno(1,2,3-cd)Pyrene	8310	SS	62-121	26	0.02	mg/Kg
PAH	Naphthalene	8310	SS	11-104	49	0.02	mg/Kg
PAH	Phenanthrene	8310	SS	63-118	20	0.02	mg/Kg
PAH	Pyrene	8310	SS	77-125	20	0.02	mg/Kg
BNA	1,2,4,5-Tetrachlorobenzene	8270C/D	GW,WW	39-116	33	0.01	mg/L
BNA	1,2,4-Trichlorobenzene	8270C/D, 625, SM6410B	GW,WW	26-103	38	0.01	mg/L
BNA	1,4-Naphthoquinone	8270C/D	GW,WW	50 - 150*	25*	0.01	mg/L

Table 12.3: QC Targets for Semi-Volatiles Accuracy (LCS), Precision and RL's
This table is subject to revision without notice

Class	Analyte	Method	Matrix	Accuracy (%)	Prec. (RPD)	RL	Unit
BNA	1-Naphthylamine	8270C/D	GW,WW	50 - 150*	25*	0.01	mg/L
BNA	2,3,4,6-Tetrachlorophenol	8270C/D	GW,WW	50 - 150*	25*	0.01	mg/L
BNA	2,4,5-Trichlorophenol	8270C/D	GW,WW	48-120	29	0.01	mg/L
BNA	2,4,6-Trichlorophenol	8270C/D, 625, SM6410B	GW,WW	49-118	28	0.01	mg/L
BNA	2,4-Dichlorophenol	8270C/D, 625, SM6410B	GW,WW	46-115	28	0.01	mg/L
BNA	2,4-Dimethylphenol	8270C/D, 625, SM6410B	GW,WW	40-124	36	0.01	mg/L
BNA	2,4-Dinitrophenol	8270C/D, 625, SM6410B	GW,WW	10-125	50	0.01	mg/L
BNA	2,4-Dinitrotoluene	8270C/D, 625, SM6410B	GW,WW	56-128	24	0.01	mg/L
BNA	2,6-Dichlorophenol	8270C/D	GW,WW	50 - 150*	25*	0.01	mg/L
BNA	2,6-Dinitrotoluene	8270C/D, 625, SM6410B	GW,WW	56-121	23	0.01	mg/L
BNA	2-Acetylaminofluorene	8270C/D	GW,WW	50 - 150*	25*	0.01	mg/L
BNA	2-Chloronaphthalene	8270C/D, 625, SM6410B	GW,WW	44-110	30	0.001	mg/L
BNA	2-Chlorophenol	8270C/D, 625, SM6410B	GW,WW	38-114	36	0.01	mg/L
BNA	2-Methylnaphthalene	8270C/D	GW,WW	28-122	36	0.001	mg/L
BNA	2-Methylphenol	8270C/D	GW,WW	42-99	26	0.01	mg/L
BNA	2-Naphthylamine	8270C/D	GW,WW	50 - 150*	25*	0.01	mg/L
BNA	2-Nitroaniline	8270C/D	GW,WW	55-124	22	0.01	mg/L
BNA	2-Nitrophenol	8270C/D, 625, SM6410B	GW,WW	35-118	35	0.01	mg/L
BNA	3,3-Dichlorobenzidine	8270C/D, 625, SM6410B	GW,WW	46-145	31	0.01	mg/L
BNA	3,3-Dimethylbenzidine	8270C/D	GW,WW	50 - 150*	25*	0.01	mg/L
BNA	3-Methylcholanthrene	8270C/D	GW,WW	50 - 150*	25*	0.01	mg/L
BNA	3-Nitroaniline	8270C/D	GW,WW	39-141	32	0.01	mg/L
BNA	4,6-Dinitro-2-Methylphenol	8270C/D, 625, SM6410B	GW,WW	24-119	50	0.01	mg/L
BNA	4-Aminobiphenyl	8270C/D	GW,WW	50 - 150*	25*	0.01	mg/L
BNA	4-Bromophenyl-Phenylether	8270C/D, 625, SM6410B	GW,WW	45-105	26	0.01	mg/L
BNA	4-Chloro-3-Methylphenol	8270C/D, 625, SM6410B	GW,WW	47-116	22	0.01	mg/L
BNA	4-Chloroaniline	8270C/D	GW,WW	27-151	36	0.01	mg/L
BNA	4-Chlorophenyl-Phenylether	8270C/D, 625, SM6410B	GW,WW	49-116	26	0.01	mg/L
BNA	4-Nitroaniline	8270C/D	GW,WW	43-144	34	0.01	mg/L
BNA	4-Nitrophenol	8270C/D, 625, SM6410B	GW,WW	10-66	37	0.01	mg/L
BNA	5-Nitro-O-Toluidine	8270C/D	GW,WW	50 - 150*	25*	0.01	mg/L
BNA	Acenaphthene	8270C/D, 625, SM6410B	GW,WW	48-110	26	0.001	mg/L
BNA	Acenaphthylene	8270C/D, 625, SM6410B	GW,WW	48-113	28	0.001	mg/L

Table 12.3: QC Targets for Semi-Volatiles Accuracy (LCS), Precision and RL's
This table is subject to revision without notice

Class	Analyte	Method	Matrix	Accuracy (%)	Prec. (RPD)	RL	Unit
BNA	Acetophenone	8270C/D	GW,WW	35-98	38	0.01	mg/L
BNA	Aniline	8270C/D	GW,WW	0-159	50	0.01	mg/L
BNA	Anthracene	8270C/D, 625, SM6410B	GW,WW	55-127	24	0.001	mg/L
BNA	Azobenzene	8270C/D	GW,WW	50-129	28	0.01	mg/L
BNA	Benzo(a)Anthracene	8270C/D, 625, SM6410B	GW,WW	57-115	20	0.001	mg/L
BNA	Benzo(a)Pyrene	8270C/D, 625, SM6410B	GW,WW	63-125	22	0.001	mg/L
BNA	Benzo(b)Fluoranthene	8270C/D, 625, SM6410B	GW,WW	50-123	32	0.001	mg/L
BNA	Benzo(g,h,i)Perylene	8270C/D, 625, SM6410B	GW,WW	39-143	31	0.001	mg/L
BNA	Benzo(k)Fluoranthene	8270C/D, 625, SM6410B	GW,WW	45-126	37	0.001	mg/L
BNA	Benzyl Alcohol	8270C/D	GW,WW	33-104	32	0.01	mg/L
BNA	Benzylbutyl Phthalate	8270C/D, 625, SM6410B	GW,WW	22-154	29	0.01	mg/L
BNA	Bis(2-Chlorethoxy)Methane	8270C/D, 625, SM6410B	GW,WW	42-116	38	0.01	mg/L
BNA	Bis(2-Chloroethyl)Ether	8270C/D, 625, SM6410B	GW,WW	26-115	50	0.01	mg/L
BNA	Bis(2-Chloroisopropyl)Ether	8270C/D, 625, SM6410B	GW,WW	32-115	47	0.01	mg/L
BNA	Bis(2-Ethylhexyl)Phthalate	8270C/D, 625, SM6410B	GW,WW	47-143	24	0.001	mg/L
BNA	Chlorobenzilate	8270C/D	GW,WW	50 - 150*	25*	0.01	mg/L
BNA	Chrysene	8270C/D, 625, SM6410B	GW,WW	58-113	21	0.001	mg/L
BNA	Diallate	8270C/D	GW,WW	50 - 150*	25*	0.01	mg/L
BNA	Dibenz(a,h)Anthracene	8270C/D, 625, SM6410B	GW,WW	39-144	30	0.001	mg/L
BNA	Dibenzofuran	8270C/D	GW,WW	50-121	26	0.01	mg/L
BNA	Diethyl Phthalate	8270C/D, 625, SM6410B	GW,WW	36-128	27	0.001	mg/L
BNA	Dimethoate	8270C/D	GW,WW	50 - 150*	25*	0.01	mg/L
BNA	Dimethyl Phthalate	8270C/D, 625, SM6410B	GW,WW	10-135	33	0.001	mg/L
BNA	Dimethylbenz (a) Anthracene	8270C/D	GW,WW	50 - 150*	25*	0.01	mg/L
BNA	Di-N-Butyl Phthalate	8270C/D, 625, SM6410B	GW,WW	51-131	22	0.001	mg/L
BNA	Di-N-Octyl Phthalate	8270C/D, 625, SM6410B	GW,WW	51-138	22	0.001	mg/L
BNA	Dinoseb	8270C/D	GW,WW	50 - 150*	25*	0.01	mg/L
BNA	Diphenylamine	8270C/D	GW,WW	50 - 150*	25*	0.01	mg/L
BNA	Disulfoton	8270C/D	GW,WW	50 - 150*	25*	0.01	mg/L
BNA	Ethyl Methanesulfonate	8270C/D	GW,WW	50 - 150*	25*	0.01	mg/L
BNA	Famphur	8270C/D	GW,WW	50 - 150*	25*	0.01	mg/L
BNA	Fluoranthene	8270C/D, 625, SM6410B	GW,WW	53-119	28	0.001	mg/L
BNA	Fluorene	8270C/D, 625, SM6410B	GW,WW	49-116	25	0.01	mg/L

Table 12.3: QC Targets for Semi-Volatiles Accuracy (LCS), Precision and RL's
This table is subject to revision without notice

Class	Analyte	Method	Matrix	Accuracy (%)	Prec. (RPD)	RL	Unit
BNA	Hexachloro-1,3-Butadiene	8270C/D, 625, SM6410B	GW,WW	21-116	50	0.01	mg/L
BNA	Hexachlorobenzene	8270C/D, 625, SM6410B	GW,WW	51-121	23	0.001	mg/L
BNA	Hexachlorocyclopentadiene	8270C/D, 625, SM6410B	GW,WW	4-126	50	0.01	mg/L
BNA	Hexachloroethane	8270C/D, 625, SM6410B	GW,WW	15-109	50	0.01	mg/L
BNA	Hexachloropropene	8270C/D	GW,WW	50 - 150*	25*	0.01	mg/L
BNA	Indeno(1,2,3-cd)Pyrene	8270C/D, 625, SM6410B	GW,WW	40-143	30	0.001	mg/L
BNA	Isodrin	8270C/D	GW,WW	50 - 150*	25*	0.01	mg/L
BNA	Isophorone	8270C/D, 625, SM6410B	GW,WW	48-126	31	0.01	mg/L
BNA	Isosafrole	8270C/D	GW,WW	50 - 150*	25*	0.01	mg/L
BNA	Kepone	8270C/D	GW,WW	50 - 150*	25*	0.01	mg/L
BNA	m&p-Cresol	8270C/D	GW,WW	36-102	31	0.01	mg/L
BNA	M-Dinitrobenzene	8270C/D	GW,WW	50 - 150*	25*	0.01	mg/L
BNA	Methapyrilene	8270C/D	GW,WW	50 - 150*	25*	0.01	mg/L
BNA	Methyl Methanesulfonate	8270C/D	GW,WW	50 - 150*	25*	0.01	mg/L
BNA	Methyl Parathion	8270C/D	GW,WW	50 - 150*	25*	0.01	mg/L
BNA	Naphthalene	8270C/D, 625, SM6410B	GW,WW	29-103	45	0.001	mg/L
BNA	Nitrobenzene	8270C/D, 625, SM6410B	GW,WW	31-105	43	0.01	mg/L
BNA	N-Nitrosodiethylamine	8270C/D	GW,WW	50 - 150*	25*	0.01	mg/L
BNA	N-Nitrosodimethylamine	8270C/D, 625, SM6410B	GW,WW	Nov-69	50	0.01	mg/L
BNA	N-Nitrosodi-N-Butylamine	8270C/D	GW,WW	50 - 150*	25*	0.01	mg/L
BNA	N-Nitrosodi-N-Propylamine	8270C/D, 625, SM6410B	GW,WW	47-122	33	0.01	mg/L
BNA	N-Nitrosodiphenylamine	8270C/D, 625, SM6410B	GW,WW	59-143	23	0.01	mg/L
BNA	N-Nitrosomethylethylamine	8270C/D	GW,WW	50 - 150*	25*	0.01	mg/L
BNA	N-Nitrosopiperidine	8270C/D	GW,WW	50 - 150*	25*	0.01	mg/L
BNA	N-Nitrosopyrrolidine	8270C/D	GW,WW	50 - 150*	25*	0.01	mg/L
BNA	O,O,O-Triethyl Phosphorothioate	8270C/D	GW,WW	50 - 150*	25*	0.01	mg/L
BNA	O-Toluidine	8270C/D	GW,WW	50 - 150*	25*	0.01	mg/L
BNA	P-(Dimethylamino) Azobenzene	8270C/D	GW,WW	50 - 150*	25*	0.01	mg/L
BNA	Parathion	8270C/D	GW,WW	50 - 150*	25*	0.01	mg/L
BNA	Pentachlorobenzene	8270C/D	GW,WW	50 - 150*	25*	0.01	mg/L
BNA	Pentachloronitrobenzene	8270C/D	GW,WW	50 - 150*	25*	0.01	mg/L
BNA	Pentachlorophenol	8270C/D, 625, SM6410B	GW,WW	20-122	50	0.001	mg/L
BNA	Phenacetin	8270C/D	GW,WW	50 - 150*	25*	0.01	mg/L
BNA	Phenanthrene	8270C/D, 625, SM6410B	GW,WW	54-112	22	0.001	mg/L

Table 12.3: QC Targets for Semi-Volatiles Accuracy (LCS), Precision and RL's
This table is subject to revision without notice

Class	Analyte	Method	Matrix	Accuracy (%)	Prec. (RPD)	RL	Unit
BNA	Phenol	8270C/D, 625, SM6410B	GW,WW	17-56	33	0.01	mg/L
BNA	Phorate	8270C/D	GW,WW	50 - 150*	25*	0.01	mg/L
BNA	P-Phenylenediamine	8270C/D	GW,WW	50 - 150*	25*	0.01	mg/L
BNA	Pronamide	8270C/D	GW,WW	50 - 150*	25*	0.01	mg/L
BNA	Pyrene	8270C/D, 625, SM6410B	GW,WW	46-130	28	0.001	mg/L
BNA	Pyridine	8270C/D	GW,WW	7-48	50	0.01	mg/L
BNA	Safrole	8270C/D	GW,WW	50 - 150*	25*	0.01	mg/L
BNA	Sym-Trinitrobenzene	8270C/D	GW,WW	50 - 150*	25*	0.01	mg/L
BNA	Thionazin	8270C/D	GW,WW	50 - 150*	25*	0.01	mg/L
BNA	Triethyl Phosphorothioate	8270C/D	GW,WW	50 - 150*	25*	0.01	mg/L
BNA	1,2,4,5-Tetrachlorobenzene	8270C/D	SS	51-112	21	0.33	mg/Kg
BNA	1,2,4-Trichlorobenzene	8270C/D, 625, SM6410B	SS	46-99	24	0.33	mg/Kg
BNA	1,4-Naphthoquinone	8270C/D	SS	50 - 150*	25*	0.33	mg/Kg
BNA	1-Naphthylamine	8270C/D	SS	50 - 150*	25*	0.33	mg/Kg
BNA	2,3,4,6-Tetrachlorophenol	8270C/D	SS	50 - 150*	25*	0.33	mg/Kg
BNA	2,4,5-Trichlorophenol	8270C/D	SS	53-110	21	0.33	mg/Kg
BNA	2,4,6-Trichlorophenol	8270C/D, 625, SM6410B	SS	56-109	24	0.33	mg/Kg
BNA	2,4-Dichlorophenol	8270C/D, 625, SM6410B	SS	54-107	21	0.33	mg/Kg
BNA	2,4-Dimethylphenol	8270C/D, 625, SM6410B	SS	58-119	23	0.33	mg/Kg
BNA	2,4-Dinitrophenol	8270C/D, 625, SM6410B	SS	16-130	45	0.33	mg/Kg
BNA	2,4-Dinitrotoluene	8270C/D, 625, SM6410B	SS	53-120	23	0.33	mg/Kg
BNA	2,6-Dichlorophenol	8270C/D	SS	50 - 150*	25*	0.33	mg/Kg
BNA	2,6-Dinitrotoluene	8270C/D, 625, SM6410B	SS	56-113	22	0.33	mg/Kg
BNA	2-Acetylaminofluorene	8270C/D	SS	50 - 150*	25*	0.33	mg/Kg
BNA	2-Chloronaphthalene	8270C/D, 625, SM6410B	SS	55-103	20	0.033	mg/Kg
BNA	2-Chlorophenol	8270C/D, 625, SM6410B	SS	52-108	24	0.33	mg/Kg
BNA	2-Methylnaphthalene	8270C/D	SS	52-107	21	0.033	mg/Kg
BNA	2-Methylphenol	8270C/D	SS	58-116	22	0.33	mg/Kg
BNA	2-Naphthylamine	8270C/D	SS	50 - 150*	25*	0.33	mg/Kg
BNA	2-Nitroaniline	8270C/D	SS	54-116	24	0.33	mg/Kg
BNA	2-Nitrophenol	8270C/D, 625, SM6410B	SS	38-110	24	0.33	mg/Kg
BNA	2-Picoline	8270C/D	SS	50 - 150*	25*	0.33	mg/Kg
BNA	3,3-Dichlorobenzidine	8270C/D, 625, SM6410B	SS	24-123	35	0.33	mg/Kg
BNA	3,3-Dimethylbenzidine	8270C/D	SS	50 - 150*	25*	0.33	mg/Kg

Table 12.3: QC Targets for Semi-Volatiles Accuracy (LCS), Precision and RL's
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Class	Analyte	Method	Matrix	Accuracy (%)	Prec. (RPD)	RL	Unit
BNA	3-Methylcholanthrene	8270C/D	SS	50 - 150*	25*	0.33	mg/Kg
BNA	3-Nitroaniline	8270C/D	SS	17-135	33	0.33	mg/Kg
BNA	4,6-Dinitro-2-Methylphenol	8270C/D, 625, SM6410B	SS	34-111	33	0.33	mg/Kg
BNA	4-Aminobiphenyl	8270C/D	SS	50 - 150*	25*	0.33	mg/Kg
BNA	4-Bromophenyl-Phenylether	8270C/D, 625, SM6410B	SS	47-98	23	0.33	mg/Kg
BNA	4-Chloro-3-Methylphenol	8270C/D, 625, SM6410B	SS	54-116	23	0.33	mg/Kg
BNA	4-Chloroaniline	8270C/D	SS	18-130	31	0.33	mg/Kg
BNA	4-Chlorophenyl-Phenylether	8270C/D, 625, SM6410B	SS	55-106	22	0.33	mg/Kg
BNA	4-Nitroaniline	8270C/D	SS	16-133	37	0.33	mg/Kg
BNA	4-Nitrophenol	8270C/D, 625, SM6410B	SS	34-123	36	0.33	mg/Kg
BNA	4-Nitroquinoline 1-Oxide	8270C/D	SS	50 - 150*	25*	0.33	mg/Kg
BNA	5-Nitro-O-Toluidine	8270C/D	SS	50 - 150*	25*	0.33	mg/Kg
BNA	A,A-Dimethylphenethylamine	8270C/D	SS	50 - 150*	25*	0.33	mg/Kg
BNA	Acenaphthene	8270C/D, 625, SM6410B	SS	54-102	20	0.033	mg/Kg
BNA	Acenaphthylene	8270C/D, 625, SM6410B	SS	56-104	20	0.033	mg/Kg
BNA	Acetochlor	8270C/D	SS	50 - 150*	25*	0.33	mg/Kg
BNA	Acetophenone	8270C/D	SS	42-92	22	0.33	mg/Kg
BNA	Alachlor	8270C/D	SS	50 - 150*	25*	0.33	mg/Kg
BNA	Ametryn	8270C/D	SS	50 - 150*	25*	0.33	mg/Kg
BNA	Aniline	8270C/D	SS	0-157	33	0.33	mg/Kg
BNA	Anthracene	8270C/D, 625, SM6410B	SS	57-112	21	0.033	mg/Kg
BNA	Aramite	8270C/D	SS	50 - 150*	25*	0.33	mg/Kg
BNA	Atraton	8270C/D	SS	50 - 150*	25*	0.33	mg/Kg
BNA	Atrazine	8270C/D	SS	50 - 150*	25*	0.33	mg/Kg
BNA	Azobenzene	8270C/D	SS	55-118	24	0.33	mg/Kg
BNA	Benfluralin	8270C/D	SS	50 - 150*	25*	0.33	mg/Kg
BNA	Benzidine	8270C/D, 625, SM6410B	SS	0-13	50	0.33	mg/Kg
BNA	Benzo(a)Anthracene	8270C/D, 625, SM6410B	SS	55-105	21	0.033	mg/Kg
BNA	Benzo(a)Pyrene	8270C/D, 625, SM6410B	SS	59-114	22	0.033	mg/Kg
BNA	Benzo(b)Fluoranthene	8270C/D, 625, SM6410B	SS	44-116	33	0.033	mg/Kg
BNA	Benzo(g,h,i)Perylene	8270C/D, 625, SM6410B	SS	41-127	29	0.033	mg/Kg
BNA	Benzo(k)Fluoranthene	8270C/D, 625, SM6410B	SS	36-119	37	0.033	mg/Kg
BNA	Benzyl Alcohol	8270C/D	SS	53-115	23	0.33	mg/Kg
BNA	Benzylbutyl Phthalate	8270C/D, 625, SM6410B	SS	57-130	27	0.33	mg/Kg

Table 12.3: QC Targets for Semi-Volatiles Accuracy (LCS), Precision and RL's
This table is subject to revision without notice

Class	Analyte	Method	Matrix	Accuracy (%)	Prec. (RPD)	RL	Unit
BNA	Bis(2-Chlorethoxy)Methane	8270C/D, 625, SM6410B	SS	52-107	21	0.33	mg/Kg
BNA	Bis(2-Chloroethyl)Ether	8270C/D, 625, SM6410B	SS	38-115	28	0.33	mg/Kg
BNA	Bis(2-Chloroisopropyl)Ether	8270C/D, 625, SM6410B	SS	49-106	25	0.33	mg/Kg
BNA	Bis(2-Ethylhexyl)Phthalate	8270C/D, 625, SM6410B	SS	50-130	29	0.33	mg/Kg
BNA	Bromacil	8270C/D	SS	50 - 150*	25*	0.33	mg/Kg
BNA	Chlorobenzilate	8270C/D	SS	50 - 150*	25*	0.33	mg/Kg
BNA	Chlorpropham	8270C/D	SS	50 - 150*	25*	0.33	mg/Kg
BNA	Chrysene	8270C/D, 625, SM6410B	SS	54-103	23	0.033	mg/Kg
BNA	Cyanazine	8270C/D	SS	50 - 150*	25*	0.33	mg/Kg
BNA	Dacthal	8270C/D	SS	50 - 150*	25*	0.33	mg/Kg
BNA	Diallate	8270C/D	SS	50 - 150*	25*	0.33	mg/Kg
BNA	Dibenz(a,h)Anthracene	8270C/D, 625, SM6410B	SS	42-128	28	0.033	mg/Kg
BNA	Dibenzofuran	8270C/D	SS	56-111	21	0.33	mg/Kg
BNA	Dichlobenil	8270C/D	SS	50 - 150*	25*	0.33	mg/Kg
BNA	Diethyl Phthalate	8270C/D, 625, SM6410B	SS	57-110	20	0.33	mg/Kg
BNA	Dimethenamid	8270C/D	SS	50 - 150*	25*	0.33	mg/Kg
BNA	Dimethoate	8270C/D	SS	50 - 150*	25*	0.33	mg/Kg
BNA	Dimethyl Phthalate	8270C/D, 625, SM6410B	SS	57-108	20	0.33	mg/Kg
BNA	Dimethylbenz (a) Anthracene	8270C/D	SS	50 - 150*	25*	0.33	mg/Kg
BNA	Di-N-Butyl Phthalate	8270C/D, 625, SM6410B	SS	56-121	22	0.33	mg/Kg
BNA	Di-N-Octyl Phthalate	8270C/D, 625, SM6410B	SS	50-128	26	0.33	mg/Kg
BNA	Dinoseb	8270C/D	SS	50 - 150*	25*	0.33	mg/Kg
BNA	Diphenylamine	8270C/D	SS	50 - 150*	25*	0.33	mg/Kg
BNA	Disulfoton	8270C/D	SS	50 - 150*	25*	0.33	mg/Kg
BNA	Eptc	8270C/D	SS	50 - 150*	25*	0.33	mg/Kg
BNA	Ethalfluralin	8270C/D	SS	50 - 150*	25*	0.33	mg/Kg
BNA	Ethofumesate	8270C/D	SS	50 - 150*	25*	0.33	mg/Kg
BNA	Ethyl Methanesulfonate	8270C/D	SS	50 - 150*	25*	0.33	mg/Kg
BNA	Ethyl Parathion	8270C/D	SS	50 - 150*	25*	0.33	mg/Kg
BNA	Famphur	8270C/D	SS	50 - 150*	25*	0.33	mg/Kg
BNA	Fluoranthene	8270C/D, 625, SM6410B	SS	51-109	26	0.033	mg/Kg
BNA	Fluorene	8270C/D, 625, SM6410B	SS	53-106	20	0.033	mg/Kg
BNA	Hexachloro-1,3-Butadiene	8270C/D, 625, SM6410B	SS	46-110	25	0.33	mg/Kg
BNA	Hexachlorobenzene	8270C/D, 625, SM6410B	SS	51-117	24	0.33	mg/Kg

Table 12.3: QC Targets for Semi-Volatiles Accuracy (LCS), Precision and RL's
This table is subject to revision without notice

Class	Analyte	Method	Matrix	Accuracy (%)	Prec. (RPD)	RL	Unit
BNA	Hexachlorocyclopentadiene	8270C/D, 625, SM6410B	SS	21-127	40	0.33	mg/Kg
BNA	Hexachloroethane	8270C/D, 625, SM6410B	SS	43-104	27	0.33	mg/Kg
BNA	Hexachlorophene	8270C/D	SS	50 - 150*	25*	0.33	mg/Kg
BNA	Hexachloropropene	8270C/D	SS	50 - 150*	25*	0.33	mg/Kg
BNA	Hexazinone	8270C/D	SS	50 - 150*	25*	0.33	mg/Kg
BNA	Indeno(1,2,3-cd)Pyrene	8270C/D, 625, SM6410B	SS	42-127	28	0.033	mg/Kg
BNA	Isodrin	8270C/D	SS	50 - 150*	25*	0.33	mg/Kg
BNA	Isophorone	8270C/D, 625, SM6410B	SS	56-116	21	0.33	mg/Kg
BNA	Isosafrole	8270C/D	SS	50 - 150*	25*	0.33	mg/Kg
BNA	Kepone	8270C/D	SS	50 - 150*	25*	0.33	mg/Kg
BNA	m&p-Cresol	8270C/D	SS	60-136	29	0.33	mg/Kg
BNA	M-Dinitrobenzene	8270C/D	SS	50 - 150*	25*	0.33	mg/Kg
BNA	Methapyrilene	8270C/D	SS	50 - 150*	25*	0.33	mg/Kg
BNA	Methyl Methanesulfonate	8270C/D	SS	50 - 150*	25*	0.33	mg/Kg
BNA	Methyl Parathion	8270C/D	SS	50 - 150*	25*	0.33	mg/Kg
BNA	Metolachlor	8270C/D	SS	50 - 150*	25*	0.33	mg/Kg
BNA	Metribuzin	8270C/D	SS	50 - 150*	25*	0.33	mg/Kg
BNA	Naphthalene	8270C/D, 625, SM6410B	SS	46-97	23	0.033	mg/Kg
BNA	Napropamide	8270C/D	SS	50 - 150*	25*	0.33	mg/Kg
BNA	Nitrobenzene	8270C/D, 625, SM6410B	SS	46-102	23	0.33	mg/Kg
BNA	N-Nitrosodiethylamine	8270C/D	SS	50 - 150*	25*	0.33	mg/Kg
BNA	N-Nitrosodimethylamine	8270C/D, 625, SM6410B	SS	35-111	35	0.33	mg/Kg
BNA	N-Nitrosodi-N-Butylamine	8270C/D	SS	50 - 150*	25*	0.33	mg/Kg
BNA	N-Nitrosodi-N-Propylamine	8270C/D, 625, SM6410B	SS	54-113	21	0.33	mg/Kg
BNA	N-Nitrosodiphenylamine	8270C/D, 625, SM6410B	SS	66-126	22	0.33	mg/Kg
BNA	N-Nitrosomethylethylamine	8270C/D	SS	50 - 150*	25*	0.33	mg/Kg
BNA	N-Nitrosomorpholine	8270C/D	SS	50 - 150*	25*	0.33	mg/Kg
BNA	N-Nitrosopiperidine	8270C/D	SS	50 - 150*	25*	0.33	mg/Kg
BNA	N-Nitrosopyrrolidine	8270C/D	SS	50 - 150*	25*	0.33	mg/Kg
BNA	Norflurazon	8270C/D	SS	50 - 150*	25*	0.33	mg/Kg
BNA	O,O,O-Triethyl Phosphorothioate	8270C/D	SS	50 - 150*	25*	0.33	mg/Kg
BNA	O-Toluidine	8270C/D	SS	50 - 150*	25*	0.33	mg/Kg
BNA	Oxadiazon	8270C/D	SS	50 - 150*	25*	0.33	mg/Kg
BNA	Oxyfluorfen	8270C/D	SS	50 - 150*	25*	0.33	mg/Kg
BNA	P-(Dimethylamino) Azobenzene	8270C/D	SS	50 - 150*	25*	0.33	mg/Kg

Table 12.3: QC Targets for Semi-Volatiles Accuracy (LCS), Precision and RL's
This table is subject to revision without notice

Class	Analyte	Method	Matrix	Accuracy (%)	Prec. (RPD)	RL	Unit
BNA	Parathion	8270C/D	SS	50 - 150*	25*	0.33	mg/Kg
BNA	Pendimethalin	8270C/D	SS	50 - 150*	25*	0.33	mg/Kg
BNA	Pentachlorobenzene	8270C/D	SS	50 - 150*	25*	0.33	mg/Kg
BNA	Pentachloroethane	8270C/D	SS	50 - 150*	25*	0.33	mg/Kg
BNA	Pentachloronitrobenzene	8270C/D	SS	50 - 150*	25*	0.33	mg/Kg
BNA	Pentachlorophenol	8270C/D, 625, SM6410B	SS	37-118	28	0.33	mg/Kg
BNA	Phenacetin	8270C/D	SS	50 - 150*	25*	0.33	mg/Kg
BNA	Phenanthrene	8270C/D, 625, SM6410B	SS	56-102	20	0.033	mg/Kg
BNA	Phenol	8270C/D, 625, SM6410B	SS	55-115	22	0.33	mg/Kg
BNA	Phorate	8270C/D	SS	50 - 150*	25*	0.33	mg/Kg
BNA	P-Phenylenediamine	8270C/D	SS	50 - 150*	25*	0.33	mg/Kg
BNA	Prometon	8270C/D	SS	50 - 150*	25*	0.33	mg/Kg
BNA	Prometryn	8270C/D	SS	50 - 150*	25*	0.33	mg/Kg
BNA	Pronamide	8270C/D	SS	50 - 150*	25*	0.33	mg/Kg
BNA	Pronamide	8270C/D	SS	50 - 150*	25*	0.33	mg/Kg
BNA	Propachlor	8270C/D	SS	50 - 150*	25*	0.33	mg/Kg
BNA	Propazine	8270C/D	SS	50 - 150*	25*	0.33	mg/Kg
BNA	Pyrene	8270C/D, 625, SM6410B	SS	53-111	26	0.033	mg/Kg
BNA	Pyridine	8270C/D	SS	22-86	41	0.33	mg/Kg
BNA	Safrole	8270C/D	SS	50 - 150*	25*	0.33	mg/Kg
BNA	Simazine	8270C/D	SS	50 - 150*	25*	0.33	mg/Kg
BNA	Sulfotep	8270C/D	SS	50 - 150*	25*	0.33	mg/Kg
BNA	Sym-Trinitrobenzene	8270C/D	SS	50 - 150*	25*	0.33	mg/Kg
BNA	Terbacil	8270C/D	SS	50 - 150*	25*	0.33	mg/Kg
BNA	Terbuthylazine	8270C/D	SS	50 - 150*	25*	0.33	mg/Kg
BNA	Terbutryn	8270C/D	SS	50 - 150*	25*	0.33	mg/Kg
BNA	Thionazin	8270C/D	SS	50 - 150*	25*	0.33	mg/Kg
BNA	Triallate	8270C/D	SS	50 - 150*	25*	0.33	mg/Kg
BNA	Triethyl Phosphorothioate	8270C/D	SS	50 - 150*	25*	0.33	mg/Kg
BNA	Trifluralin	8270C/D	SS	50 - 150*	25*	0.33	mg/Kg
Explosives	1,3,5-Trinitrobenzene	8330A/B	SS	83-126	20	0.5	mg/Kg
Explosives	1,3-Dinitrobenzene	8330A/B	SS	75-111	20	0.5	mg/Kg
Explosives	2,4,6-Trinitrotoluene	8330A/B	SS	34-186	20	0.5	mg/Kg
Explosives	2,4-Dinitrotoluene	8330A/B	SS	75-111	20	0.5	mg/Kg
Explosives	2,6-Dinitrotoluene	8330A/B	SS	80-118	20	0.5	mg/Kg
Explosives	2-Nitrotoluene	8330A/B	SS	76-127	20	0.5	mg/Kg
Explosives	3-Nitrotoluene	8330A/B	SS	77-113	20	0.5	mg/Kg

Table 12.3: QC Targets for Semi-Volatiles Accuracy (LCS), Precision and RL's
This table is subject to revision without notice

Class	Analyte	Method	Matrix	Accuracy (%)	Prec. (RPD)	RL	Unit
Explosives	4-Nitrotoluene (4-NT)	8330A/B	SS	70-119	20	0.5	mg/Kg
Explosives	Hexahydro-1,3,5-Trinitro-1,3,5-Triazine	8330A/B	SS	74-115	20	0.5	mg/Kg
Explosives	Methyl-2,4,6-Trinitrophenylnitramine	8330A/B	SS	28-157	37	0.5	mg/Kg
Explosives	Nitrobenzene	8330A/B	SS	80-109	20	0.5	mg/Kg
Explosives	Octahydro - 1,3,5,7 -tetranitro-1,3,5,7-tetrazocine (HMX)	8330A/B	SS	77-122	20	0.0005	mg/Kg
Explosives	Pentaerythritol Tetranitrate (PETN)	8330A/B	SS	50-150	20	2	mg/Kg
Explosives	Nitroglycerine	8330A/B	SS	50-150	20	2	mg/Kg
Explosives	Nitroguanidine	8330A/B	SS	50-150	20	8	mg/Kg
Explosives	1,3,5-Trinitrobenzene	8330A/B	GW	72-105	20	0.0005	mg/L
Explosives	1,3-Dinitrobenzene	8330A/B	GW	56-97	20	0.0005	mg/L
Explosives	2,4,6-Trinitrotoluene	8330A/B	GW	60-95	20	0.0005	mg/L
Explosives	2,4-Dinitrotoluene	8330A/B	GW	39-92	20	0.0005	mg/L
Explosives	2,6-Dinitrotoluene	8330A/B	GW	34-101	20	0.0005	mg/L
Explosives	2-Nitrotoluene	8330A/B	GW	44-90	20	0.0005	mg/L
Explosives	3-Nitrotoluene	8330A/B	GW	37-91	20	0.0005	mg/L
Explosives	4-Nitrotoluene (4-NT)	8330A/B	GW	37-92	20	0.0005	mg/L
Explosives	Hexahydro-1,3,5-Trinitro-1,3,5-Triazine	8330A/B	GW	46-90	20	0.0005	mg/L
Explosives	Methyl-2,4,6-Trinitrophenylnitramine	8330A/B	GW	47-92	20	0.0005	mg/L
Explosives	Nitrobenzene	8330A/B	GW	61-97	20	0.0005	mg/L
Explosives	Octahydro - 1,3,5,7 -tetranitro-1,3,5,7-tetrazocine (HMX)	8330A/B	GW	37-94	20	0.0005	mg/L
Explosives	Pentaerythritol Tetranitrate (PETN)	8330A/B	GW	57-94	20	0.0005	mg/L
Explosives	Nitroglycerine	8330A/B	GW	34-140	20	0.0005	mg/L
Explosives	Nitroguanidine	8330A/B	GW	50-150	20	0.0005	mg/L
GC	1, 2 Dibromoethane (EDB)	504/8011	DW,GW, WW	70 - 130	<30	0.00002	mg/L
GC	1, 2 Dibromo-3-chloropropane	504/8011	DW,GW, WW	70 - 130	<30	0.00002	mg/L
GC	1,2,3-Trichloropropane	504/8011	DW,GW, WW	70 - 130	<30	0.0005	mg/L
THAA	Bromoacetic Acid	552.2	DW	70 - 130	<30	0.001	mg/L
THAA	Chloroacetic Acid	552.2	DW	70 - 130	<30	0.002	mg/L
THAA	Dibromoacetic Acid	552.2	DW	70 - 130	<30	0.001	mg/L
THAA	Dichloroacetic Acid	552.2	DW	70 - 130	<30	0.001	mg/L
THAA	Trichloroacetic Acid	552.2	DW	70 - 130	<30	0.001	mg/L
TPH	Petroleum Range Organics (TRPH)	FL-PRO	GW,	50 - 150	<20	0.1	mg/L
TPH	Petroleum Range Organics (TRPH)	FL-PRO	SS	50 - 150	<20	4.0	mg/Kg
TPH	Petroleum Range Organics (TRPH)	EPH TN	GW	50 - 150	<20	0.1	mg/L
TPH	Petroleum Range Organics (TRPH)	EPH TN	SS	50 - 150	<20	4.0	mg/Kg

Table 12.3: QC Targets for Semi-Volatiles Accuracy (LCS), Precision and RL's
This table is subject to revision without notice

Class	Analyte	Method	Matrix	Accuracy (%)	Prec. (RPD)	RL	Unit
TPH	Petroleum Range Organics (TRPH) - C9-C18, C19-C36, C11-C22	MADEP EPH	GW, WW	50 - 150	<20	0.1	mg/L
TPH	Petroleum Range Organics (TRPH) - C9-C18, C19-C36, C11-C22	MADEP EPH	SS	50 - 150	<20	5.5	mg/Kg
TPH	Petroleum Range Organics (TRPH) - C10-C28	DRO, 8015Mod	GW, WW	50 - 150	<20	0.1	mg/L
TPH	Petroleum Range Organics (TRPH) - C10-C28	DRO, 8015Mod	SS	50 - 150	<20	4.0	mg/Kg
TPH	Petroleum Range Organics (TRPH) – C10-C20, C20-C34	OHIO DRO	GW, WW	50 - 150	<20	0.1	mg/L
TPH	Petroleum Range Organics (TRPH) – C10-C20, C20-C34	OHIO DRO	SS	50 - 150	<20	4.0	mg/Kg
TPH	Petroleum Range Organics (TRPH) – gas, diesel, motor oil, etc.	OA2	GW, WW	50 - 150	<20	0.1	mg/L
TPH	Petroleum Range Organics (TRPH) – gas, diesel, motor oil, etc.	OA2	SS	50 - 150	<20	4.0	mg/Kg
TPH	Petroleum Range Organics - C10-C28, C28-C40	DRORLA	GW, WW	50 - 150	<20	0.1	mg/L
TPH	Petroleum Range Organics - C10-C28, C28-C40	DRORLA	SS	50 - 150	<20	4.0	mg/Kg
TPH	Petroleum Range Organics – C10-C32	DROWY	GW, WW	50 - 150	<20	0.1	mg/L
TPH	Petroleum Range Organics – C10-C32	DROWY	SS	50 - 150	<20	4.0	mg/Kg
TPH	Petroleum Range Organics – gas, diesel, motor oil, etc.	NWTPH-Dx	GW, WW	50 - 150	<20	0.25	mg/L
TPH	Petroleum Range Organics – gas, diesel, motor oil, etc.	NWTPH-Dx	SS	50 - 150	<20	25	mg/Kg
TPH	Petroleum Range Organics – C10-C28	DROWM	GW, WW	75 - 115	<20	0.1	mg/L
TPH	Petroleum Range Organics – C10-C28	DROWM	SS	70 - 120	<20	10	mg/Kg
TPH	Petroleum Range Organics – C10-C22	TPHAZ	SS	70-130	<20	30	mg/Kg
TPH	Petroleum Range Organics – C22-C32	TPHAZ	SS	70-130	<20	100.	mg/Kg
TPH	Petroleum Range Organics – C10-C32	TPHAZ	SS	70-130	<20	130.	mg/Kg
TPH	Petroleum Range Organics - C6-C12, C12-C28, C28-C35, C6-C35	TX TPH	SS	75 - 125	<20	50	mg/Kg
TPH	Petroleum Range Organics - C10-C21, C21-C35	DROMO	GW, WW	75 - 125	<20	1.0	mg/L
TPH	Petroleum Range Organics - C10-C21, C21-C35	DROMO	SS	75 - 125	<20	20	mg/Kg
IH	Aromatic Hydrocarbons	NIOSH 1501	Air	85-115	<20	10	ug/sample

13.0 CORRECTIVE ACTION

13.1 In the event that a nonconformance occurs in conjunction with the analytical batch, a corrective action response (CAR) form must be completed. The cause of the event is stated on the form and the measures taken to correct the nonconformance clearly defined. The effectiveness of the corrective action must be assessed and noted. The CAR is kept on file by the QA Department. Corrective action procedures are documented in SOP #030208, *Corrective and Preventive Action*

13.2 Required Corrective Action

Control limits have been established for each type of analysis. When these limits are exceeded, corrective action must be taken. Calculated sample spike control limits are also used.

All samples and procedures are governed by ESC's quality assurance program. General corrective actions are as follows; however additional and more specific direction is provided in the specific determinative procedure. For more information, see the appropriate determinative SOP.

13.2.1 Laboratory QC Criteria and Appropriate Corrective Actions

If the analytical method contains acceptance/rejection criteria and it is more stringent than those controls generated by the laboratory the method criteria will take precedence.

13.2.2 Out Of Control Blanks: Applies to Method, Trip, Rinsate & Instrument Blanks

Rejection Criteria - Blank reading is more than twice the background absorbance or more than 1/2 RL.

Corrective Action - Blanks are re-analyzed and the response is assessed. Standard curves and samples are evaluated for any obvious contamination that is isolated or uniform throughout the run. If necessary, reagents are re-prepared. Analyses are not initiated until the problem is identified and solved. If samples have already been prepared or analyzed, the Department Manager or QA Department is consulted to determine if data needs to be rejected or if samples need to be re-prepared.

13.2.3 Out Of Control Laboratory Control Standards (LCS & LCSD)

Rejection Criteria - If the performance is outside of lab-generated control limits which are calculated as the mean of at least 20 data points ±3 times the standard deviation of those points (Listed in Section 12) and the marginal excedence allowance is surpassed (see section 12.2).

Corrective Action - Instrument settings are checked and the LCS standard is reanalyzed. If the LCS is still out of control, instrumentation is checked for systemic problems and repaired (if necessary). Re-calibration is performed and the samples affected since the last in control reference standard are rerun. The group leader, Department Manager, or QA Department is consulted for further action.

13.2.4 Out Of Control Matrix Spike Samples

Rejection Criteria - If sample is outside of lab-generated control limits from accuracy charts on matrix spike samples from a similar matrix (i.e., water, solid, etc). Limits are calculated as the mean of at least 20 data points ±3 times the standard deviation of those points.

Corrective Action - Spiking technique is assessed to ascertain if the sample has been spiked correctly. The spiked sample should be 1 – 5 times the client sample concentration; otherwise, the percent recovery (%R) or relative percent difference (%RPD) of the MS/MSD is flagged as not meaningful or usable. The sample is re-spiked and re-analyzed, along with several other similar samples in subset. If an out of control situation persists, sample matrix interference is indicated. Samples to be analyzed by standard additions are prepared (where appropriate), and the group leader, Department Manager, or QA Department is notified.

13.2.5 Out Of Control Duplicate Samples

Rejection Criteria - Lab-generated maximum RPD limit (as listed under precision in Section 12)

Corrective Action - Instrument and samples are checked to see if precision variance is likely (i.e., high suspended solids content, high viscosity, etc.). They are re-analyzed in duplicate and samples just before and just after the duplicated sample are re-checked. If problem still exists, Department Manager, or QA Department is notified to review the analytical techniques.

13.2.6 Out Of Control Matrix Spike Duplicates

Rejection Criteria - These QC samples can be out of control for accuracy, precision, or both.

Corrective Action - The appropriate corrective actions listed for either matrix spikes, duplicate samples, or both are followed.

NOTE: Some samples cannot be duplicated. This is the case for wipe samples, filters, and some water samples. When possible, sampling personnel should collect duplicate samples.

13.2.7 Out Of Control Calibration Standards: ICV, CCV, SSCV

Rejection Criteria - If the performance is outside of method requirements.

Corrective Action - Instrument settings are checked, calibration verification standard is reanalyzed. If the standard is still out of control, recalibration is performed, and samples affected since the last in control reference standard are rerun. The group leader, Department Manager, or QA Department will be consulted for further action.

14.0 RECORD KEEPING

Record keeping is outlined in SOP #010103, *Document Control and Distribution*, SOP #030203, *Reagent Logs and Records* and SOP #030201, *Data Handling and Reporting.* Semi-Volatile organics calibration data are recorded and integrated using HP Enviroquant software. Calibration data from the semi-volatile analyses, in addition to the initial and daily calibration, includes GC/MS autotunes, DFTPP reports and surrogate recovery reports. Hard copy records of initial calibration and daily calibration are stored with chromatograms and integrated with sample data by date analyzed.

15.0 QUALITY AUDITS

System and data quality audits are outlined in the ESC Quality Assurance Manual Version 8.0.

1.0 SIGNATORY APPROVALS

Air Laboratory
QUALITY ASSURANCE MANUAL

APPENDIX VIII TO THE ESC QUALITY ASSURANCE MANUAL

for

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NOTE: The QAM has been approved by the following people. A signed cover page is available upon request

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2.0 APPENDIX TABLE OF CONTENTS

3.0 SCOPE AND APPLICATION

This appendix discusses specific QA requirements for general analytical protocols to ensure that analytical data generated from the Air Laboratory are scientifically valid and are of acceptable quality. Any deviations from these requirements and any deviations that result in nonconforming work must be immediately evaluated and their corrective actions documented.

4.0 LABORATORY ORGANIZATION AND RESPONSIBILITIES

ESC Lab Sciences offers diverse environmental capabilities that enable the laboratory to provide the client with both routine and specialized services, field sampling and broad laboratory expertise. A brief outline of the organization and responsibilities as they apply to the ESC Quality Assurance Program is presented in *Section 4.0 in the ESC Quality Assurance Manual Version 8.0.*

5.0 PERSONNEL AND TRAINING

5.1 PERSONNEL

Kenneth W. Buckley, with a B.S. degree in General Science, is the Department Manager of Organics and Wet Chemistry laboratories. Mr. Buckley reviews and approves all data reduction associated with analyses in these areas and is responsible for the overall production of these laboratories; including the management of the staff and scheduling. Mr. Buckley has over 9 years of environmental laboratory experience. In his absence, J. D. Gentry, with a B.S. degree in Chemistry and over 9 years of environmental laboratory experience, assumes responsibility for Air Department decisions.

5.2 TRAINING

The primary analyst or Manager trains new laboratory analysts according to ESC protocol. ESC's training program is outlined in *SOP 030205 Technical Training and Personnel Qualifications.* Performance is documented using an initial demonstration of capability (IDOCs) and continuing demonstration of capability (CDOC). Documentation of analyst training is maintained on file within the department.

6.0 FACILITIES AND LABORATORY SAFETY

6.1 FACILITIES

The main area of the laboratory has approximately 670 square feet of area with roughly 150 square feet of bench area. There are 670 square feet of additional storage and the lighting is fluorescence. The air system is a ten-ton Trane split unit with natural gas for heating. The laboratory reagent water is provided through the US Filter deionizer system. Waste disposal containers are located in the laboratory and Clean Harbors serves as ESC's hazardous waste disposal company. ESC's building information guides and site plan are shown in Appendix I.

6.2 LABORATORY SAFETY

- Laboratory access is limited when work is being performed.
- All procedures where chemicals are prepared or splashes may occur are conducted in laboratory exhaust hoods.

ESC's laboratory safety guidelines are detailed in the *ESC Chemical Hygiene and Safety Plan.*

7.0 SAMPLING PROCEDURES

7.1 FIELD SAMPLING PROCEDURES, SAMPLE STORAGE, AND HANDLING

- Field Sampling procedure is described in Appendix III of this ESC Quality Assurance Manual. Sample information is recorded and kept on the ESC chain of custody and field logbooks.
- Samples for air analysis are collected in four ways:
  - Samples may be collected directly in evacuated Summa canisters fit with the appropriately adjusted regulator that controls sampling flow to fill the canister over a given time period.
  - Summa canisters may also be collected as "grab" samples by simply opening the canister without the aid of a flow regulator and allowing the canister to fill quickly by virtue of the canister vacuum.
  - The third method entails collection of field samples using various sized bags specifically designed for air sampling (i.e. Tedlar). This type of sampling allows a pump connected to the bag to sample the air over the appropriate timeframe needed by the client.
  - The headspace of containers housing water samples may also be analyzed for specific volatile components.

- Air samples taken in summa canisters should be shipped in bubble wrapped boxes. Tedlar bags and water samples can be shipped in a container or cooler that is sufficiently rigid and protects the samples from damage that may be incurred in shipping. The chain of custody is also placed in the container. The shipping label containing the name and address of the shipper is affixed to the outside of the cooler.
- Samples are received in the laboratory login area and are tracked using LIMS (Laboratory Information Management System). A Chain of Custody Form accompanies all samples received by the lab. This is necessary to prove the traceability of the samples and to document the change in possession from sampling to delivery to receipt by the laboratory. Prior to analysis samples are checked for integrity. Sample handling, tracking and acceptance procedures are outlined in *SOP #060105, Sample Receiving.*

8.0 EQUIPMENT

8.1 EQUIPMENT LIST

LABORATORY EQUIPMENT LIST: MAJOR ITEMS - Air Analysis
This table is subject to revision without notice

Item	*Manufacturer*	*Model*	*Instrument Name*	*#*	*Serial #*	*Location*
Gas Chromatograph	HP	6890N TCD	AIRGC1	1	US10726007	Air Lab
Gas Chromatograph/Mass Spectrometer	HP	6890 GC/5973MSD	AIRMS1	1	GCUS00024616 MSUS63810244	Air Lab
Gas Chromatograph/Mass Spectrometer	Agilent	6890N/5975	AIRMS2	2	CN10551083	Air Lab
Gas Chromatograph/Mass Spectrometer	Agilent	6890/5973	AIRMS3	3	US000011333 US91911078	Air Lab
Preconcentrator	Entech	7100A			1089	Air Lab
Canister Autosampler	Entech	7016CA			1039	Air Lab
Tedlar Autosampler	Entech	7032A-L			1019	Air Lab
Dynamic Diluter	Entech	Model 4600A			1086	Air Lab
Canister Cleaner	Entech	Model 3100A			1045	Air Lab
Canister Cleaner	Entech	Model 3100A			1178	Air Lab
Canister cleaner	Entech	Model 3100A			B33-02663	Air Lab
Preconcentrator	Entech	7100A			1137	Air Lab
Canister Autosampler	Entech	7016CA			1137	Air Lab
Tedlar Autosampler	Entech	7032A-L			1017	Air Lab
GC/FID	Agilent	6890N	AIRGC2	2	US10137006	Air Lab
Headspace Autosampler	Tekmar	7000			9507018	Air Lab
TO Canister	Restek/Entech	TO-Can/ SiloniteCan	860 cans owned		N/A	Air Lab
Passive Sampling Kit	Restek		380 owned		N/A	Air Lab

LABORATORY EQUIPMENT LIST: MAJOR ITEMS - Air Analysis ***This table is subject to revision without notice***						
Item	***Manufacturer***	***Model***	***Instrument Name***	***#***	***Serial #***	***Location***
Field hand held PID	RAE Systems	MiniRae2000			110-012980	Air Lab
Field hand held PID	RAE Systems	MiniRAE2000				Air Lab

8.2 EQUIPMENT PREVENTIVE MAINTENANCE, EQUIPMENT CALIBRATION

INSTRUMENT	***P. M. DESCRIPTION***	***FREQUENCY***
Gas Chromatograph Detectors: FID	Change Quartz jet; clean; replace flame tip	As needed - when deterioration is noticeable
Gas Chromatograph/Mass Spectrometer	•Autotune Report	Inspected daily
Gas Chromatograph/Mass Spectrometer	•Clean ion source	As needed to maintain high mass resolution
Gas Chromatograph/Mass Spectrometer	•Replace vacuum pump oil	Every 6 months
Gas Chromatographs/Mass Spectrometer & Gas Chromatographs	•Replace column	When separation begins to degrade

8.3 STANDARDS AND REAGENTS

Table 8.3A: Standard stock sources, description and calibration information. ***This table is subject to revision without notice***					
Method	**Vendor**	**Description**	**Conc.**	**Storage Req.**	**Expiration**
TO-15/8260B (VAP)/Method 8-mod. ISTD Stock Standard	Spectra Gases	ISTD and Tuning Mixture	1 ppmv	3395 L (2A) cylinder	1 year
TO-15/ 8260B(VAP)/ Method 18-mod. Stock Standard*	Spectra Gases	Target Analytes except Bromoform at 3 ppmv, m&p Xylene at 2 ppmv and GRO at 40 ppmv	100 ppbv	3395 L (2A) cylinder	1 year
TO-15/ 8260B(VAP)/ Method 18-mod. Laboratory Control Stock Standard*	Spectra Gases	Target Analytes – Second Source	100 ppbv	3395 L (2A) cylinder	1 year
Landfill Gases Stock (CO_2, CO, CH_6, N_2, O_2, He)	Spectra Gases	Target Analytes	3 Levels	3395 L (2A) cylinder	1 year
Landfill Gases Laboratory Control Stock Standard	Spectra Gases	Target Analytes – Second Source	20%	3395 L (2A) cylinder	1 year
RSK-175 (Methane, Ethane, Ethene) Stock Standard	Scotty Gases	Target Analytes	1000 ppmv	3395 L (2A) cylinder	1 year
RSK-175 Laboratory Control Stock Standard	Scotty Gases	Target Analytes – Second Source	1000 ppmv	3395 L (2A) cylinder	1 year

TABLE 8.3B: Intermediate/Working Standard Concentrations ***This table is subject to revision without notice***					
Organic Compounds	**Method #**	**Working Standard Concentrations**	**Volume of Stock Used**	**Final Volume**	**Expiration**
ISTD and Tuning Intermediate Standard	TO-15/8260B (VAP)/Method 18.	20 ppbv	900 cc	45L in 15L Canister	1 year

TABLE 8.3B: Intermediate/Working Standard Concentrations *This table is subject to revision without notice*					
Organic Compounds	**Method #**	**Working Standard Concentrations**	**Volume of Stock Used**	**Final Volume**	**Expiration**
Target Analytes* Intermediate Standard	TO-15/8260B (VAP)/Method 18	5 ppbv except Bromoform at 5ppbv, m&p Xylene at 10 ppbv and GRO at 200 ppbv	225 cc	45L in 15L Canister	1 year
TO-15/ 8260B(VAP)/ Method 18-mod. Laboratory Control* Intermediate Standard	TO-15/8260B (VAP)/Method 18	Second Source: 5 ppbv except Bromoform at 15ppbv, m&p Xylene at 10 ppbv and GRO at 200 ppbv	225 cc	45L in 15L Canister	1 year

** see analytes listed in Table 12.3.*

8.4 INSTRUMENT CALIBRATION

TO-15, 8260B(Ohio VAP Air), Gasoline Range Components (Method 18) – Volatiles in Air by GC/MS – SOP Numbers 330367, 330368, & 330369

Detector mass calibration is performed daily using the autotune function of the GC/MS analytical system and PFTBA (Perfluorotributylamine). Following verification of the appropriate masses, the instrument sensitivity is verified by injecting a tuning solution containing Bromofluorobenzene (BFB). The BFB must meet the following ion abundance criteria:

Mass	Ion Abundance Criteria
50	15.0-40.0% of mass 95
75	30.0-60.0% of mass 95
95	base peak, 100% relative abundance
96	5.0-9.0% of mass 95
173	< 2.0% of mass 174
174	> 50.0% of mass 95
175	5.0-9.0% of mass 174
176	> 95.0%, but less than 101% of mass 174
177	5.0-9.0% of mass 176

Successful tuning must occur every 24 hours for method TO-15 and Method 18 and every 12 hours for method 8260B.

Following successful tuning, the GC/MS is calibrated using the internal standard procedure. A standard curve is prepared using a minimum of five standards. The calibration standards are tabulated according to peak height or area against concentration and the concentrations and responses of the internal standard analytes. The results are used to determine a response factor for each analyte in each standard injected.

A TO-15 or Method 18 calibration curve is constructed and determined to be acceptable if each analyte is found to be constant over the working range (<30 % RSD with no more than 2 compounds being between 30 and 40 % RSD). When this condition is met, linearity through the origin can be assumed and the average RF can be used in place of a calibration curve.

When analyzing air by method 8260B, specific target analytes in the calibration standards are defined as calibration check compounds (CCCs) or system performance check compounds (SPCCs).

SPCCs:	
Analyte	Minimum Relative Response Factor
Chloromethane	0.10
1,1-Dichloroethane	0.10
Bromoform	0.10
Chlorobenzene	0.30
1,1,2,2-Tetrachloroethane	0.30

CCCs:	
1,1-Dichloroethene	Toluene
Chloroform	Ethylbenzene
1,2-Dichloropropane	Vinyl Chloride

Analytes identified by the method as SPCCs must meet the minimum average response factors listed above for successful initial calibration. Compounds identified as CCCs must have a %RSD of less than 30% in the initial calibration curve. The remaining target analytes in the calibration standards must be <15% RSD. Initial 8260B calibration that does not meet these requirements is not accepted and re-calibration must be performed. Linear regression can be used for any target compound exceeding the 15% RSD criteria providing that the correlation coefficient is 0.990 or better.

For all methods, the initial calibration range must represent the typical air sample and include the lowest standard at or below the RL. The linear range of the instrument must be monitored to ensure that the maximum calibration point is within the range. Following successful calibration, the analysis of field and QC samples may begin. Analysis may be performed only during the timeframe of a valid tuning cycle (12 hours for 8260B and 24 hours for TO-15 and Method 18). Following the expiration of the tuning clock, the instrument must be retuned and either recalibrated or the existing calibration may be verified prior to further sample analysis.

For 8260B analyses, daily continuing calibration verification (CCV) includes successful demonstration of BFB sensitivity and the injection of a mid-level CCV standard

containing all the target analytes of interest, the CCC, and SPCC compounds. The BFB tune must meet the ion abundance criteria (see table above). Each SPCC in the calibration verification standard must meet a minimum response factors listed above. The CCCs must achieve the criteria of +/- 20% RSD. Each internal standard in the CCV must recover between -50% to + 100%, when compared to the same internal standard compound in the mid-point standard of the initial calibration curve. Additionally, if the retention time of an internal standard changes by more than 30 seconds from the retention time of the same internal standard in the mid-level standard of the most recent initial calibration, the system must be evaluated, corrected, and possibly re-calibrated.

For TO-15 and Method 18 analyses, daily calibration verification is accomplished by a successful demonstration of BFB sensitivity and the injection of a mid-level CCV standard containing all the target analytes of interest. The BFB tune must meet the same ion abundance criteria as previously listed and the CCV standard must recover within 30% of predicted response for all analytes of interest.

Landfill Gases (Carbon Dioxide, Carbon Monoxide, Methane, Nitrogen, Oxygen) – SOP Number 330366

Optimize the conditions of the Gas Chromatograph with Thermal Conductivity Detection according to the manufacturer's specification to provide good resolution and sensitivity. Verify that the gas flows and column and detector temperatures are at optimum levels for analysis, based on peak resolution and chromatograph performance. Allow sufficient time between each temperature adjustment to attain a stable reading (typically one hour). Standards are injected at a minimum of three concentration levels from purchased certified standards. Generation of the initial calibration is performed using Chrom-Perfect Spirit software and a linear regression model. The correlation coefficient must be at least 0.990. Instrument calibration must be verified initially on days when a full calibration curve is not analyzed, following every 10 injections during the analytical sequence, and at the end of each sequence by the analysis of a check standard. These standards must recover within 30% of the expected concentration. Each sample is analyzed in triplicate and the average sample area for each compound is calculated. The sample results are considered acceptable when the injections agree within 5% of their average. If this criteria is not met, additional injections are analyzed until consistent area data is obtained.

Methane, Ethane, Ethene based on RSK-175 – SOP Number 330370

Optimize the conditions of the Gas Chromatograph with Thermal Conductivity Detection according to the manufacturer's specification to provide good resolution and sensitivity. Verify that the gas flows and column and detector temperatures are at optimum levels for analysis, based on peak resolution and chromatograph performance. Allow sufficient time between each temperature adjustment to attain a stable reading (typically one hour). Standards are injected at a minimum of three concentration levels. The target analytes in the calibration standards must be <15% RSD. Linear regression can be used for any

target compound exceeding the 15% RSD criteria providing that the correlation coefficient is 0.990 or better. Headspace is created in each field sample by forcing 20cc of helium into each sample vial. Following sufficient time for the sample and headspace to reach equilibrium, 100 uL of air is removed from each vial and injected into the GC. Instrument calibration must be verified initially on days when a full calibration curve is not analyzed, following every 10 injections during the analytical sequence, and at the end of each sequence by the analysis of a check standard. These standards must recover within 15% of the expected concentration.

8.5 ACCEPTANCE/REJECTION OF CALIBRATION

The initial calibration curve is compared with previous curves for the same analyte. All new standard curves are immediately checked with a secondary source or laboratory control standard prepared from a separate source than those used for calibration. All curves are visually reviewed to ensure that acceptable correlation represents linearity. Calibration curves may be rejected for nonlinearity, abnormal sensitivity, or poor response of the laboratory control standard.

Continuing calibration verification is performed on each day that initial calibration is not performed and following every tenth sample. If a check standard does not perform within established criteria then the instrument will undergo evaluation to determine the problem. Once the problem is corrected, all samples between the last in control sample and the first out of control check will be re-analyzed.

TABLE 8.5: INSTRUMENT CALIBRATION & QC

Analysis/ Instrument	Calibration Type	Number of Standards	Acceptance/ Rejection Criteria	Frequency
TO-15 & Method 18/ GC/MS	Initial/ Continuing	1 - Tuning Solution	Mass m/z Abundance Criteria 50 8-40% of mass 95 75 30-66% of mass 95 95 Base peak, 100% 96 5-9% of mass 95 173 <2% of mass 174 174 >50% of mass 95 175 4-9% of mass 174 176 >93% but <101% of mass 174 177 5-9% of mass 176	TO-15/ M-18: Every 24 hours 8260 VAP: Every 12 hours
TO-15 & Method 18/ GC/MS	Initial	5 minimum	Average Response Factor: <30 % RSD with no more than 2 compounds being between 30 and 40 % RSD	As needed
8260B VAP/ GC/MS	Initial	5	Average Response Factor: Target analytes in the calibration standards must be <15% RSD, CCCs must have a %RSD of less than 30% & SPCCs must meet the minimum	As needed

TABLE 8.5: INSTRUMENT CALIBRATION & QC

Analysis/ Instrument	Calibration Type	Number of Standards	Acceptance/ Rejection Criteria	Frequency
			average response factors. Linear regression can be used for any target compound exceeding the 15% RSD	
TO-15 & Method 18/ GC/MS	Continuing	1 cal. check verification (CCV)	Percent Difference for all compounds <30%	Daily, when init. calibration is not required.
TO-15 VAP/ GC/MS	Continuing	1 cal. check verification (CCV)	Average Response Factor: Target analytes in the calibration standards must be <15% RSD, CCCs must have a %RSD of less than 20% & SPCCs must meet the minimum average response factors.	Daily, when init. calibration is not required.
TO-15 & Method 18	Initial/ Continuing	1 - Blank	< ½ RL, concentrations of common laboratory contaminants shall not exceed the reporting limit	Following init. calibration or daily cal. verification
TO-15 & Method 18	Initial/ Continuing	2 – Second source (LCS/LCSD)	Must be within +/-30% with an RPD of <25.	Following initial calibration or daily cal. Verification
Landfill Gas	Initial	3	Average Response Factor: Target analytes in the calibration standards must be <15% RSD. Linear regression can be used for any target compound exceeding the 15% RSD	As needed
Landfill Gas	Continuing	1 - cal. check verification (CCV)	Target analytes in the calibration standards must be <15% RSD.	Daily, when init. calibration is not required, following every 10th injection, and the end of the sequence.
Landfill Gas	Initial/ Continuing	1 - Blank	< ½ RL, concentrations of common laboratory contaminants shall not exceed the reporting limit	Following init. calibration or daily cal. verification
Landfill Gas	Initial/ Continuing	2 – Second source (LCS/LCSD)	Must be within +/-30% with an RPD of <25.	Following initial calibration or daily cal. verification
RSK-175	Initial	3	Average Response Factor: Target analytes in the calibration standards must be <15% RSD. Linear regression can be used for any target compound exceeding the 15% RSD	As needed
RSK-175	Continuing	1 - cal. check verification (CCV)	Target analytes in the calibration standards must be <15% RSD.	Daily, when init. calibration is not required, following every 10th injection, and the end of the sequence.
RSK-175	Initial/ Continuing	1 - Blank	< ½ RL, concentrations of common laboratory contaminants shall not exceed the reporting limit	Following init. calibration or daily cal. verification
RSK-175	Initial/	2 – Second source	Must be within +/-30% with an RPD of <25.	Following initial

TABLE 8.5: INSTRUMENT CALIBRATION & QC				
Analysis/ Instrument	**Calibration Type**	**Number of Standards**	**Acceptance/ Rejection Criteria**	**Frequency**
	Continuing	(LCS/LCSD)		calibration or daily cal. verification

9.0 LABORATORY PRACTICES

9.1 REAGENT GRADE WATER

Reagent Grade water –Type II used in the Microbiology Laboratory is periodically checked for contamination. Type II water is checked annually for single and total heavy metals. Monthly checks for total organic carbon, ammonia and organic nitrogen, total residual chlorine and a heterotrophic plate count are also conducted. Conductivity and pH are checked continuously or with each use.

9.2 SAMPLER CLEANING AND CERTIFICATION PROCEDURE

Canisters are cleaned in the laboratory using the Entech 3100 4-Position Canister Cleaner. Canisters are cleaned in batches of 4 to 8 per cleaning cycle. Prior to cleaning, canisters are inspected for integrity, damage and visible contamination. Acceptable canisters are connected to the manifold on the Entech cleaner and the cleaning cycle is controlled using Entech SmartLab software. Programmable cleaning cycles include: light, medium and heavy-duty and the cycle selected depends on the previous use of the dirtiest canister being cleaned. The cleaner automatically performs a leak check for the canisters and the manifold prior to the initial evacuation cycle. Heating bands are placed on each canister to elevate the temperature of the metallic canister to a level that provides for efficient cleaning. The typical cleaning cycle parameters are:

	Operating temperature = 120°C
1	Initial evacuation of canister to 1000 mtorr
2	Refill canister to 20psi
3	Evacuate the canister to 50 mtorr
4	Repeat items 2 & 3 for 8 total cycles
5	Final zero air pressure in clean canister is 50 mtorr.

Following cleaning, a single canister is selected as a QC sample for the entire batch and the sample is filled with zero air or nitrogen and analyzed to verify that successful cleaning has occurred. If the analysis indicates that the batch is clean (i.e. <0.2 ppbv for target analytes and free of additional contamination), the QC sample is returned to the cleaner manifold. The entire batch is evacuated to less than 50 mtorr and clearly labeled as clean and ready for sample collection. If the QC sample indicates that canister contamination is still present, the batch may be recycled through the cleaning process until residual contamination is no longer present. If following repeated cleaning cycles,

residual contamination is still observed, canisters may be permanently removed from service and clearly identified as unusable.

Tedlar bags and vials, as used for headspace analyses, are purchased as certified pre-cleaned from approved providers.

9.3 TYPICAL ENTECH AUTOSAMPLER OPERATING PARAMETERS

These parameters are provided as an example and may be modified to improve analytical system performance or better address project needs.

Line Temp = 100°C	Module 2 Desorb = 180°C
Bulk Head 1 = 30°C	Module 2 Bake = 190°C
Bulk Head 2 = 30°C	Module 2 Desorb Time = 3.5 min
Module 1 Trap = -150°C	Module 3 Trap = -180°C
Module 1 Preheat = 20°C	Module 3 Inject = 2 min
Module 1 Desorb = 20°C	Module 3 Bake Time = 2 min
Module 1 Bake = 130°C	Module 3 Event = 3
Module 1 Bake Time = 5 min	Module 3 Wait Time = 25 min.
Module 2 Trap = -30°C	Pressure Comp Factor = 14
Module 2 Preheat = off	Loop Flush = 30 seconds

10.0 ANALYTICAL PROCEDURES

10.1 A list of laboratory SOP's associated with the air laboratory can be found in the following table:

TABLE 10.1: AIR DEPARTMENT SOP'S

This Table is subject to revision without notice

SOP #	Title/Description
330366	Determination of Carbon Dioxide, Carbon Monoxide, Methane, Nitrogen and Oxygen in Air Samples.
330367	Measurement of Volatile Organic Compound in Ambient Air by GC/MS (EPA TO-15)
330368	Gasoline Range Organics in Ambient Air by GC/MS – Method 18 Modified
330369	Volatile Organic Compounds in Air by GC/MS 8260B for the Ohio VAP Program (with provisions for GRO determination based on 8015B)
330370	Method for Determination of Methane, Ethane, and Ethene (Based on RSK-175)

10.2 Sample Dilutions:

Dilutions for air samples from summa canisters and Tedlar bags may take three forms depending on the level of dilution required. These dilution techniques are demonstrated below:

Autosampler Dilution:

- First, a smaller sample volume can be analyzed using the capabilities of the Entech autosampler. For example, for a standard sample volume of 400cc, if 40cc were analyzed, that would be equivalent to a 10-fold dilution.
- The smallest sample volume that can be accurately analyzed using the autosampler method is l0cc (or a 40x).

Pressurized Manual Dilution:

- Sometimes, a 40X dilution is not sufficient to bring the concentration of a target analyte within the calibration range. In those cases, the sample canister is pressurized resulting in a dilution of the target analytes present.
- The act of introducing more pure air into the canister performs a dilution.
- The canister can then be analyzed at 400cc or diluted using a lesser autosampler volume, if necessary.

Secondary Manual Dilution:

- In extreme cases, the canister may need to be diluted into a second evacuated canister.
- This is accomplished by using a gas tight syringe to remove an aliquot of sample (1-l0mL) from the initial canister then injecting it into a clean evacuated second canister.
- The second canister is then analyzed and quantified taking into account the dilution based on the amount of sample injected and the total volume of the canister utilized.

Tedlar Bag Dilutions:

- Dilutions on Tedlar bags can be performed in much the same manner as summa canisters using either the autosampler dilution or the secondary manual dilution using a second Tedlar bag and filling it with pure air then adding an aliquot of field sample using a gas tight syringe.

11.0 QUALITY CONTROL CHECKS

NOTE: For specific guidance on each determinative method, including required quality control and specific state requirements/modifications, refer to the relevant laboratory standard operating procedure(s).

11.1 Initial Demonstrations of Capability (IDOC's) are performed during new analyst training and/or prior to acceptance and use of any new method/instrumentation. Continuing Demonstration of Capability (CDOC's) must be updated at least annually. The associated data is filed within the department and available for review.

11.2 A Laboratory Control Sample (LCS) and LCS Duplicate are analyzed per batch of samples and must yield recoveries within 70-130% of the expected concentration for all analytes and this pair must not exceed and RPD of 25%. LCS stock standards are prepared from sources independent of the calibration standards and also serve to verify the original calibration curve.

11.3 A method preparation blank is performed per batch of samples processed. If one-half the reporting limit [RL] is exceeded, the laboratory shall evaluate whether reprocessing of the samples is necessary, based on the following criteria:

- The blank contamination exceeds a concentration greater than 1/10 of the measured concentration of any sample in the associated preparation batch or
- The blank contamination is greater than 1/10 of the specified regulatory limit.

The concentrations of common laboratory contaminants shall not exceed the reporting limit. Any samples associated with a blank that fail these criteria shall be reprocessed in a subsequent preparation batch, except when the sample analysis resulted in non-detected results for the failing analytes.

12.0 DATA REDUCTION, VALIDATION AND REPORTING

12.1 DATA REDUCTION

The analyst performs the data calculation functions and is responsible for the initial examination of the finished data. Data reduction steps applied to the raw data are outlined in SOP #030201, *Data Handling and Reporting.* The Quality Control Department performs the secondary review of the data package using the ESC SOP #030227, *Data Review*. The QC Reviewer verifies that the analysis has performed as required and meets method criteria, all associate data is present and complete, and also ensures that any additional documentation is completed as required (i.e. Ohio VAP checklists, required flags on test reports, etc.)

TABLE 12.1 Data Reduction Formulas	
PARAMETER	**FORMULA**
GC/MS – Analyte Response Factor	response of analyte primary ion {*area*} x concentration of analyte (ug/L) response of ISTD primary ion {*area*} x concentration of ISTD (ug/L) *Calculations performed by HP Enviroquant Software*
GC/MS – Sample Analyte Concentration	response of primary ion in analyte x int. std concentration. {*ppbv*} x dilution factor response factor {*area/(mg/ml)*} x initial volume-mass {*ml or g*} x int. std cal. {area} *Calculations performed by HP Enviroquant Software*

12.2 VALIDATION

The validation process consists of data generation, reduction review, and reporting results. Once data reduction is complete, validation is conducted by verification that the QC samples are within acceptable QC limits and that all documentation is complete, including the analytical report and associated QC. See Table 12.3 by method for current QC targets and controls and current reporting limits.

Organic Control Limits - The organic QC targets are statutory in nature; warning and control limits for organic analyses are initially set for groups of compounds based on preliminary method validation data. When additional data becomes available, the QC targets are reviewed. All QC targets are routinely re-evaluated at least annually (and updated, if necessary) against laboratory historical data to insure that the limits continue to reflect realistic, method achievable goals.

12.3 REPORTING

Reporting procedures are documented in SOP #030201, *Data Handling and Reporting.*

Table 12.3: QC Targets for Air Accuracy (LCS), Precision and RL's
This table is subject to revision without notice

Analyte	Method	Matrix	Accuracy (%)	Prec. (% RPD)	RL	Unit
1,1,1-Trichloroethane	TO-15	Air	70-130	25	0.2	ppbv
1,1,2,2-Tetrachloroethane	TO-15	Air	70-130	25	0.2	ppbv
1,1,2,2-Tetrachloroethane	TO-15	Air	70-130	25	0.2	ppbv
1,1,2-Trichloroethane	TO-15	Air	70-130	25	0.2	ppbv
1,1-Dichloroethane	TO-15	Air	70-130	25	0.2	ppbv
1,1-Dichloroethene	TO-15	Air	70-130	25	0.2	ppbv
1,2,4-Trichlorobenzene	TO-15	Air	70-130	25	0.63	ppbv

Table 12.3: QC Targets for Air Accuracy (LCS), Precision and RL's
This table is subject to revision without notice

Analyte	Method	Matrix	Accuracy (%)	Prec. (% RPD)	RL	Unit
1,2,4-Trimethylbenzene	TO-15	Air	70-130	25	0.2	ppbv
1,2-Dibromoethane	TO-15	Air	70-130	25	0.2	ppbv
1,2-Dichlorobenzene	TO-15	Air	70-130	25	0.2	ppbv
1,2-Dichloroethane	TO-15	Air	70-130	25	0.2	ppbv
1,2-Dichloropropane	TO-15	Air	70-130	25	0.2	ppbv
1,3,5-Trimethylbenzene	TO-15	Air	70-130	25	0.2	ppbv
1,3-Butadiene	TO-15	Air	70-130	25	0.2	ppbv
1,3-Dichlorobenzene	TO-15	Air	70-130	25	0.2	ppbv
1,4-Dichlorobenzene	TO-15	Air	70-130	25	0.2	ppbv
1,4-Dioxane	TO-15	Air	70-130	25	0.2	ppbv
1,1,1-Trichloroethane	TO-15	Air	70-130	25	0.2	ppbv
2,2,4-Trimethylpentane	TO-15	Air	70-130	25	0.2	ppbv
2-Chlorotoluene	TO-15	Air	70-130	25	0.2	ppbv
2-Propanol	TO-15	Air	70-130	25	0.2	ppbv
4-Ethyltoluene	TO-15	Air	70-130	25	0.2	ppbv
Acetone	TO-15	Air	70-130	25	1.25	ppbv
Allyl Chloride	TO-15	Air	70-130	25	0.2	ppbv
Benzene	TO-15	Air	70-130	25	0.2	ppbv
Benzyl Chloride	TO-15	Air	70-130	25	0.2	ppbv
Bromomethane	TO-15	Air	70-130	25	0.2	ppbv
Bromodichloromethane	TO-15	Air	70-130	25	0.2	ppbv
Bromoform	TO-15	Air	70-130	25	0.6	ppbv
Carbon Disulfide	TO-15	Air	70-130	25	0.2	ppbv
Carbon Tetrachloride	TO-15	Air	70-130	25	0.2	ppbv
Chlorobenzene	TO-15	Air	70-130	25	0.2	ppbv
Chloroethane	TO-15	Air	70-130	25	0.2	ppbv
Chloroform	TO-15	Air	70-130	25	0.2	ppbv
Chloromethane	TO-15	Air	70-130	25	0.2	ppbv
Cis-1,2-Dichloroethene	TO-15	Air	70-130	25	0.2	ppbv
Cis-1,3-Dichloropropene	TO-15	Air	70-130	25	0.2	ppbv
Cyclohexane	TO-15	Air	70-130	25	0.2	ppbv
Dibromochloromethane	TO-15	Air	70-130	25	0.2	ppbv
Ethanol	TO-15	Air	70-130	25	0.63	ppbv
Ethyl Acetate	TO-15	Air	70-130	25	0.2	ppbv
Ethylbenzene	TO-15	Air	70-130	25	0.2	ppbv

Table 12.3: QC Targets for Air Accuracy (LCS), Precision and RL's
This table is subject to revision without notice

Analyte	Method	Matrix	Accuracy (%)	Prec. (% RPD)	RL	Unit
Freon-11	TO-15	Air	70-130	25	0.2	ppbv
Freon-12	TO-15	Air	70-130	25	0.2	ppbv
Freon-113	TO-15	Air	70-130	25	0.2	ppbv
Freon-114	TO-15	Air	70-130	25	0.2	ppbv
Gasoline Range Organics	TO-15	Air	70-130	25	50	ppbv
Heptane	TO-15	Air	70-130	25	0.2	ppbv
Hexachloro-1,3-Butadiene	TO-15	Air	70-130	25	0.63	ppbv
Hexane	TO-15	Air	70-130	25	0.2	ppbv
Isopropylbenzene	TO-15	Air	70-130	25	0.2	ppbv
M&P-Xylene	TO-15	Air	70-130	25	0.4	ppbv
Methyl Butyl Ketone	TO-15	Air	70-130	25	1.25	ppbv
Methyl Ethyl Ketone	TO-15	Air	70-130	25	1.25	ppbv
Methyl Isobutyl Ketone	TO-15	Air	70-130	25	1.25	ppbv
Methyl Methacrylate	TO-15	Air	70-130	25	0.2	ppbv
Methyl tert Butyl Ether	TO-15	Air	70-130	25	0.31	ppbv
Methylene Chloride	TO-15	Air	70-130	25	0.63	ppbv
Naphthalene	TO-15	Air	70-130	25	0.63	ppbv
o-Xylene	TO-15	Air	70-130	25	0.2	ppbv
Propene	TO-15	Air	70-130	25	0.4	ppbv
Styrene	TO-15	Air	70-130	25	0.2	ppbv
t-Butyl Alcohol	TO-15	Air	70-130	25	0.2	ppbv
Tetrachloroethylene	TO-15	Air	70-130	25	0.2	ppbv
Tetrahydrofuran	TO-15	Air	70-130	25	0.2	ppbv
Toluene	TO-15	Air	70-130	25	0.2	ppbv
Trans-1,3-Dichloropropene	TO-15	Air	70-130	25	0.2	ppbv
Trans-1,2-Dichloroethene	TO-15	Air	70-130	25	0.2	ppbv
Trichloroethylene	TO-15	Air	70-130	25	0.2	ppbv
Vinyl Acetate	TO-15	Air	70-130	25	0.2	ppbv
Vinyl Bromide	TO-15	Air	70-130	25	0.2	ppbv
Vinyl Chloride	TO-15	Air	70-130	25	0.2	ppbv
Methane	RSK-175	Air/ Headspace	70-130	25	0.01	ppmv
Ethane	RSK-175	Air/ Headspace	70-130	25	0.129	ppbmv
Ethene	RSK-175	Air/ Headspace	70-130	25	0.127	ppmv
Carbon Dioxide	Method 3C	Air	70-130	25	0.50 / 200	% / ppmv

Table 12.3: QC Targets for Air Accuracy (LCS), Precision and RL's
This table is subject to revision without notice

Analyte	Method	Matrix	Accuracy (%)	Prec. (% RPD)	RL	Unit
Carbon Monoxide	Method 3C	Air	70-130	25	0.50 / 200	% / ppmv
Methane	Method 3C	Air	70-130	25	0.50 / 200	% / ppmv
Nitrogen	Method 3C	Air	70-130	25	0.50 / 200	% / ppmv
Oxygen	Method 3C	Air	70-130	25	0.50 / 200	% / ppmv

13.0 CORRECTIVE ACTION

13.1 In the event that a nonconformance occurs in conjunction with the analytical batch, a corrective action response (CAR) form must be completed. The reason for the nonconformance is stated on the form and the measures taken to correct the nonconformance clearly defined. The effectiveness of the corrective action must be assessed and noted. The CAR is kept on file by the QA department. Corrective action procedures are documented in SOP #030208, *Corrective and Preventive Action*

13.2 Required Corrective Action

All samples and procedures are governed by ESC's quality assurance program. Designated corrective actions are as follows.

All samples and procedures are governed by ESC's quality assurance program. General corrective actions are as follows; however additional and more specific direction is provided in the specific determinative procedure. For more information, see the appropriate determinative SOP

13.2.1 Laboratory QC Criteria and Appropriate Corrective Actions

If the analytical method contains acceptance/rejection criteria and it is more stringent than those controls generated by the laboratory the method criteria will take precedence.

13.2.2 Calibration Verification Criteria Are Not Met.

Rejection Criteria – See Table 8.5.

Corrective Action – Instrument settings are checked. The standard is reviewed for obvious cause. The standard may require re-analysis or the instrument may require recalibration.

13.2.3 Out Of Control Blanks:

Rejection Criteria - Blank reading is more than ½ the RL.

Corrective Action - Instrument settings are checked. The Blank is re-analyzed. If the blank is still out of control, bakeout of the system is performed and the blank is re-analyzed.

13.2.4 Out Of Control Laboratory Control Standards (LCS)

Rejection Criteria - If the performance is outside of lab-generated control (Listed in Table 12.3).

Corrective Action - Instrument settings are checked. The LCS standard is re-analyzed. If the LCS is still out of control, re-calibration is performed, and samples affected since the last in control reference standard are re-analyzed.

14.0 RECORD KEEPING

Record keeping is outlined in SOP #010103, *Document Control and Distribution*, SOP #030203, *Reagent Logs and Records* and SOP #030201, *Data Handling and Reporting*

15.0 QUALITY AUDITS

System and data quality audits are outlined in the ESC Quality Assurance Manual Version 8.0.

1.0 SIGNATORY APPROVALS

Aquatic Toxicity Laboratory QUALITY ASSURANCE MANUAL

APPENDIX IX TO THE ESC QUALITY ASSURANCE MANUAL

for

ESC LAB SCIENCES
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Prepared by

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NOTE: The QAM has been approved by the following people. A signed cover page is available upon request

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2.0 APPENDIX TABLE OF CONTENTS

3.0 SCOPE AND APPLICATION

This appendix discusses specific QA requirements for general analytical protocols to ensure that analytical data generated from the Aquatic Toxicity laboratory are scientifically valid and are of acceptable quality. Any deviations from these requirements and any deviations that result in non-conforming work must be immediately evaluated and their corrective actions documented.

4.0 LABORATORY ORGANIZATION AND RESPONSIBILITIES

ESC Lab Sciences offers diverse environmental capabilities that enable the laboratory to provide the client with both routine and specialized services, field sampling and broad laboratory expertise. A brief outline of the organization and responsibilities as they apply to the ESC Quality Assurance Program is presented in *Section 4.0 in the ESC Quality Assurance Manual Version 8.0.*

5.0 PERSONNEL AND TRAINING

5.1 PERSONNEL

Kim Johnson, with a B.S. degree in Biological Sciences, is the Department Manager of the Aquatic Toxicity laboratory. Ms. Johnson reviews and approves all data reduction associated with Aquatic Toxicity analysis. Her responsibilities include the coordination with clients regarding sample analysis for regulatory compliance, scheduling of testing and personnel, and data reduction, interpretation and validation for Toxicity analyses. Ms. Johnson is also involved in microbiological assessments of wastewater, sludges and drinking water and oversees the Protozoan laboratory. She is also a certified mold analyst. In her absence, Shain Schmitt assumes responsibility for departmental decisions.

5.2 TRAINING

All new analysts to the laboratory will be trained by the primary analyst or Manager according to ESC protocol. ESC's training program is outlined in *SOP 350355 Technical Training and Personnel Qualification for Biology.*

6.0 FACILITIES AND LABORATORY SAFETY

6.1 FACILITIES

The main area of the laboratory has approximately 1440 square feet of area with roughly 280 square feet of bench area. There are 300 square feet of additional storage and the lighting is fluorescence. The air system is a five-ton Trane split unit with natural gas for heating. The laboratory reagent water is provided through the Siemans Elga UltraPure deionizer system. Waste disposal containers are located in the laboratory and Clean Harbors serves as ESC's hazardous waste disposal company. Biohazard containers are located in the laboratory and Stericycle Waste Removal serves as ESC'S biological waste disposal contractor. ESC's building information guides and site plan are shown in Appendix I.

6.2 LABORATORY SAFETY

- Laboratory access is limited when work is being performed.
- All procedures where chemicals are prepared or splashes may occur are conducted in laboratory exhaust hoods, where applicable.

ESC's laboratory safety guidelines are detailed in *the ESC Chemical Hygiene and Safety Plan.*

7.0 SAMPLING PROCEDURES

7.1 FIELD SAMPLING PROCEDURES, SAMPLE STORAGE, AND HANDLING

- Field Sampling procedure is described in Appendix III of this ESC Quality Assurance Manual. Sample information is recorded and kept on the ESC chain of custody and field logbooks.
- Samples are received in the laboratory login area and are tracked using LIMS (Laboratory Information Management System). A Chain of Custody Form accompanies all samples received by the lab. This is necessary to prove the traceability of the samples and to document the change in possession from sampling to delivery to receipt by the laboratory. Prior to analysis samples are checked for integrity. Once samples are checked to confirm integrity, the samples are logged with unique sample identification information and a label is affixed to each container. Chronic Toxicity samples are uniquely identified with "sample 1, sample 2 and sample 3". A sample custodian then transports samples to the laboratory. Sample handling and tracking procedures are outlined in *SOP 060105, Sample Receiving.*

- Requirements for sample acceptance is located in *SOP 060105, Sample Receiving.* At a minimum, the following physical and chemical parameters are analyzed for each sample received:

  - Temperature - recorded up to twice daily.
  - pH - initial and final measurements recorded
  - D.O. - initial and final measurements recorded
  - Specific Conductance
  - Alkalinity
  - Hardness
  - Total Residual Chlorine
- Samples must be immediately cooled and maintained at 0-6^{o}C during shipment and prior to testing.

Residual Chlorine Treatment

- Residual chlorine in biomonitoring samples are monitored using a pocket colorimeter and these checks are documented. Chlorine removal is not performed.

Dissolved Oxygen

- For acute tests, samples that are $\leq$ 4.0mg/L are aerated until the sample reaches 90% saturation. For chronic tests, samples that are $\leq$ 4.0 mg/L are aerated until the sample reaches 90% saturation.

8.0 EQUIPMENT

8.1 EQUIPMENT LIST

LABORATORY EQUIPMENT LIST: MAJOR ITEMS – Aquatic Toxicity Lab ***This table is subject to revision without notice.***			
Item	***Manufacturer***	***Model***	***Location***
Analytical Balance	Mettler	AT261 Delta Range	Aquatic Tox Lab
Class "I" weights (2)	Troemner		Aquatic Tox Lab
Conductivity Meter	Orion	150 A+	Aquatic Tox Lab
Dissolved Oxygen Meter	YSI	Model 50	Aquatic Tox Lab
Stereoscope	Olympus	SZX-IllK100	Aquatic Tox Lab
Oven	Fisher	655F	Aquatic Tox Lab
Incubator	Thermo-Kool	Environmental chamber	Aquatic Tox Lab
Incubator	Percival Scientific	1-37 VL	Aquatic Tox Lab
Incubator	Precision Sci.	818	Aquatic Tox Lab
Incubator (2)	Precision Sci.	818	Aquatic Tox Lab
Microscope	Olympus	CHT	Aquatic Tox Lab
pH Meter	Beckman	pH/Temp/mV/ISE	Aquatic Tox Lab
Refrigerator (2)	Beverage Air	E Series	Aquatic Tox Lab
Stereoscope	Olympus	SZH-ILLD	Aquatic Tox Lab
Stereoscope	Olympus	SZH-ILLD	Aquatic Tox Lab
Refrigerator	Frigidaire	FRC445GB	Aquatic Tox Lab
Refrigerator	True	T-49	Aquatic Tox Lab
Water Purifier	Siemans	Elga Purelab	Aquatic Tox Lab
Refrigerator	Fridgidaire	FRC 445GB	Aquatic Tox Lab
Freezer	Kenmore	198130582	Aquatic Tox Lab

8.2 EQUIPMENT PREVENTIVE MAINTENANCE, EQUIPMENT CALIBRATION

PREVENTATIVE MAINTENANCE FOR LABORATORY EQUIPMENT		
INSTRUMENT	***P. M. DESCRIPTION***	***FREQUENCY***
Analytical Balances	•Check with Class "I" weights	Daily-tolerance 1 gm - ±0.0001 gm
Analytical Balances	•Service/Calibration (semiannual contract maintenance and calibration check)	10 gm - ±0.01 gm
Analytical Balances	•Service/Calibration (semiannual contract maintenance and calibration check)	Semi-annually
Refrigerators & Incubators	•Maintenance service	As needed - determined by twice daily temperature performance checks @ least 4 hours apart
Dissolved oxygen meter	•Calibrate with each use	Daily
Dissolved oxygen meter	•Change probe membrane	Every two to four weeks
Conductivity Meter	•Check probe cables	As needed
Conductivity Meter	•Clean probe	Daily
Conductivity Meter	•Replace or replatinize probe	Poor response not corrected by above
Conductivity Meter	•Calibrate with each use	Daily (or prior to each use)
Microscope/Stereoscope	•Service/calibration of each ocular micrometer	Annually
Microscope/Stereoscope	• Clean optics and stage	Each Use
pH Meters	•Reference junction & electrode replacement	As needed
pH Meters	•Probe stored in pH standard 4	At all times when not in use
pH Meters	•Other	As described in the manufacturer's manual
pH Meters	•Calibrate with each use	Daily (or prior to each use)
Bottle top dispenser/repipettor	•Calibrate	Quarterly
Bottle top dispenser/repipettor	•Clean to prevent residue buildup	As needed
Water Purifier	Tank Exchange, UV bulb and sleeve replacement (service contract maintenance and check	As needed and annually
Water Purifier	•Replace cartridge and filter	As needed and semi-annual

8.3 STANDARDS , REAGENTS AND ORGANISM CULTURES

All reagents and standards must meet the requirements listed in the analytical methods.

Table 8.3A: Stock solution sources, description and related information.
(subject to revision as needed)

Description	Vendor	Storage Req.	Expiration
Conductivity standard 100	Fisher	Ambient	1 yr
Conductivity standard 1000	Fisher	Ambient	1 yr
pH buffer 7	Fisher	Ambient	1 yr
pH buffer 10	Fisher	Ambient	1 yr
Bromothymol blue solution	Fisher	Ambient	1 yr
Potassium phosphate monobasic	Fisher	Ambient	1 yr
Magnesium chloride	JT Baker	Ambient in dessicator	1 yr
Potassium Chloride	EMD	Ambient in dessicator	1 yr
Brine shrimp eggs	Argentemia	Ambient, tightly sealed.	1 yr
Calcium sulfate	EM	Ambient in dessicator	1 yr
EDTA	Fisher	Ambient in dessicator	1 yr
Sodium thiosulfate	JT Baker	Ambient in dessicator	1 yr
pH buffer 4	Fisher	Ambient.	1yr
YCT	Made in-house	-10 to -20^{o}C	14 days after thawing
Selenastrum capricornatum	Aq. Biosystems	1-6^{o}C	NA
Vitamin B12	Fisher	1-6^{o}C	NA

TABLE 8.3B: Working Solution Descriptions and Related Information.
(subject to change)

Solution	Concentrations	Storage Requirements	Expiration
KCl stock solution	31.237g KCl to 2L of 20% DMW	1-4^{o}C	14 days
B12 Solution	0.01125g to 1L of DI Water	1-4^{o}C	NA

Source and Maintenance of in-house cultures:

Source of Biological Organisms (subject to change):
The primary source for all fathead minnows is:
Aquatic Biosystems Inc.
2821 Remington Street
Fort Collins, CO 80525

The source for their organisms is documented on each packing slip received. ESC accepts the packing slip as documentation and verification by the supplier with regards to the taxonomic identification of the bioassay species. The packing slips for bioassay test organisms are kept on file.

The amount of food added to culture vessels will depend upon the number of organisms within a given culture. As standard procedure, *Ceriodaphnia dubia* batch cultures are fed 4.5mL of YCT and algal suspension on the day of initiation. Batches are fed daily as needed. The date, time and the amount the organisms are fed are documented. All brewers yeast purchased is at least food grade and has passed FDA standards. All yeast trout chow is made in-house. New lots are tested for pesticides, metals, and PCB's.

Ceriodaphnia dubia, fresh batch cultures are set up on Monday, Wednesday and Friday using newly hatched neonates less than 24 hours old. In addition, a minimum of 4 brood trays are set up daily in order to guarantee organisms of the right age to use in bioassays. Condition of cultures is monitored daily and documented in the daily log. The *C. dubia* brood trays are fed daily. The *C. dubia* are transferred into fresh water daily after their first brood of neonates is born. Third generation neonates, less than 24 hours old, are used for batch cultures and brood trays. Third generation neonates, less than 24 hours old and hatched within 8 hours of each other, are used for tests. Adults are used as sources for neonates until 14 days of age.

C.dubia are taxonomically identified to species on a quarterly basis. All taxonomy information is documented and kept on file for a year.

Pimephales promelas batch cultures are cleaned as needed by siphoning off the excess food and waste from the bottom of the culture vessel and renewing the water. Cultures are aerated as needed to maintain adequate dissolved oxygen.

The water used for culturing is dilute mineral water prepared by diluting (6) 750mL bottles of Perrier to 20 Liters with deionized water and aerating for 24 hours. The physical and chemical parameters for each new tank of water prepared are recorded and should fall within the following acceptable range:

1. pH - 7.9 to 8.3 units
2. D.O. - greater than 80% saturation in mg/L
3. Specific Conductance - ~215 micromhos/cm
4. Alkalinity - 57 to 64 mg $CaCO_3$/L
5. Hardness - 80 to 100 mg $CaCO_3$/L
6. Total Residual Chlorine - <0.1 mg/L

Pimephales promelas are taxonomically identified to species on a quarterly basis. All taxonomy information is documented and kept on file for a year.

8.4 INSTRUMENT CALIBRATION

Lighting

All testing and culturing is maintained in incubators in which temperature is constant and the photoperiod is on a 16-hour light/8-hour dark cycle. The photoperiod is verified and documented quarterly. The light intensity must be within 50 – 100 foot candles and is verified and documented semi-annually. All incubators are monitored at least weekly for proper light intensity.

pH Meter

With each use of pH meters, calibrate the instrument according to manufacturer's instructions. The slope is documented on a daily basis. Acceptable pH slope range is 95-105%. All calibration information is documented.

Volumetric Equipment

Equipment such as filter funnels, bottles, pipettes non-Class A and other containers with graduations are calibrated once per lot prior to first use. Volumetric equipment that is not disposed of after use is calibrated on an annual basis. The error of calibration must not exceed 2.5%.

Analytical Balance

Analytical balances are checked and calibrated semi-annually by a certified technician. Calibration is checked before each use with Class I weights. Class I weights are calibrated annually.

Stereoscope

All glass surfaces are kept clean using a 3:7 mixture of alcohol and ether or a small amount of xylene. Maintenance is performed by a trained technician on an annual basis.

Conductivity Meter

With each use of conductivity meters, calibrate the instrument according to manufacturer's instructions.

Dissolved Oxygen Meter

With each use of the DO meter, calibrated according to manufacturer's instructions. The probe membrane is changed every two to four weeks to maintain accurate readings.

Test Chambers

Each test chamber is rinsed with DI water prior to introducing the test organisms.

Bottle Top Dispenser/Repipettor

Repipettors are calibrated quarterly to ensure the instrument is dispensing the correct amount. Periodic cleaning is performed to maintain the accuracy and to prevent buildup of residue.

Colorimeter Chlorine tester

The colorimeter is calibrated before each use using standards to verify the instrument is accurate.

9.0 LABORATORY PRACTICES

9.1 REAGENT GRADE WATER

Deionized water or reverse-osmosis produces water free from bactericidal and inhibitory substances and shall be used in the preparation of media, solutions and buffers. The quality of the water shall be monitored for chlorine residual, specific conductance, and heterotrophic bacteria plate count monthly (when in use), when maintenance is performed on the water treatment system, or at startup after a period of disuse longer than one month.

Analysis for metals is performed quarterly and the Bacteriological Water Quality Test or Use Test (to determine presence of toxic agents or growth promoting substances) shall be performed annually. Results of these analyses shall meet the specifications of the required method and records of analyses shall be maintained for five years. (An exception to performing the Bacteriological Water Quality Test shall be given to laboratories that can supply documentation to show that their water source meets the criteria, as specified by the method, for Type I or Type II reagent water.)

9.2 pH BUFFERS/CONDUCTIVITY STANDARDS

pH buffer and conductivity standard aliquots are used only once. Reagents containers are dated upon receipt and the date opened.

9.3 SPEC√ SECONDARY STANDARDS

Standards are used for retrieval and verification of the factory calibrated colorimeter and is used to verify consistent instrument calibration.

9.4 LABORATORY CONTROL WATER

Control water (20% dilute mineral water) is prepared by diluting (6) 750mL bottles of Perrier to 20 Liters with deionized water and aerating for 24 hours. The physical and chemical parameters for each new tank of water prepared are recorded and should fall within the following acceptable range:

1. pH - 7.9 to 8.3 units
2. D.O. - greater than 80% saturation in mg/L
3. Specific Conductance - ~215 micromhos/cm
4. Alkalinity - 57 to 64 mg $CaCO_3$/L
5. Hardness - 80 to 100 mg $CaCO_3$/L
6. Total Residual Chlorine - <0.1 mg/L

Control water (10% dilute mineral water) is prepared by diluting (3) 750mL bottles of Perrier to 20 Liters with deionized water and aerating for 24 hours. The physical and chemical parameters for each new tank of water prepared are recorded and should fall within the following acceptable range:

1. pH – 6.5 to 8.5 units
2. D.O. - greater than 80% saturation in mg/L
3. Specific Conductance - ~215 micromhos/cm
4. Alkalinity - 60 to 70mg $CaCO_3$/L
5. Hardness - 30 to 50mg $CaCO_3$/L
6. Total Residual Chlorine - <0.1mg/L

A given batch of control water is not used for more than 14 days following preparation.

9.5 BRINE SHRIMP

Artemia cysts are of platinum or gold grade, certified brine shrimp eggs from ARGENT chemical Laboratories. To determine the quality of the new lots of Brine shrimp, a side-by-side comparison test is performed using the new food and the food of known acceptable quality.

9.6 YCT

YCT is prepared in the laboratory. To determine the quality of the new lots of YCT a side-by-side comparison test is performed using the new food and the food of known acceptable quality.

9.7 ALGAE

Algae is commercially prepared. Upon arrival, each batch received has an accompanying Certificate of Algae Preparation History. The certificate provides the following quality control data: date prepared, species name, inoculation date, harvest date, concentration date and cell count.

9.8 GLASSWARE WASHING, STERILIZATION PROCEDURES AND EQUIPMENT STERILITY CHECKS

Glassware washing and preparation/sterilization procedures are performed according to EPA guidelines and are outlined in *SOP 030701 Glassware Cleaning* and *SOP 350334 Sterilization, Sanitization and Residue Testing of Microbiological Glassware and Equipment*. Before use, examine and discard items with chipped edges or etched inner surfaces. Reusable glassware is cleaned using the following protocol:

- Soak for 15 minutes in hot tap water with detergent and scrub. Rinse thoroughly with tap water. Rinse thoroughly with dilute nitric acid (10%). Rinse thoroughly with deionized water. Rinse thoroughly with pesticide grade acetone. Rinse well with deionized water.
- New glassware will be cleaned according to the same procedure as listed above except the first step will be preceded by soaking overnight in 10 % $HNO_{3.}$

Inspect glassware after washing for excessive water beading and rewash, if necessary. Perform checks on pH and test for inhibitory residues on glassware and plastic ware. Use utensils and containers of borosilicate glass, stainless steel, aluminum, or other corrosion resistant material for media preparation. All biological glassware is purchased pre-sterilized. Sterilization of any auxiliary equipment is performed via autoclave.

Pipettes of all sizes are checked for sterility by drawing up non-selective media into the pipette and re-dispensing the volume back into original tube that contained the media. The tube is then incubated and monitored for growth. All results are recorded and maintained within the laboratory.

10.0 ANALYTICAL PROCEDURES

10.1 A list of laboratory SOP's associated with the microbiology laboratory can be found in the following table:

TABLE 10.1: AQUATIC TOXICITY DEPARTMENT SOP'S

This Table is subject to revision without notice

SOP #	Title/Description
350301	Fathead Minnow, *Pimephales promelas*, Larval Survival and Growth Test, EPA Method 1000.0
350302	Cladoceran, *Ceriodaphnia dubia*, Chronic Survival and Reproduction Test, EPA Method 1002.0
350303	*Pimephales promelas* Acute Toxicity Testing
350304	*Ceriodaphnia dubia* Acute Toxicity Testing
350317	WET Reference toxicant testing
350318	Mini Chronic *C. dubia* NC
350319	Phase II Chronic *C. dubia* NC
350320	Acceptability Test of New Food Batches for WET Testing
350321	Pocket Colorimeter Chlorine Tester Maintenance and Calibration
350322	DO Meter Maintenance and Calibration
350323	Fluke Thermometer Operation and Maintenance
350324	Digital Light Meter Maintenance and Method of Operation
350325	pH Meter Maintenance and Calibration
350326	Thermometer Operation, Maintenance and Calibration Procedure
350327	Bottle Top Dispenser Maintenance and Method of Operation
350328	Conductivity Meter Maintenance and Calibration
350329	Taxonomic Verification/Identification of *Pimephales promelas* - Fathead Minnow
350330	Taxonomic Verification/Identification of *Ceriodaphnia dubia*
350303NC	Acute Toxicity - Minnow NC
350304NC	Acute Toxicity - *C. dubia* NC

10.2 Additional information regarding microbiological testing can be found in:

Method Resources: EPA/821/R-02/013, EPA/821/R-02/012

- 7-Day Fathead Minnow (*Pimephales promelas*) Larval Survival and Growth Test; Test Method 1000.0 from "Short Term Methods for Estimating the Chronic Toxicity of Effluents and Receiving Waters to Freshwater Organisms" (EPA 821-R-02-013).
- 3-Brood *Ceriodaphnia dubia* Survival and Reproduction Test; Test Method 1002.0 from "Short Term Methods for Estimating the Chronic Toxicity of Effluents and Receiving Waters to Freshwater Organisms" (EPA 821-R-02-013).
- Fathead Minnow (*Pimephales promelas*) Acute Toxicity Test (24, 48 or 96 hour duration); referenced in "Methods for Measuring the Acute Toxicity of Effluents and Receiving Waters to Freshwater and Marine Organisms" (EPA 821-R-02-012, 10-02).
- *Ceriodaphnia dubia* Acute Toxicity Test (24, 48 or 96 hour duration); referenced in "Methods for Measuring the Acute Toxicity of Effluents and Receiving Waters to Freshwater and Marine Organisms" (EPA 821-R-02-012, 10-02)

11.0 QUALITY CONTROL CHECKS

11.1 At a minimum, the following physical and chemical parameters are analyzed for each biomonitoring sample received:

- Temperature - recorded up to twice daily.
- pH - initial and final measurements recorded
- D.O. - initial and final measurements recorded
- Specific Conductance
- Alkalinity
- Hardness
- Total Residual Chlorine

11.2 FEEDING REGIME

- 7-Day Fathead Minnow Larval Survival and Growth Test - Test organisms are fed 0.15mL, per container of 10 organisms. Newly hatched brine shrimp (*Artemia*) are fed to minnow batches 2-3 times daily. Batch cultures are fed depending on organism density.
- 3-Brood *Ceriodaphnia dubia* Survival and Reproduction Test - test organisms are fed 0.15mL of Yeast, Cereal leaves, Trout chow (YCT) and 0.15mL *Selenastrum capricornutum* algal suspension once daily.
- 24 and 48 Hour Acute Toxicity Tests - organisms are fed 2-5 hours prior to introduction into sample but are not fed for the duration of the test.
- 96-Hour Acute Toxicity Tests – organisms are fed at the 48 hour renewal period.
- 3-Brood *Ceriodaphnia dubia* Survival and Reproduction Test for North Carolina - test organisms are fed .05mL of YCT/15mL test solution and .05 Selanastrum capricornutum algal concentrate once daily (1.7x10 to the 7th power cells/mL).

11.3 BATCH CULTURES

Batch cultures are identified by date set up or date received. The set-up date is recorded for each batch.

Ceriodaphnia dubia, fresh batch cultures are set up on Monday, Wednesday and Friday using newly hatched neonates less than 24 hours old. In addition, a minimum of 4 brood trays are set up daily in order to guarantee organisms of the right age to use in bioassays. Condition of cultures is monitored daily and documented in the daily log. The *C. dubia* brood trays are fed daily. The *C. dubia* are transferred into fresh water daily after their first brood of neonates is born. Third generation neonates, less than 24 hours old, are used for batch cultures and brood trays. Third generation neonates, less than 24 hours old and hatched within 8 hours of each other, are used for chronic tests. Adults are used as sources for neonates until 14 days of age.

Pimephales promelas, organisms less than 36 hours old are obtained from a commercial supplier and are used immediately for chronic bioassays. Upon receipt, temperature, conductivity, pH, alkalinity and hardness are recorded and the organisms are slowly acclimated to a temperature of 25°C. If more than 10% mortality has occurred in the batch shipment, the batch is rejected and supplier is contacted. The date of the batch culture is recorded and batches are maintained for 14 days after receipt to use in acute tests. Batch cultures are monitored and fed daily. The number of organisms used is recorded in the daily log. Lots are cleaned as needed by siphoning off the excess food and waste from the bottom of the vessel and renewing the water. Minnow lots are aerated to maintain adequate dissolved oxygen. *Pimephales promelas* lots are fed 2.5 mL of newly-hatched brine shrimp per batch, 2-3 times daily. The date, time and the amount the organisms are fed are documented.

11.4 REFERENCE TOXICANT

The reference toxicant used at ESC is potassium chloride. Acute and chronic reference toxicant tests are performed at a minimum of once monthly and upper and lower control limits have been established. In respect to FDER related samples ESC will perform acute and chronic reference toxicant tests for all in-house cultures done with each batch.

12.0 DATA REDUCTION, VALIDATION AND REPORTING

12.1 DATA REDUCTION

The analyst performs the data calculation functions and is responsible for the initial examination of the finished data. Data reduction steps applied to the raw data are outlined in *SOP 030201 Data Handling and Reporting.* The primary analyst reviews the quality of data based on the following guidelines:

- The appropriate SOP has been followed
- Sample preparation is correct and complete
- Analytical results are correct and complete
- QC is within criteria and complete

All calculations are performed according to the EPA methods manual. When applicable, software is used to perform statistical analysis. All formulas are chosen appropriately depending on the conditions and outcome of each individual test. Due to the complexity of each formula please see EPA/821/R-02/013 for formulas pertaining to Chronic Toxicity tests and EPA/821/R-02/012 for formulas pertaining to Acute Toxicity tests.

TABLE 12.1 Data Reduction Formulas

PARAMETER	FORMULA
IC25, NOEC, LC50, AEC	Toxcalc 5.0 Software

For chronic tests the PMSD and the % CV is calculated and reported.

12.2 VALIDATION

The validation process consists of data generation, reduction review, and reporting results. Once data reduction is complete, validation is conducted by reviewing all data entries and calculations for errors, reviewing all documentation to assure that sample information is correct, and that the tests have been performed appropriately and within the appropriate holding times. The secondary analyst reviews the quality of data based on the following guidelines:

- The appropriate SOP has been followed
- Sample preparation is correct and complete
- Analytical results are correct and complete

12.3 REPORTING

Reporting procedures are documented in *SOP 030201 Data Handling and Reporting.*

13.0 CORRECTIVE ACTION

13.1 In the event that a nonconformance occurs in conjunction with the analytical batch, a corrective action response (CAR) form must be completed. The reason for the nonconformance will be stated on the form and the measures taken to correct the nonconformance clearly defined. The effectiveness of the corrective action must be assessed and noted. The CAR will be kept on file by the QA department. Corrective action procedures are documented in SOP 030208 *Corrective and Preventive Action*

13.2 Required Corrective Action

All samples and procedures are governed by ESC's quality assurance program. Designated corrective actions are as follows:

13.2.1 Laboratory QC Criteria and Appropriate Corrective Actions

If the analytical method contains acceptance/rejection criteria and it is more stringent than those controls generated by the laboratory the method criteria will take precedence.

13.2.2 Out of control acute toxicity tests.

Rejection Criteria –More than 10% mortality occurs in the control organisms within the specified time frame of the test.

Corrective Action – The test will be considered invalid and must be repeated using fresh control water.

13.2.3 Out of control 3-Brood *Ceriodaphnia dubia* Survival and Reproduction Test.

Rejection Criteria –If more than 10% mortality occurs in the control organisms within 96 hours or more than 20% mortality occurs in the test organisms in the 3-brood period (approx. 7 days)

Corrective Action – The test will be considered invalid and must be repeated using fresh control water.

13.2.4 Out of control 3-Brood *Ceriodaphnia dubia* Survival and Reproduction Test.

Rejection Criteria – If the average number of young produced in the control is less than 15 per organism

Corrective Action – The test will be considered invalid and must be repeated using fresh control water.

13.2.5 Out of control 3-Brood *Ceriodaphnia dubia* Survival and Reproduction Test.

Rejection Criteria – A test will be considered invalid if or less than 60% (80% for NC tests) of the original number of adult daphnia loaded do not produce three broods within an eight day maximum (7 day maximum for NC tests).

Corrective Action – The test will be considered invalid and must be repeated using fresh control water.

13.2.6 Out of control 7-Day *Pimephales promelas* Larval Survival and Growth Test.

Rejection Criteria –If more than 10% mortality occurs in the control organisms within 96 hours or more than 20% mortality occurs in the test organisms in 7 day period.

Corrective Action – The test will be considered invalid and must be repeated using fresh control water.

13.2.7 Out of control 7-Day *Pimephales promelas* Larval Survival and Growth Test.

Rejection Criteria – The average weight of the control minnows is less than 0.2500 mg.

Corrective Action – The test will be considered invalid and must be repeated using fresh control water.

13.2.8 Out of control Monthly Reference Toxicant:

Rejection Criteria – KCl is the reference toxicant used for acute and chronic testing for the following methods: 1000.0, 1002.0, 2000.0, and 2002.0. If reference toxicant test results fail to meet ESC in-house established criteria (± 2 standard deviations from the mean and median).

Corrective Action – The test is deemed invalid and must be repeated twice. No test will be performed using organisms that fail to meet reference toxicant criteria.

13.2.9 Out of control PMSD 7-Day *Pimephales promelas* Larval Survival and Growth Test.

Rejection Criteria – The PMSD value is greater than the upper value of 30.

Corrective Action - The test may be deemed invalid and should be repeated.

13.2.10 Out of control PMSD 3-Brood *Ceriodaphnia dubia* Survival and Reproduction Test.

Rejection Criteria – The PMSD value is greater than the upper value of 47.

Corrective Action - The test may be deemed invalid and should be repeated.

13.2.11 Out of control %CV 3-Brood *Ceriodaphnia dubia* Survival and Reproduction Test and 7-Day *Pimephales promelas* Larval Survival and Growth Test.

Rejection Criteria – The %CV value is greater than the upper value of 40%.

Corrective Action - The test is deemed invalid and must be repeated.

14.0 RECORD KEEPING

Record keeping is outlined in SOP #010103 *Document Control and Distribution*, SOP #030203 *Reagent Logs and Records* and SOP #030201 *Data Handling and Reporting*

15.0 QUALITY AUDITS

System and data quality audits are outlined in the ESC Quality Assurance Manual Version 8.0.

1.0 SIGNATORY APPROVALS

Microbiology Laboratory
QUALITY ASSURANCE MANUAL

APPENDIX X TO THE ESC
QUALITY ASSURANCE MANUAL

for

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2.0 APPENDIX TABLE OF CONTENTS

3.0 SCOPE AND APPLICATION

This appendix discusses specific QA requirements for general analytical protocols to ensure that analytical data generated from the Microbiology laboratory are scientifically valid and are of acceptable quality. Any deviations from these requirements and any deviations that result in non-conforming work must be immediately evaluated and their corrective actions documented.

4.0 LABORATORY ORGANIZATION AND RESPONSIBILITIES

ESC Lab Sciences offers diverse environmental capabilities that enable the laboratory to provide the client with both routine and specialized services, field sampling and broad laboratory expertise. A brief outline of the organization and responsibilities as they apply to the ESC Quality Assurance Program is presented in *Section 4.0 in the ESC Quality Assurance Manual Version 8.0.*

5.0 PERSONNEL AND TRAINING

5.1 PERSONNEL

Kimberly Johnson, with a B.S. degree in Biological Sciences, is the Department Manager of the Microbiology laboratory. Ms. Johnson reviews and approves all data reduction associated with Microbiological analyses. Her responsibilities include the coordination with clients regarding sample analysis for regulatory compliance, scheduling of testing and personnel, and data reduction, interpretation and validation. Ms. Johnson is also involved in biological assessments of aquatic habitats and Toxicity Identification Evaluations. Additionally, Ms. Johnson oversees the Protozoan laboratory and is also a certified mold analyst. In her absence, Shain Schmitt assumes responsibility for Microbiological and Aquatic Toxicity departmental decisions.

5.2 TRAINING

The primary analyst or Manager trains new laboratory analysts according to ESC protocol. ESC's training program is outlined in SOP #350355, *Technical Training and Personnel Qualification for Biomonitoring-Microbiology.* Performance is documented using an initial demonstration of capability (IDOCs) and continuing demonstration of capability (CDOC). On-going acceptable capability in microbiological analysis is also demonstrated by acceptable participation in the ERA proficiency testing program (PTs). Documentation of analyst training is maintained on file within the department.

6.0 FACILITIES AND LABORATORY SAFETY

6.1 FACILITIES

The main area of the laboratory has approximately 1440 square feet of area with roughly 280 square feet of bench area. There are 300 square feet of additional storage and the lighting is fluorescence. The air system is a five-ton Trane split unit with natural gas for heating. The laboratory reagent water is provided through the Siemans Elga UltraPure deionizer system. Waste disposal containers are located in the laboratory and Clean Harbors serves as ESC's hazardous waste disposal company. Biohazard containers are located in the laboratory and Stericycle Waste Removal serves as ESC'S biological waste disposal contractor. ESC's building information guides and site plan are shown in Appendix I.

6.2 LABORATORY SAFETY

- Laboratory access is limited when work is being performed.
- All procedures where chemicals are prepared or splashes may occur are conducted in laboratory exhaust hoods, where applicable.

ESC's laboratory safety guidelines are detailed in *the ESC Chemical Hygiene and Safety Plan.*

7.0 SAMPLING PROCEDURES

7.1 FIELD SAMPLING PROCEDURES, SAMPLE STORAGE, AND HANDLING

- Field Sampling procedure is described in Appendix III of this ESC Quality Assurance Manual. Sample information is recorded and kept on the ESC chain of custody and field logbooks.
- Samples for bacterial analysis are collected directly into pre-sterilized high-density polyethylene (HDPE) sample containers preserved with sodium thiosulfate. The container should be kept closed until sample collection. Once the container is open, do not wash, rinse or contaminate the cap or the inside of the container. For microbiological samples, the container is filled allowing at least 1 inch of headspace per container.
- Sources for microbiological samples are surface waters, waste and drinking water, ground water and soil/sludge.
- Holding times for microbiological drinking water samples is 30 hours (except HPC which has a 6 hour holding time). Soil and sludge samples have a holding time of 24 hour and 8 hours depending on the method used. All other water samples have a 6-hour hold time.

- Microbiological samples are shipped in a cooler lined with a heavy-duty plastic bag. Once the sample container lids are secure the samples are placed in appropriately sized polyethylene bags. The chain of custody is also placed in a plastic bag. The cooler liner is completely filled with ice and the plastic bag sealed tightly with a cable tie. The shipping label contains the name and address of the shipper and is affixed to the outside of the cooler.
- Samples are received in the laboratory login area and are tracked using LIMS (Laboratory Information Management System). A Chain of Custody Form accompanies all samples received by the lab. This is necessary to prove the traceability of the samples and to document the change in possession from sampling to delivery to receipt by the laboratory. Prior to analysis samples are checked for integrity. Sample handling, tracking and acceptance procedures are outlined in *SOP 060105, Sample Receiving.*

8.0 EQUIPMENT

8.1 EQUIPMENT LIST

LABORATORY EQUIPMENT LIST: MAJOR ITEMS - Microbiological Analysis ***This table is subject to revision without notice***			
Item	***Manufacturer***	***Model***	***Location***
Analytical Balance	Mettler	AT261 Delta Range	Microbiology Lab
Class “I” weights	(2 sets) Troemner		Microbiology Lab
Conductivity Meter	Orion	150 A+	Microbiology Lab
Autoclave	Pelton and Crane	Validator 8	Microbiology Lab
Water Bath	Lindberg Blue	WB1130A	Microbiology Lab
Water Bath	Blue M	MW-1110A-1	Microbiology Lab
Oven	Fisher	655F	Microbiology Lab
Incubator	Percival Scientific	1-37 VL	Microbiology Lab
Incubator	VWR	2030 22MFG	Microbiology Lab
Quantitray Sealer	IDEXX	2X	Microbiology Lab
Incubator	Precision Sci.	818	Microbiology Lab
Colony Counter	Quebecor		Microbiology Lab
pH Meter	Beckman	pH/Temp/mV/ISE	Microbiology Lab
Refrigerator	True	T-49	Microbiology Lab
Stereoscope (2)	Olympus	SZH-ILLD	Microbiology Lab
UV light; short and long wave	UVP		Microbiology Lab
Water Bath	VWR Scientific	1295PC	Microbiology Lab
Autoclave	SterlieMax	Harvey	Microbiology Lab
Stereoscope	Olympus	SZX-ILLK100	Microbiology Lab
Water Purifier	Siemans	Elga Purelab Plus	Microbiology Lab

8.2 EQUIPMENT PREVENTIVE MAINTENANCE, EQUIPMENT CALIBRATION

PREVENTATIVE MAINTENANCE FOR LABORATORY EQUIPMENT		
INSTRUMENT	***P. M. DESCRIPTION***	***FREQUENCY***
Analytical Balances	•Check with Class "I" weights	Daily-tolerance 1 gm - ±0.0001 gm
Analytical Balances	•Service/Calibration (semiannual contract maintenance and calibration check)	10 gm - ±0.01 gm
Analytical Balances	•Service/Calibration (semiannual contract maintenance and calibration check)	Semi-annually
Refrigerators, Incubators, and Water Baths	•Maintenance service	As needed - determined by twice daily temperature performance checks @ least 4 hours apart
Water Bath	•Check thermometer vs. N.B.S.	Annually
Water Bath	•Remove from service when not maintaining temperature and send off for repair or replace	As needed
Autoclave	•Check sterilization efficiency	Monthly – Geobacillus Stearothermophilus ampoule
Autoclave	•Check sterilization efficiency	Daily – Chemical Indicator Strip
Conductivity Meter	•Calibrate and clean probe	Daily
Conductivity Meter	•Replace or replatinize probe	Poor response not corrected by above
Stereoscope	• Clean optics and stage	Each Use
pH Meters	•Reference junction & electrode replacement	As needed
pH Meters	•Probe stored in 4 pH standard	At all times when not in use.
pH Meters	•Other	As described in the manufacturer's O & M manual
Autoclave	•Check timing device	Quarterly
pH meter	•Calibrate and check slope (acceptable range of 95-105 %)	Daily
Quanti-Tray Sealer	•Check sealer for leaks	Monthly
Water Purifier	•Conductivity check using a calibrated conductivity meter	Monthly
Water Purifier	•Check for TOC's, ammonia, nitrogen, TRC and heterotrophic bacteria	Monthly
Water Purifier	•Check for single and heavy total metals	Annually
Incubators and Water Baths	Perform temperature stability and load testing	Annually
Autoclave	•Check pressure (annual contract maintenance)	Annually
Stereoscope	• Clean optics and stage; microscope alignment (annual maintenance contract)	Annually

8.3 STANDARDS AND REAGENTS

All reagents and standards must meet the requirements listed in the analytical methods.

Table 8.3A: Commercially prepared agar/broth, reagent sources, and storage information. ***(subject to revision as needed)***

Agar Type	*Source*	*Storage*
M-FC Broth w/ Rosolic acid	Millipore	4 ± 2°C
mColiBlue Broth	Millipore	4 ± 2°C
A-1 Media (broth)	Hach	4 ± 2°C
mEndo Broth	Hach	4 ± 2°C
Lauryl Tryptose Broth	Hach	4 ± 2°C
Brilliant Green Lactose Broth	Hach	4 ± 2°C
EC media w/ mug broth	Hach	4 ± 2°C
HPC	Hach	4 ± 2°C
Colilert reagent powder	IDEXX	Room temp
Enterolert reagent powder	IDEXX	Room temp
Xylose Lysisne Deoxycholate Agar (XLD)	HealthLink	4 ± 2°C
Brilliant Green (BG) Agar	HealthLink	4 ± 2°C
Phosphate Buffer Solution	Weber Scientific	Room temp

All stock agar expirations are per manufacturer specification.

Table 8.3B: In-house prepared agar/broth, reagent sources, and storage information.
(subject to revision as needed)

Agar Type-Stock	*Source*	*Stock Storage*	*Stock Expiration*	*Preparation Components Media*	*Prepared Storage*	*Prepared Expiration*
Xylose Lysisne Deoxycholate Agar (XLD)	Fisher/Difco	Room Temp	As specified by Manufacturer	XLD + Water	4 ± 2°C	2 weeks
Brilliant Green (BG) Agar	Fisher/Difco	Room Temp	As specified by Manufacturer	BG + Water	4 ± 2°C	2 weeks
Plate Count Agar	Fisher/Difco	Room Temp	As specified by Manufacturer	PCA + Water	4 ± 2°C	3 months
Tryptic Soy Agar	Fisher/Difco	Room Temp	As specified by Manufacturer	TSA + Water	4 ± 2°C	3 months
Triple Sugar Iron (TSI)	Fisher/Difco	Room Temp	As specified by Manufacturer	TSI + Water	4 ± 2°C	3 months
Lysine Iron Agar (LIA)	Fisher/Difco	Room Temp	As specified by Manufacturer	LIA + Water	4 ± 2°C	3 months
Tetrathionate Broth (TTB)	Fisher/Difco	Room Temp	As specified by Manufacturer	TTB +Water + 1 drops Iodine	4 ± 2°C	24 hrs
Tryptic Soy Broth (TSB)	Fisher/Difco	Room Temp	As specified by Manufacturer	TSB + Water	4 ± 2°C	3 months
Lauryl Tryptose Broth (LTB)	Fisher/Difco	Room Temp	As specified by Manufacturer	LTB + Water	4 ± 2°C	3 months
Buffered Rinse Water	Fisher/Difco	4 ± 2°C	As specified by Manufacturer	KH_2PO_4 + $MgCl_2$+Water	Room temp.	1 year

Membrane Filters and Pads

Membrane filters and pads are purchased and certified to meet the following specifications:

- Filter diameter - 47 mm, mean pore diameter - 0.45 µm. Alternate filter and pore sizes may be used if the manufacturer provides data verifying performance equal to or better than that of 47mm-diam, 0.45-µm-pore size filter. At least 70% of filter area must be pores.
- When filters are floated on reagent water, the water diffuses uniformly through the filters in 15 s with no dry spots on the filters.
- Flow rates are at least 55 mL/min/cm2 at 25°C and a differential pressure of 93kPa.
- Filters are nontoxic, free of bacterial-growth-inhibiting or stimulating substances, and free of materials that directly or indirectly interfere with bacterial indicator systems in the media. Ink grid is nontoxic. The arithmetic mean of five counts on filters must be at least 90% of the arithmetic mean of the counts on five agar spread plates using the same sample volumes and agar media.
- Filters retain the organisms from a 100mL suspension of *Serratia marcescens* containing 1×10^3 cells.
- Water extractables in filters do not exceed 2.5% after the membrane is boiled in 100mL reagent water for 20min, dried, cooled, and brought to constant weight.
- Absorbent pad has diameter 47mm, thickness 0.8mm, and is capable of absorbing 2.0 ± 0.2mL Endo broth.
- Pads release less than 1mg total acidity calculated as $CaCO_3$ when titrated to the phenolphthalein endpoint with 0.02*N* NaOH.
- If the filter and absorbent pad are not sterile, they should not be degraded by sterilization at 121°C for 10min. Confirm sterility by absence of growth when a membrane filter is placed on a pad saturated with tryptic soy broth and incubated at 35 ± 0.5°C for 24h.

8.4 INSTRUMENT CALIBRATION

Autoclave

Prior to first use, autoclaves must be initially evaluated for performance. All initial checks must be recorded and records must be retained on file. With each use, a record of items sterilized, temperature, pressure, and time is kept for each batch processed. Operating temperature is checked and recorded at least weekly with a minimum/maximum thermometer. Performance is tested monthly with *Bacillus stearothermophilus* ampoules. Chemical strips are used daily to verify that supplies and materials have been sterilized. Records of autoclave operations shall be maintained for every cycle. Records shall include: date, contents, maximum temperature reached, pressure, time in sterilization mode, total run time (may be recorded as time in and time out) and analyst's initials.

Quebecor Colony counter

A dark field colony counter is used to count Heterotrophic Plate Count colonies. Maintenance is performed per manufacturer's instructions.

Quanti-tray Sealer

The Quanti-tray sealer is checked monthly using 100mL of bromcresol purple, or equivalent dye. The solution is poured into a test tray, sealed, and tested for leaks.

pH Meter/Conductivity Meter

With each use, calibrate the instrument according to the manufacturer's instructions. Verify that the slope of the calibration is within the 95-105% acceptable range prior to use.

Incubators & Waterbaths

Records of temperature checks are documented twice daily at least 4 hours apart when in use. Thermometers used for temperature checks are verified at least annually. Temperature stability and load testing is performed on an annual basis.

Analytical Balances

Analytical balances are checked at least daily prior to each use with class "I" weights. Records of these verifications are maintained within the laboratory. Balances are also serviced and verified and/or calibrated by an external calibration service at least semi-annually.

Volumetric Equipment, IDEXX and Commercially Prepared Phosphate Buffer Bottles

Equipment such as filter funnels, bottles, pipettes, non-Class A glassware and other containers with graduation must be calibrated once per lot prior to the first use.

IDEXX Bottles and Quanti-trays

Prior to first use, IDEXX bottles and Quanti-trays must be checked for fluorescence using a long wave UV light.

9.0 LABORATORY PRACTICES

9.1 REAGENT GRADE WATER

Reagent Grade water –Type II used in the Microbiology Laboratory is periodically checked for contamination. Type II water is checked annually for single and total heavy metals. Monthly checks for total organic carbon, ammonia and organic nitrogen, total residual chlorine and a heterotrophic plate count are also conducted. Resistivity and pH are checked continuously or with each use. Conductivity is also checked monthly using a calibrated conductivity meter.

9.2 GLASSWARE WASHING , STERILIZATION PROCEDURES AND EQUIPMENT STERILITY CHECKS

Glassware washing and preparation/sterilization procedures are performed according to EPA guidelines and are outlined in *SOP 030701 Glassware Cleaning and SOP 350334 Sterilization, Sanitization and Residue Testing of Microbiological Glassware and Equipment.* Before use, examine and discard items with chipped edges or etched inner surfaces. Reusable glassware is cleaned using the protocol established by the EPA:

- Soak for 15 minutes in hot tap water with detergent and scrub. Rinse thoroughly with tap water. Rinse thoroughly with dilute nitric acid (10%). Rinse thoroughly with deionized water. Rinse thoroughly with pesticide grade acetone. Rinse well with deionized water.
- New glassware will be cleaned according to the same procedure as listed above except the first step will be preceded by soaking overnight in 10 % HNO_3.

Inspect glassware after washing for excessive water beading and rewash, if necessary. Perform checks on pH and test for inhibitory residues on glassware and plastic ware. Use utensils and containers of borosilicate glass, stainless steel, aluminum, or other corrosion resistant material for media preparation. All biological glassware is purchased pre-sterilized. Sterilization of any auxiliary equipment is performed via autoclave.

Pipettes of all sizes are checked for sterility by drawing up non-selective media into the pipette and re-dispensing the volume back into original tube that contained the media. The tube is then incubated and monitored for growth. All results are recorded and maintained within the laboratory.

Inoculating loops are cultured by aseptically transferring the entire tip of the loop into a tube containing non-selective media. The tube is incubated and monitored for growth. Results are maintained within the laboratory.

A sterility check is performed on each batch of dilution and rinse water prepared in the laboratory and on each batch of commercially prepared water with non-selective growth media prior to first use.

In addition, stock solutions used for preparing rinse water are checked for turbidity prior to each use. If turbid, the stock buffer is discarded or re-sterilized.

9.3 MEDIA STERILITY VERIFICATION PROCEDURES

A sterility check must be analyzed for each lot of pre-prepared media and for each lot of media prepared in the laboratory. This is done prior to the first use of the media used for membrane filtration, MPN, pour plate and chromofluorogenic methods. For media used in the pour plate analytical technique, sterility blanks of the media must be made by pouring an uninoculated plate for each run in addition to sterility and lot comparison tests being performed on each lot prior to first use. Reagents and containers used in chromofluorogenic method tests are checked for fluorescence prior to first use.
All results of the sterility and lot comparison tests are documented.

9.4 POSITIVE AND NEGATIVE CONTROLS USING PURE CULTURES

ATCC Pure Cultures

Positive culture controls demonstrate that the media can support the growth of the target organism(s), and that the media produces the specified or expected reaction to the target organism(s). All media must be tested with at least one pure culture of a known positive reaction. This must be done prior to first use of the media.

Negative culture controls demonstrate that the media does not support the growth of non-target organisms or does not demonstrate the typical positive reaction of the target organism(s). All batches of selective media in the laboratory must be analyzed with one or more known negative culture controls. This must be done prior to first use of the media.

10.0 ANALYTICAL PROCEDURES

10.1 A list of laboratory SOP's associated with the microbiology laboratory can be found in the following table:

TABLE 10.1: MICROBIOLOGICAL DEPARTMENT SOP'S

This Table is subject to revision without notice

SOP #	Title/Description
350305	Fecal Coliform: Membrane Filter Technique
350334	HPC, Method 9215 B
350315	Fecal Coliform Determination in Biosolids: Membrane Filter Technique (SM9222D)
350316	Total Coliform
350325	PH Meter Maintenance and Calibration
350326	Thermometer Operation, Maintenance and Calibration Procedure
350328	Conductivity Meter Maintenance and Calibration
350331	Salmonella in Sludge
350332	Laboratory Maintenance of Bacteria Reference Cultures
350333	QA/QC of Microbiological Equipment and Testing Materials
350369	Sterilization, Sanitization and Residue Testing of Microbiological Glassware and Equipment
350359	Calibration and Maintenance of Autoclaves
350343	Colilert
350344	m-ColiBlue
350355	Technical Training and Personnel Qualification for Biomonitoring-Microbiology
350356	Water bath and Incubator Temperature Stability and Load Testing
350348	Enterolert

10.2 Additional information regarding microbiological testing can be found in:

- Standard Methods for the Examination of Water and Wastewater, 20th Edition, Section 9000.
- Heterotrophic Plate Count, SM 9215B
- Fecal Coliform Direct Test (A-1 Media), SM9221E
- Standard Total Coliform Membrane Filter Procedure, SM9222B.
- Fecal Coliform Membrane Filter Procedure, SM9222D.
- Enzyme Substrate Test, SM 9223B.
- Quantitative Salmonella Procedures, SM9260D.
- Environmental Regulations and Technology, Control of Pathogens and Vector Attraction in Sewage Sludge, Appendix F.

11.0 QUALITY CONTROL CHECKS

11.1 ESC participates in microbiological proficiency testing (PT's) by analyzing samples provided by Environmental Resource Associates (ERA). Unknowns are received and analyzed according to instructions from ERA and the standard operating procedure.

11.2 Plate count comparison between two analysts is conducted monthly. Acceptable plate count comparisons must be within 10%. Analyst deviations that are outside the 10% range are repeated. If the repeat inter-analyst count is unacceptable additional procedural training and method reviews are conducted.

11.3 Duplicate analyses are performed on 10% of samples or at least one sample per month for total and fecal coliform and *E.coli* tests. Due to the infrequent laboratory receipt of some samples, duplicate analysis is conducted per sample. If the RPD exceeds 20%, the data is qualified.

11.4 For membrane filtration analyses sterility control checks are conducted on the filter assembly at the beginning and end of each sequence and following every 10 samples analyzed. If QC blank fails, the run is rejected or qualified.

11.5 Verification of total coliform and fecal coliform colonies must be conducted monthly (10 colonies/month for wastewater). Colonies found in drinking water samples must have at least five typical sheen colonies and five atypical colonies verified.

11.6 For HPC analysis, duplicate plates are run for each dilution. A positive control and an uninoculated plate performed for each run. If the QC fails, the run is rejected or qualified.

12.0 DATA REDUCTION, VALIDATION AND REPORTING

12.1 DATA REDUCTION

The analyst performs the data calculation functions and is responsible for the initial examination of the finished data. Data reduction steps applied to the raw data are outlined in *SOP 030201 Data Handling and Reporting.* The primary analyst reviews the quality of data based on the following guidelines:

- The appropriate SOP has been followed
- Sample preparation is correct and complete
- Analytical results are correct and complete
- QC is within criteria and complete

12.2 VALIDATION

The validation process consists of data generation, reduction review, and reporting results. Once data reduction is complete, validation is conducted by reviewing all data entries and calculations for errors, reviewing all documentation to assure that sample information is correct, and that the tests have been performed appropriately and within the appropriate holding times. The secondary analyst reviews the quality of data based on the following guidelines:

- The appropriate SOP has been followed
- Sample preparation is correct and complete
- Analytical results are correct and complete

12.3 REPORTING

Reporting procedures are documented in *SOP 030201 Data Handling and Reporting.* Microbiological data is reported as Colony Forming Units (CFU) per unit volume, Presence/Absence, or Most Probable Number (MPN)/100mL.

13.0 CORRECTIVE ACTION

13.1 In the event that a nonconformance occurs in conjunction with the analytical batch, a corrective action response (CAR) must be completed. The reason for the nonconformance will be stated on the form and the measures taken to correct the nonconformance clearly defined. The effectiveness of the corrective action must be assessed and noted. The CAR will be kept on file by the QA department. Corrective action procedures are documented in SOP 030208 *Corrective and Preventive Action*

13.2 Required Corrective Action

All samples and procedures are governed by ESC's quality assurance program. Designated corrective actions are as follows:

13.2.1 Laboratory QC Criteria and Appropriate Corrective Actions

If the analytical method contains acceptance/rejection criteria and it is more stringent than those controls generated by the laboratory the method criteria will take precedence.

13.2.2 Out of control plate count comparisons between analysts.

Rejection Criteria – Comparisons must be within ±10% for monthly plate count comparisons.

Corrective Action – Duplicate counts are repeated. If repeat counts are still beyond acceptance range, procedural training and method reviews are conducted.

13.2.3 Out of control duplicate analyses for total and/or fecal coliform or *E.coli* .

Rejection Criteria – Duplicate RPDs must not exceed 20% for total and/or fecal coliform or *E.coli.*

Corrective Action – Data is qualified or the analysis is repeated. If repeat analysis is still beyond acceptance range, procedural training and method reviews are conducted.

13.2.4 Out of control QC blank for membrane filtration analysis.

Rejection Criteria – Blank analyses performed either at the beginning or end of the analytical sequence is positive.

Corrective Action – The analytical sequence may be rejected and reprocessed or qualified based on the nature of the contamination.

14.0 RECORD KEEPING

Record keeping is outlined in SOP #010103 *Document Control and Distribution,* SOP #030203 *Reagent Logs and Records* and SOP #030201 *Data Handling and Reporting*

15.0 QUALITY AUDITS

System and data quality audits are outlined in the ESC Quality Assurance Manual Version 8.0.

1.0 SIGNATORY APPROVALS

Mold Laboratory
QUALITY ASSURANCE MANUAL

APPENDIX XI TO THE ESC
QUALITY ASSURANCE MANUAL

for

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2.0 APPENDIX TABLE OF CONTENTS

3.0 SCOPE AND APPLICATION

This appendix discusses specific QA requirements for general analytical protocols to ensure that analytical data generated from the Mold laboratory are scientifically valid and are of acceptable quality. Any deviations from these requirements and any deviations that result in nonconforming work must be immediately evaluated and their corrective actions documented.

4.0 LABORATORY ORGANIZATION AND RESPONSIBILITIES

ESC Lab Sciences offers diverse environmental capabilities that enable the laboratory to provide the client with both routine and specialized services, field sampling and broad laboratory expertise. A brief outline of the organization and responsibilities as they apply to the ESC Quality Assurance Program is presented in Section 4.0 in the *ESC Quality Assurance Manual Version 8.0.*

5.0 PERSONNEL AND TRAINING

5.1 PERSONNEL

Dr. Christabel Fernandes-Monteiro, with a Ph.D. in Applied Biology, is the Department Manager of the Mold laboratory. She gained experience in Mold analytical techniques at ESC, an AIHA accredited laboratory, and obtained additional training in microscopic techniques at the McCrone Research Institute. Her responsibilities include sample analysis, protocol development and quality control. Dr. Fernandes-Monteiro oversees the review and approval processes of all data associated with the Mold laboratory. She also reviews AIHA and EPA online training modules related to the methods being performed in the Mold Laboratory. In her absence, David Cooper assumes responsibility for departmental decisions.

David Cooper, with a BS degree in Biological Sciences, is the Primary Analyst in the Mold laboratory. He is proficient in Mold analytical methods as per AIHA guidelines. David has gained analytical experience at ESC, an AIHA accredited laboratory, and obtained additional training in Mold analysis at the McCrone Research Institute. He reviews AIHA and EPA online training modules related to the methods being performed in the Mold Laboratory.

5.2 TRAINING

All new analysts to the laboratory are trained by the Primary Analyst or Manager according to ESC protocol. ESC's training program is outlined in SOP #350355, *Technical Training and Personnel Qualification for Biomonitoring-Mold*. Performance for BOD analysis is documented using an initial demonstration of capability (IDOCs) and continuing demonstration of capability (CDOC). On-going acceptable capability in mold analysis is demonstrated by acceptable participation in the AIHA proficiency testing programs (EMPAT). On-going acceptable capability in BOD analysis is demonstrated by acceptable participation in the WP proficiency testing program and daily Quality Control sample analyses. Documentation of analyst training, including a copy of college transcripts or degree, is maintained on file within the department.

6.0 FACILITIES AND LABORATORY SAFETY

6.1 FACILITIES

MOLD LAB

The main area of the MOLD laboratory has approximately 532 square feet with 167 square feet of bench space. The lighting throughout the laboratory is fluorescence. The air system is a five-ton Trane split unit with natural gas for heating. The laboratory reagent water is provided through the ELGA PureLab Ultra deionizer system. Biohazard containers are located in the laboratory and Commodore Waste Removal serves as ESC'S biological waste disposal contractor. ESC's building information guides and site plan are shown in Appendix I.

BOD LAB

The main area of the BOD laboratory has approximately 532 square feet of area with 151 square feet of bench space. The lighting standard throughout the laboratory is fluorescence. The air system is a five-ton Trane split unit with natural gas for heating. The laboratory reagent water is provided through the ELGA PureLab Ultra deionizer system. Biohazard containers are located in the laboratory and Commodore Waste Removal serves as ESC'S biological waste disposal contractor. ESC's building information guides and site plan are shown in Appendix I.

6.2 LABORATORY SAFETY

- Laboratory access is limited when work is being performed.
- All procedures where infectious aerosols or splashes may occur are conducted in biological safety II cabinets.
- The following Biosafety Level 2 (BSL2) guidelines are adhered to:
  - Closed-toe shoes are worn in the laboratory
  - Floors and work surfaces are cleaned on a regular basis
  - Emergency numbers are posted in the laboratory
  - Biological safety hoods are tested and certified annually
  - Laboratory personnel are trained in the use of the biological spill kit and emergency safety equipment
- ESC's laboratory safety guidelines are detailed in the ESC *Chemical Hygiene and Safety Plan*.

7.0 SAMPLING PROCEDURES

7.1 FIELD SAMPLING PROCEDURES, SAMPLE STORAGE, AND HANDLING

- Field Sampling procedure is described in Appendix III of this ESC Quality Assurance Manual. Sample information is recorded and kept on the ESC chain of custody and field logbooks.
- Samples are received in the laboratory login area and are tracked using LIMS (Laboratory Information Management System). A Chain of Custody Form accompanies all samples received by the lab. This is necessary to prove the traceability of the samples and to document the change in possession from sampling to delivery to receipt by the laboratory. Prior to analysis samples are checked for integrity. Sample handling, tracking and acceptance procedures are outlined in SOP #060105, *Sample Receiving.*
- Sample storage procedures are followed using guidance from each approved method and associated department SOP.

8.0 EQUIPMENT

8.1 EQUIPMENT LIST

LABORATORY EQUIPMENT LIST: MAJOR ITEMS – Mold/ BOD Analysis
This table is subject to revision without notice

Item	Manufacturer	*Model*	*Serial #*	*Location*
Analytical Balance	Mettler	PL602-S	1125081657	Bacteriology Lab
Analytical Balance	Ohaus	Adventure Pro	8029211055	Bacteriology Lab
Autoclave	Tuttnauer	2540EK	2906170	Mold Lab
Class I BSC	AirFiltronix	AirFiltronix HS 4500	41031	Mold Lab
Class II BSC	Labconco	Labconco 36213	60554894	Mold Lab
Class II BSC	Labconco	Labconco 36209	03076555	Bacteriology Lab
COD Reactor	HACH	45600	900903221	BOD
Microscope	NIKON	LABOPHOT	242008	Mold Lab
Microscope	NIKON	LABOPHOT	235267	Mold Lab
Microscope	Olympus	CH2	900216	Mold Lab
Microscope	Olympus	BH-2	708821	Mold Lab
Microscope	Leitz	Laborlux	512663	Mold Lab
Microscope	VWR Scientific	VWRC1	V167173	Mold Lab
Refrigerator	Whirlpool			Bacteriology Lab
Refrigerator	Whirlpool	El05PPXMQ	EEP3524864	Mold Lab
Refrigerator	Whirlpool	EL7ATRRMQ07	EWR4973976	Mold Lab
Refrigerator	Frigidaire	FRT17G4BW9	BA703306	Mold Lab
Stereoscope	VWR Scientific	VWRS1	V168430	Mold Lab
Incubator	Precision Scientific	FV199LRW2	WB02401046	Mold Lab
Incubator	Quincy Lab	10-100	I11-2454	Mold Lab
Incubator	Precision Scientific	30M	9303590	Bacteriology Lab
Incubator	Precision Scientific	30M		Bacteriology Lab
Incubator	VWR	2030	802202	BOD
Incubator	Fisher	Not Visible	100212	BOD
Incubator	Thermo Scientific Precision	3271	317217-1241	BOD
Incubator	Precision	818	35AK-10	BOD
Waterbath	VWR	129PC	1000797	Mold Lab
Waterbath	Blue M-MagniWhirlpool	MW-1110A	14991	Bacteriology Lab
Biolog MicroStation	Biolog, Inc.	Micrologg 3	342689	Bacteriology Lab
Turbidimeter	Biolog, Inc.	21907	6093898	Bacteriology Lab
Plate Reader	Biotek	ELX808BLG	203222	Bacteriology Lab
Vortex Genie2 Mixer	VWR	G-560	2-223236	Mold Lab
Vortex Genie2 Mixer	VWR	G-560	2-223236	Bacteriology Lab
Stir Plate	Corning	PC-420D	023507102961	Bacteriology Lab

LABORATORY EQUIPMENT LIST: MAJOR ITEMS – Mold/ BOD Analysis ***This table is subject to revision without notice***				
Item	**Manufacturer**	***Model***	***Serial #***	***Location***
Stir Plate	Fisher	118	102	Bacteriology Lab
Stir Plate	VWR	205	7852	BOD
Stir Plate	VWR	220	5031	BOD
BOD SP Robotic Analyzer	Skalar	SP50	08124	BOD
BOD SP Robotic Analyzer	Skalar	SP50	08123	BOD
DO meter	YSI	5000	081C101451	BOD
DO meter	YSI	5000	081C101450	BOD
pH meter	Thermo	Orion 3 star	BOD pH	BOD
Spectrophotometer	Hach	DR 4000U	Not available	BOD

8.2 EQUIPMENT PREVENTIVE MAINTENANCE

INSTRUMENT	***P. M. DESCRIPTION***	***FREQUENCY***
Analytical Balances	•Check with Class "I" weights	Daily-tolerance 1 gm - ±0.0001gm
Analytical Balances	•Service/Calibration (semiannual contract maintenance and calibration check)	10 gm - ±0.01 gm
Analytical Balances	•Service/Calibration (semiannual contract maintenance and calibration check)	Semiannually
Refrigerators & Incubators	•Maintenance service	As needed - determined by daily temperature performance checks
Water Bath	•Check thermometer vs. NIST	Once each year
Water Bath	•Remove from service when not maintaining temperature and send off for repair or replace	As needed
Autoclave	•Check sterilization efficiency	Weekly – *G. stearothermophilus*
Autoclave	•Check sterilization efficiency	Per Use – Chemical Indicator
Class II Biosafety Cabinet	•Monitor air and UV lamps	Monthly
Class II Biosafety Cabinet	•Inspect for air flow	Quarterly
Class II Biosafety Cabinet	•Recertification according to NSF standard 49	Annually
Turbidimeter	•Maintenance Service	Annually
Turbidimeter	•Check for accuracy using NIST traceable stds	Per Use
Biolog MicroStation	•Maintenance Service	Annually
Microscope	•Service/calibration of each ocular micrometer	Annually
Microscope	•Clean optics and stage, Kohler Alignment	Each Use
pH meters	Reference junction & electrode replacement	As needed
pH meters	Probe stored in KCl	At all times when not in use
pH meters	Other	As described in manufacturer's O
BOD SP Robotic Analyzer	Calibrate DO probe	Daily
BOD SP Robotic Analyzer	Clean and Change DO probe membrane	Every week
BOD SP Robotic Analyzer	Rinse ATU (seed) dispenser using rinse pump option	As needed
BOD SP Robotic Analyzer	Clean rinsing vessel	Every 3 months or as needed
BOD SP Robotic Analyzer	Replace tubing for dispenser, diluent pump, and rinsing vessel	Annually or as needed

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8.3 STANDARDS AND REAGENTS

Table 8.3A lists commercially prepared agar sources. Table 8.3 B lists in-house prepared agar sources and storage information. Table 8.3C lists standard sources, receipt, and preparation information for BOD Analysis. Table 8.3D is designed to provide general calibration range information for BOD analysis. These ranges may change depending on regulatory requirements, procedural changes, or project needs.

Table 8.3A: Commercially prepared agar sources and storage information.
(subject to revision as needed)

Agar Type	***Source***	***Storage***
Malt Extract Agar w/chloramphenicol (MEA)	HealthLink	4 ± 2°C
DG18 Agar	HealthLink	4 ± 2°C
Modified Cellulose Agar	HealthLink	4 ± 2°C
Potato Dextrose Agar w/chloramphenicol (PDA)	HealthLink	4 ± 2°C
Tryptic Soy Agar w/Sheep Blood	HealthLink	4 ± 2°C
R2A w/cycloheximide	HealthLink	4 ± 2°C
2 % Malt Extract	Biolog	4 ± 2°C
Biolog Universal Agar (BUG)	Biolog	4 ± 2°C
BUG w/BL	Biolog	4 ± 2°C
Biolog Universal Anaerobic Agar (BUA)	Biolog	4 ± 2°C
BUA w/BL	Biolog	4 ± 2°C
Biolog Universal Yeast Agar (BUY)	Biolog	4 ± 2°C
TSA w/SB contact	HealthLink	4 ± 2°C
BUG w/0.25% Maltose	Biolog	4 ± 2°C
Malt Extract Agar w/chloramphenicol contact	HealthLink	4 ± 2°C
Chocolate Agar	Biolog	4 ± 2°C
Czapek Yeast Extract Agar	HealthLink	4 ± 2°C

All stock agar expirations are per manufacturer specification.

Table 8.3B: In-house prepared agar sources and storage information.
(subject to revision as needed)

Agar Type-Stock	*Source*	*Stock Storage*	*Stock Expiration*	*Preparation Components Media*	*Prepared Storage*	*Prepared Expiration*
Malt Extract Agar (MEA)	Fisher/Difco	Room Temp	As specified by Manufacturer	MEA + Water	4 ± 2°C	3 weeks
Potato Dextrose Agar (PDA)	Fisher/Difco	Room Temp	As specified by Manufacturer	PDA + Water	4 ± 2°C	3 weeks
Corn Meal Agar (CMA)	Fisher/Difco	Room Temp	As specified by Manufacturer	CMA +Water	4 ± 2°C	3 weeks
Inhibitory Mold Agar (IMA)	Fisher/Difco	Room Temp	As specified by Manufacturer	IMA + Water	4 ± 2°C	3 weeks
Modified Saboraud's Agar (MSA)	Fisher/Difco	Room Temp	As specified by Manufacturer	M-SAB Dex + Water	4 ± 2°C	3 weeks
R2A	Fisher/Difco	Room Temp	As specified by Manufacturer	R2A + Water	4 ± 2°C	3 weeks
2 % Malt Extract	Fisher/Oxoid	Room Temp	As specified by Manufacturer	Bacteriological Agar + Malt	4 ± 2°C	3 weeks
Biolog Universal Agar (BUG)	Biolog	Room Temp	As specified by Manufacturer	BUG + Water	4 ± 2°C	3 weeks
Biolog Universal Anaerobic Agar (BUA)	Biolog	Room Temp	As specified by Manufacturer	BUA + Water	4 ± 2°C	3 weeks
Biolog Universal Yeast Agar (BUY)	Biolog	Room Temp	As specified by Manufacturer	BUY + Water	4 ± 2°C	3 weeks
Biolog Universal Agar (BUG) with 0.25%	Biolog	Room Temp	As specified by Manufacturer	BUG + Water + Maltose	4 ± 2°C	3 weeks

Table 8.3C: Standard sources, description and calibration information.
(This table is subject to revision without notice)

Instrument Group	*Standard Source*	*How Received*	*Source/Storage*	*Preparation from Source*	*Lab Stock Storage*	*Preparation Frequency*
BOD	Lab preparation	As dry glucose and glutamic acid	Dessicator	150mg each/L	4 ± 2°C	Made fresh daily
pH meter	Commercial source	pH 7.0 buffer	Ambient	No prep required	NA	Annual/Expiration Date
pH meter	Commercial source	pH 10.0 buffer	Ambient	No prep required	NA	Annual/Expiration Date
Turbidity meter	Commercial source	Turbidity standard	Ambient	No prep required	NA	Annual/Expiration Date

Table 8.3D: Working Standard Calibration

Analysis	*Calibration Standard*
BOD	D.O.- Barometric pressure/temp, Glucose and glutamic acid reference standard

Source of Fungi

A collection of fungi is maintained in the laboratory as training and reference material. The fungi are isolated from various sources and stored as Malt Extract Agar slants for 3 months at 4 ± 2ºC. Cultures are sub-cultured every 3 months. Each culture is assigned an accession number, genus, specific epithet, authority, source, and name of collector. Records are maintained in the laboratory in the accession list database.

8.4 INSTRUMENT CALIBRATION

Autoclave

Operating temperature is checked and recorded with each use with a minimum/maximum thermometer. Performance is tested weekly with *Bacillus stearothermophilus* ampoules. Chemical strips are used with each use to verify that supplies and materials have been sterilized. Records of autoclave operations are maintained for every cycle. Records include: date, contents, maximum temperature reached, pressure, time in sterilization mode, total run time (may be recorded as time in and time out) and analyst initials.

Incubators & Waterbaths

The record of temperature checks is documented twice daily at least 4 hours apart when in use. Thermometers used for temperature checks are verified at least annually.

Analytical Balances

Analytical balances are checked at least daily prior to each use with class "I" weights. Records of these verifications are maintained within the laboratory. Balances are also serviced and verified and/or calibrated by an external calibration service at least semi-annually.

Microscope

A record of cleaning and alignment for each microscope is maintained in the laboratory. Each microscope has an ocular micrometer that is verified annually with a stage micrometer. All microscopes are calibrated annually by an external calibration service.

Biochemical Oxygen Demand Robotic Analyzer – SOP Number 340303A

The Dissolved oxygen meter is calibrated according to manufacturer's instructions with each use. Air calibration is performed on the DO meter probes to correct DO for the ambient temperature and pressure. The air calibration is confirmed daily using the Winkler Test. During the analytical sequence, the calibration stability of the DO probes is verified after every ten samples and at the end of sequence, by the analysis of continuing calibration verification (CCV). If either of the readings differs from the initial readings by more than 0.2 mg DO/L., the instrument automatically re-calibrates the DO meters and re-reads everything after the last passing CCVs.

A laboratory control sample (LCS) is prepared from glucose and glutamic acid, and is analyzed exactly like a field sample at the beginning of the workgroup, after every twenty samples throughout the, run and at the end of the workgroup, one for each probe to verify that the analytical process is performing accurately.

pH meter

With each use of pH meters, calibrate the instrument according to manufacturer's instructions. The slope is documented on a daily basis. Acceptable pH slope range is 95-105%.

Turbidimeter

With each use, calibrate instrument according to manufacturer's instructions. Adjust transmittance to a 100% using a blank reference test tube. Establish appropriate turbidity range on turbidimeter by adding or subtracting 2% T to the percent transmittance measured with appropriate turbidity standard.

Volumetric equipment

Equipment such as pipettes non-Class A and other containers with graduations are calibrated once per lot prior to first use. Volumetric equipment that is not disposed off after use is calibrated on an annual basis. The error of calibration must not exceed 2.5%.

9.0 LABORATORY PRACTICES

9.1 REAGENT GRADE WATER

Reagent Grade water –Type II used in the Mold Laboratory is periodically checked for contamination. Type II water is checked annually for single and total heavy metals. Monthly checks for total organic carbon, ammonia and organic nitrogen, total residual chlorine and a heterotrophic plate count are also conducted. Conductivity and pH are checked continuously or with each use.

Prior to first use, a sterility check with non-selective growth media is performed on each batch of dilution and rinse water prepared in the laboratory and on each batch of commercially prepared water.

9.2 GLASSWARE WASHING AND STERILIZATION PROCEDURES

Glassware washing and preparation/sterilization procedures are performed according to EPA guidelines and are outlined in SOP #030701, *Glassware Cleaning*. The glassware used in the mold laboratory is restricted to microscopic slides, cover slips, and screw capped bottles, vials or flasks for preparation of media. Before use, examine microscope slides, and discard items with chipped edges or etched inner surfaces. Prior to use, clean microscopic slides with 70 % isopropyl alcohol. Examine screw-capped bottles, vials or flasks for chipped inner edges that could leak. Screw-capped bottles, vials or flasks are cleaned using the following protocol:

- Prewash with hot tap water. Wash with hot tap water. Wash with non-foaming powder detergent. Rinse with tap water. Rinse with DI water. Dry and cool.
- New glassware will be cleaned according to the same procedure as listed above.

Inspect glassware after washing for excessive water beading and re-wash, if necessary. Perform checks on pH and test for inhibitory residues on glassware and plastic ware. Use utensils and containers of borosilicate glass, stainless steel, aluminum, or other corrosion resistant material for media preparation. Sterilization of any auxiliary equipment is performed via autoclave.

Pipettes of all sizes are checked for sterility by drawing up non-selective media into the pipette and re-dispensing the volume back into original tube that contained the media. The tube is then incubated and monitored for growth. All results are recorded and maintained within the laboratory.

Inoculating loops are cultured by aseptically transferring the entire tip of the loop into a tube containing non-selective media. The tube is incubated and monitored for growth. Results are maintained within the laboratory.

BOD analysis is performed in disposable, pre-sterilized bottles. In the event that glass bottles must be used, the BOD glassware is washed in a commercial laboratory dishwasher using a phosphate free detergent, followed by a nitric acid rinse, with a final rinse of laboratory DI water.

9.3 MEDIA STERILITY VERIFICATION PROCEDURES

A sterility check must be analyzed for each lot of pre-prepared media and for each lot of media prepared in the laboratory. This is done prior to the first use of the media used for membrane filtration or MPN or pour plate and chromofluorogenic methods. For media used in the pour plate testing technique, sterility blanks of the media must be made by pouring an uninoculated plate for each run in addition to sterility and lot comparison tests being performed on each lot prior to first use. All results are documented.

9.4 POSITIVE AND NEGATIVE CONTROLS USING PURE CULTURES

Positive culture controls demonstrate that the media can support the growth of the target organism(s), and that the media produces the specified or expected reaction to the target organism(s). All prepared media must be tested with at least one pure culture of a known positive reaction. This must be done prior to first use of the media.

Negative culture controls demonstrate that the media does not support the growth of non-target organisms or does not demonstrate the typical positive reaction of the target organism(s). All batches of prepared selective media in the laboratory must be analyzed with one or more known negative culture controls. This must be done prior to first use of the media.

New lots of pre-prepared media are evaluated for suitability using manufacturer QC data.

10.0 ANALYTICAL PROCEDURES

A list of laboratory SOPs associated with the mold laboratory can be found in the following table:

TABLE 10.1: MOLD DEPARTMENT SOPs

This Table is subject to revision without notice

SOP #	Title
340303	Biochemical Oxygen Demand
340303A	Biochemical Oxygen Demand, Automated
350306	Spore Traps
350307	Fungal Anderson
350308	Fungal Quantification
350309	Fungal Rodac
350310	Direct Exam Prep Procedure
350311	Fungal Identification
350312	Mold QA/QC
350313	Mold Lab Safety
350314	MUG Ecoli/Coliforms
350319	Processing of Bacterial Andersen Samples for Quantification
350334	Microscope Usage
350335	Fungal Spore Identification
350342	BART Testing
350347	Processing of Bacterial Swabs, Bulk, Dust and Water Samples for Quantification
350349	Bacterial Identifiication Using Biolog
350357	Actinomycetes Identification
350367	Labconco Flaskscrubber Operation and Maintenance
350371	Mold lab Autoclave Maintenance and Operation
350372	Mold Lab Balance Calibration and Verification
350373	Preparation of Culture media

11.0 QUALITY CONTROL CHECKS

11.1 ESC participates in proficiency testing (PT's) in support of various laboratory accreditations/recognitions. For mold analyses, PTs are administered quarterly by AIHA. The samples are received and analyzed by method according to the vendor's instructions and according to the ESC SOP.

For BOD and *Total coliform/E. coli* analysis, environmental PTs are purchased from Environmental Resource Associates (ERA). The WP and WS studies are completed every 6 months.

11.2 As part of the total spore analysis QC, the laboratory maintains a slide collection with various count levels and genera/groups of spores. Acceptance criteria for the slide collection include counts that are statistically determined (e.g. ±3STD). Each analyst reviews one slide from this collection on each day of analysis. The slides are reviewed on a rotational basis such that a different slide is reviewed each day until the entire slide collection has been examined. The total spore count and acceptance criteria for each slide are calculated and compared with the statistically determined acceptance criteria.

11.3 Each week, a different pure culture is chosen by the lab supervisor and is identified by each analyst as part of training and continuing QC program.

11.4 Inter- and intra-analyst precision is determined by the re-analysis of samples by the same and different analysts (where possible). The rate of re-analysis by the same analyst and by a second qualified analyst is 5%.

11.5 Media blanks for viable count analysis are used to monitor media and laboratory procedures for contamination. These blanks are utilized in two ways:

- Laboratory media blanks are unexposed fresh media (either recently received from the manufacturer or newly laboratory prepared) that is incubated under the same conditions as those used for analysis.
- Field blanks are unopened media that is handled identically to field samples. These samplers are returned to the laboratory with sampled media to demonstrate that media utilized was not originally contaminated and did not become contaminated during transport.

11.6 Environmental monitoring of the laboratory air and the surfaces in the mold laboratory is performed monthly. BSLII hoods are also monitored in the mold laboratory.

11.7 Round Robin studies are performed for direct examination of fungal air samples in accordance with AIHA policy requirements. Results for these studies include raw counts and final concentrations for each fungal structure. Acceptance criteria include organism identification, ranking and quantification.

11.8 Analysts also participate in other continuing education activities, including attending seminars and conferences, in-house training meetings, reviews of journal publications and self-taught training on CD.

11.9 For BOD analysis, Initial Demonstrations of Capabilility (IDOC's) are performed during new analyst training and/or prior to acceptance and use of any new method/instrumentation. Continuing Demonstration of Capability must be updated at least annually. The associated data is filed within the department and available for review.

11.10 For BOD analysis, samples are analyzed in batches of 1-20 samples. Each batch must include the following: method blank, seed blank, seed control, seed check, 1 laboratory control sample, 1 sample duplicate/ 10 samples. A calibration check (CCV) is performed every 10 samples and an additional LCS every twenty samples including the end of the sequence.

11.11 A method blank is analyzed for each probe at the beginning and end of the sequence. The method blank is used to define the level of laboratory background and reagent contamination. Only one acceptable method blank is required for each batch. If all method blanks fail, data is qualified. The depletion of the method blank should be between - 0.2 and + 0.2mg DO/L.

11.12 The Seed Blank/Seed Control/Seed Check must deplete to show that the microorganism population is viable. The seed correction factor should be 0.6-1 mg/L

11.13 The CCV should not vary more than 0.2g DO/L within a run.

11.14 The BOD value for the LCS must be within 167.5 and 228.5.

11.15 The RPD for the sample duplicate must be $\leq$5%.

12.0 DATA REDUCTION, VALIDATION AND REPORTING

12.1 DATA REDUCTION

The analyst performs the data calculation functions and is responsible for the initial examination of the finished data. Data reduction steps applied to the raw data are outlined in SOP #030201, *Data Handling and Reporting.* The primary analyst reviews the quality of data based on the following guidelines:

- The appropriate SOP is followed
- Sample preparation is correct and complete
- Analytical results are correct and complete
- QC is within criteria and complete

For BOD analysis, the Quality Control Department performs the secondary review of the data package using the ESC SOP#030227, *Data Review*. The QC Reviewer verifies that the analysis has performed as required and meets method criteria, all associate data is present and complete, and also ensures that any additional documentation is completed as required (i.e. Ohio VAP checklists, required flags on test reports, etc.)

TABLE 12.1 Mold Data Reduction Formulas

PARAMETER	FORMULA
Non-viable (Spore Traps) Mold	$\frac{SporeCount}{m^3} = \frac{\text{number on trace} \times 1000}{\text{Volume of air sampled in liters}}$
Andersen Fungal Viable (Culturable) Mold Spore Andersen Bacterial Viable (Culturable) Bacteria	$\frac{CFU}{m^3} = \frac{\text{raw counts} \times 1000}{\text{Volume of air sampled in liters}}$ $P_c = N\,[1/N + 1/N\text{-}1 + 1/N\text{-}2 + \ldots\ldots 1/N\text{-}r + 1]$
Quantitative Fungal/Bacterial	$\frac{CFU}{gm}$ or $\frac{CFU}{Swab} = \frac{\#\text{ of Colonies} \times \text{Dilution Factor}}{\text{Sample Amount}}$
BOD, 5-DAY	$\frac{\text{Initial D.O.} - \text{Final D.O.} - CF}{\%\text{ Dilution Sample}}$ *Calculations are performed by computer software*

12.2 VALIDATION

The validation process consists of data generation, reduction review, and reporting results. Once data reduction is complete, validation is conducted by reviewing all data entries and calculations for errors, reviewing all documentation to assure that sample information is correct, and that the tests have been performed appropriately and within the appropriate holding times. The secondary analyst reviews the quality of data based on the following guidelines:

- The appropriate SOP is followed
- Sample preparation is correct and complete
- Analytical results are correct and complete

For BOD analysis, once data reduction is complete, validation is conducted by verification that the QC samples are within acceptable QC limits and that all documentation is complete, including the analytical report and associated QC. See Table 12.3 for current QC targets, controls and current reporting limits for BOD analysis.

12.3 REPORTING

Reporting procedures are documented in SOP #030201, *Data Handling and Reporting.*

BOD Control Limits: BOD QC targets are statutory. The laboratory calculated limits verify the validity of the regulatory limits. The BOD QC targets are within the range of 5 to 15% for accuracy, depending on determinative method requirements, and, where applicable, <20 RPD for precision, unless laboratory-generated data indicate that tighter control limits can be routinely achieved. When using a certified reference material for QC sample analysis, the acceptance limits used in the laboratory will conform to the provider's certified ranges for accuracy and precision.

Table 12.3: QC Targets for BOD Lab Accuracy (LCS), Precision and RL's *This table is subject to revision without notice*					
Analyte	**Analysis Method**	**Matrix**	**Accuracy Range (%)**	**Precision (RPD)**	**RL (ppb)**
Biochemical Oxygen Demand	SM5210B	W	85-115	≤5	5000
Biochemical Oxygen Demand - Carbonaceous	SM5210B	W	85-115	≤5	5000

13.0 CORRECTIVE ACTION

13.1 In the event that a nonconformance occurs in conjunction with the analytical batch, a corrective action response (CAR) form must be completed. The reason for the nonconformance is stated on the form and the measures taken to correct the nonconformance clearly defined. The effectiveness of the corrective action must be assessed and noted. The CAR will be kept on file by the QA department. Corrective action procedures are documented in SOP #030208, *Corrective and Preventive Action*

13.2 Required Corrective Action

Control limits have been established for each type of analysis. When these control limits are exceeded, corrective action must be taken. All samples and procedures are governed by ESC's quality assurance program. General corrective actions are as follows; however additional and more specific direction is provided in the specific determinative procedure. For more information, see the appropriate SOP.

13.2.1 Laboratory QC Criteria and Appropriate Corrective Actions

If the analytical method contains acceptance/rejection criteria and it is more stringent than those controls generated by the laboratory the method criteria will take precedence.

13.2.2 Out of Control RPD for inter- and/or intra-analyst reanalysis.

Rejection Criteria - RPD value of the original analysis is calculated and must be below the current control limit.

Corrective Action - Both first and second analysts re-analyze the sample until a consensus is reached and the RPD value falls within control limits.

13.2.3 Out of Control RPD for inter-analyst analysis.

Rejection Criteria – All organisms must be accurately identified.

Corrective Action - Both first and second analysts review the sample. The second analyst results are reported to the client.

13.2.4 Calibration Verification criteria are not met: BOD Analysis

Rejection Criteria see section 8.4

Corrective Action- If the CCV fails, the data may still be used. If the failure persists, check cleanliness of the equipment and stability of the DO probe for subsequent runs. If a problem persists, the group supervisor or QA Department is notified for further action.

13.2.5 Out of Control Blanks: Applies to Method Blank

Rejection Criteria- Blank depletion is greater than established limit.

Corrective action- If samples have already been partially prepared or analyzed, the group leader or QA will be consulted to determine if data needs to be rejected or if samples need to be re-prepped.

13.2.6 Out of Control Laboratory Control Standards (LCS)

Rejection Criteria- If the performance of associated laboratory control sample(s) is outside of lab-generated control limits calculated as the mean of at least 20 data points +/- 3 times the standard deviation of those points. (Listed in Section 12).

Corrective Action- All samples bracketed by the failed LCS must be reported with a qualifier.

13.2.7 Out of Control Duplicate Samples

Rejection Criteria- Lab-generated maximum RPD limit (as listed under precision in Section12)

Corrective Action- The sample and duplicate are reported with a qualifier.

14.0 RECORD KEEPING

Record keeping is outlined in SOP #010103, *Document Control and Distribution*, SOP #030203, *Reagent Logs and Records* and SOP #030201, *Data Handling and Reporting*

15.0 QUALITY AUDITS

System and data quality audits are outlined in the ESC Quality Assurance Manual Version 8.0.

1.0 SIGNATORY APPROVALS

Protozoa Laboratory
QUALITY ASSURANCE MANUAL

APPENDIX XII TO THE ESC
QUALITY ASSURANCE MANUAL

for

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NOTE: The QAM has been approved by the following people. A signed cover page is available upon request

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2.0 APPENDIX TABLE OF CONTENTS

3.0 SCOPE AND APPLICATION

This manual discusses specific QA requirements for EPA Methods 1622 and 1623 to ensure that analytical data generated from the protozoan laboratory are scientifically valid and are of acceptable quality. Any deviations from these requirements and any deviations that result in nonconforming work must be immediately evaluated and their corrective actions documented.

4.0 LABORATORY ORGANIZATION AND RESPONSIBILITIES

ESC Lab Sciences offers diverse environmental capabilities that enable the laboratory to provide the client with both routine and specialized services, field sampling and broad laboratory expertise. A brief outline of the organization and responsibilities as they apply to the ESC Quality Assurance Program is presented in Section 4.0 in the *ESC Quality Assurance Manual Version 8.0.*

5.0 PERSONNEL AND TRAINING

5.1 PERSONNEL

Kasey Stapler, with a B.S. degree in Biological Sciences, is the Principal Analyst for the Protozoan laboratory. Ms. Stapler is proficient in performing EPA Methods 1622 and 1623. She gained analytical experience from an accredited Protozoan laboratory and obtained additional training on microscopic techniques. Also, she frequently reviews EPA online training modules related to the methods being performed.

5.2 TRAINING

The certified analyst trains all new analysts to the Protozoan laboratory according to ESC protocol and EPA guidelines. ESC's training program is outlined in SOP #350405, *Training Protocol for Method 1622/1623.* Documentation of training received and authorizations to perform these analyses are maintained within the department.

6.0 FACILITIES AND LABORATORY SAFETY

6.1 FACILITIES

The main area of the laboratory is approximately 420 square feet and has roughly 67.5 square feet of bench area. The microscope dark room is located in the back of the laboratory is 36 square feet with 18 square feet of bench area. Additionally, there is 40 square feet of storage and fluorescent lighting throughout all areas. The air handling system is a five-ton Trane split unit with natural gas for heating. The laboratory reagent water is provided through the Siemans® deionizer system. Biohazard containers are located in the protozoan laboratory and Stericycle serves as ESC'S biological waste disposal contractor. ESC's building information guides and site plan are shown in Appendix I.

6.2 LABORATORY SAFETY

- Laboratory access is limited when work is being performed.
- All procedures where infectious aerosols or splashes may occur are conducted in biological safety II cabinets.
- The following Biosafety Level 2 (BSL2) guidelines are adhered to:
  - Closed-toe shoes are worn in the laboratory
  - Floors and work surfaces are cleaned on a regular basis
  - Emergency numbers are posted in the laboratory
  - Biological safety hoods are tested and certified annually
  - Laboratory personnel are trained in the use of the biological spill kit and emergency safety equipment
- ESC's laboratory safety guidelines are detailed in SOP #350408, *Biosafety Guidelines for the Cryptosporidium Laboratory*.

7.0 SAMPLING PROCEDURES

7.1 FIELD SAMPLING PROCEDURES, SAMPLE STORAGE, AND HANDLING

- A description of field sample collection, containers, storage, temperature, and transport times are located in SOP #350402, *Method 1622/1623 Field-Filtering Sample Collection and Laboratory Delivery* and SOP #350403, *Method 1622/1623 Bulk Sample Collection and Laboratory Delivery*.
- Laboratory sample identification, handling, tracking and the information recording system are found in the following procedures: SOP #350404, *Method 1622/1623 Sample Receiving* and SOP #060105, *Sample Receiving*.
- A Chain of Custody and LT2 Sample Collection Form accompanies all samples received by the lab. This is necessary to prove the traceability of the samples and to document the change in possession from sampling through receipt by the laboratory. Prior to analysis, all samples are checked for integrity.
- Following analysis, the slides are maintained for a minimum of 2 months and disposed of following all State and Federal regulations governing disposal.
- Requirements for sample acceptance is located in SOP #350404, Section 7.0, *Method 1622/1623 Sample Receiving*.

8.0 EQUIPMENT

Laboratory equipment specifications are outlined in SOP #350407, *Microscope Analyst Verification,* SOP #350410, *IEC CRU-500 Centrifuge Operation and Maintenance,* SOP #350411, *Lab-Line Multi-Wrist Shaker Operation and Maintenance* and SOP #350413, *Olympus BX40 Microscope Operation and Maintenance.*

8.1 EQUIPMENT LIST

LABORATORY EQUIPMENT LIST: MAJOR ITEMS - Protozoan		
Item	***Manufacturer***	***Model***
Flow control valve	Plast-o-matic	FC050B
Centrifugal pump	Jabsco	18610-0271
Graduated container	Nalgene	20 Liter Carboy
Laboratory shaker	Lab-Line	3587-4
Laboratory shaker side arms	Lab-Line	3589
1500 XG swinging bucket centrifuge	Damon/IEC Division	CRU-5000
Sample mixer/rotator	DYNAL	Cat#: 947.01
Magnetic Particle Concentrator	DYNAL	MPC-1
Magnetic Particle Concentrator	DYNAL	MPC-S
Magnetic Particle Concentrator	DYNAL	MPC-6
Flat-sided sample tubes	DYNAL	Cat#: 740.03
Epifluorescence/differential interference contrast microscope	Olympus	BX-40
Excitation/band pass microscope for fluorescein isothiocyanate (FTIC)	C-Squared	UN3100
Excitation/band pass filters for 4',6-diamidino-2-phenylindole (DAPI)	C-Squared	UN41001

8.2 EQUIPMENT PREVENTIVE MAINTENANCE, EQUIPMENT CALIBRATION

Calibration of equipment is conducted on an annual and/or semi-annual basis and is documented. Maintenance and cleaning is conducted on an as needed basis or per manufacturer's instructions. Equipment cleaning is specified in SOP #350412, *Cryptosporidium Laboratory Equipment Cleaning.*

8.3 STANDARDS AND REAGENTS

Table 8.3A: Stock solution sources, description and related information.
(subject to revision as needed)

Description	Vendor	Concentration	Storage Req.	Expiration
Sodium Hydroxide (NaOH)	Fisher	Concentrated	ambient	1 year
Hydrochloric Acid (HCl)	Fisher	Concentrated	ambient	1 year
Laureth-12	VWR	--	ambient	1 year
Tris Stock	Fisher	--	ambient	1 year
EDTA	Sigma	0.5 M, pH 8.0	1-10°C	1 year
Antifoam A	Sigma Chemical	--	ambient	1 year
Dynabeads® GC-Combo/Crypto	Dynal	--	0 ± 8°C	2 years
Direct labeling kit for det. of oocysts and cysts, Merifluor Cryptosporidium/Giardia	Meridian Diagnostics	--	0 ± 8°C	1 year
Phosphate Buffered Saline (PBS) Solution, pH 7.4	Sigma Chemical	--	ambient	1 year
4', 6-diamidino-2-phenylindole (DAPI) stain	Waterborne, Inc	2mg/mL	0 ± 8°C/Darkness	When positive control fails
Purified, live *Cryptosporidium* oocysts stock suspension	WSLH	--	0 ± 8°C	1 month
Purified, live *Giardia* cysts stock suspension	WSLH	--	0 ± 8°C	1 month

TABLE 8.3B: Working Solution Descriptions and Related Information.
(subject to change)

Solution	Concentrations	Storage Requirements	Expiration
Sodium Hydroxide (NaOH)	6.0 N	ambient	1 year
Sodium Hydroxide (NaOH)	1.0 N	ambient	1 year
Hydrochloric Acid (HCl)	6.0 N	ambient	1 year
Hydrochloric Acid (HCl)	1.0 N	ambient	1 year
Hydrochloric Acid (HCl)	0.1 N	ambient	1 year
Laureth-12 stock vials	10g/100mL	-10°C to –20°C	1 year
Tris Working Solution	1 M, pH 7.4	ambient	3 months
Elution Buffer	--	ambient	1 week
1X SL Buffer A Solution	--	0 ± 8°C	1 week
Staining 1X wash buffer	--	ambient	3 months
Phosphate Buffered Saline (PBS) Solution, pH 7.4	--	ambient	1 week
Working DAPI stain	10mL Stock/50ml Phosphate Buffer	Ambient/Dark container	1 day

9.0 LABORATORY PRACTICES

9.1 REAGENT GRADE WATER

ASTM Type I grade water: Siemans® supplies reagent grade water. Reagent water is analyzed for total chlorine, heterotrophic bacteria and specific conductance on a monthly basis. Reagent water is tested for metals: Lead, Cadmium, Chromium, Copper, Nickel, and Zinc on an annual basis.

9.2 GLASSWARE WASHING AND STERILIZATION PROCEDURES

Glassware washing and preparation/sterilization procedures are outlined in SOP #350414, *Steamscrubber Operation and Maintenance*, SOP #350408, *Biosafety Guidelines for Cryptosporidium Laboratory* and SOP #350412, *Cryptosporidium Laboratory Equipment Cleaning*.

Laboratory glassware and plastic ware are checked for acceptability prior to use. Glassware acceptance criteria are documented in SOP #350412, *Cryptosporidium Laboratory Equipment Cleaning*.

9.3 FILTER ACCEPTANCE

Each new lot of filters are checked for acceptability prior to use by performing method blanks (MB) and ongoing precision and recovery testing (OPR) on the lot.

10.0 ANALYTICAL PROCEDURES

10.1 A list of laboratory SOP's associated with the protozoan laboratory can be found in the following table:

TABLE 10.1: PROTOZOAN DEPARTMENT SOP'S

This Table is subject to revision without notice

SOP #	Title
350401	Isolation & Identification of *Giardia* and/or *Cryptosporidium* in Water
350402	Method 1622/1623 Field-Filtering Sample Collection and Laboratory
350403	Method 1622/1623 Bulk Sample Collection and Laboratory Delivery
350404	Method 1622/1623 Sample Receiving
350405	Training Protocol for Method 1622/1623
350406	Data Collection and Verification for Method 1622/1623
350407	Microscope Analyst Verification
350408	Biosafety Guidelines for *Cryptosporidium* Laboratory
350409	IPR, OPR and MS Spiking Procedures and Corrective Actions
350410	IEC CRU-5000 Centrifuge Operation and Maintenance
350411	Lab-Line Multi-Wrist Shaker Operation and Maintenance
350412	*Cryptosporidium* Laboratory Equipment Cleaning
350413	Olympus BX40 Microscope Operation and Maintenance
350414	Steamscrubber Dishwasher Operation and Maintenance

10.2 The following references are used for analytical procedures conducted in the laboratory:

- EPA. Method 1623: *Cryptosporidium* and *Giarda* in Water by Filtration/IMS/FA, December 2005.
- EPA. Method 1622: *Cryptosporidium* in Water by Filtration/IMS/FA, December 2005.
- EPA. Microbial Laboratory Guidance Manual for the Final Long Term 2 Enhanced Surface Water Treatment Rule. February 2006.

11.0 QUALITY CONTROL CHECKS

11.1 ESC participates in proficiency testing (PT) through the analysis of spiked vials received from Wisconsin State Laboratory of Hygiene (WSLH) and analyzed according to study instructions and the ESC SOP. When the analysis is completed, the results are reported to Computer Sciences Corporation (CSC). CSC issues the testing results as either a "pass" or "fail". If the laboratory fails a PT round, a follow-up test is performed in an attempt to meet the necessary requirements. If the follow-up test results in a second failure, the laboratory takes part in a re-training program offered by the EPA or another accredited laboratory.

11.2 An Ongoing Precision and Recovery sample (OPR) is analyzed once weekly or per 20 samples. The OPR is spiked with 100-500 cysts and/or oocysts from a spiking vial received from the WSLH. Recoveries from the OPR must fall within EPA approved QC limits: Oocyts = 22-100% and Cysts = 14-100%.

11.3 A Method Blank is also analyzed once weekly or per 20 samples. The Method Blank must be free of other test organisms and serves as a sterility control on the analytical system.

11.4 If either sample falls outside acceptance parameters, corrective action must be taken and the samples re-analyzed until the QC criteria are met. Client samples may only be analyzed following acceptable QC sample results. Quality control information is located in SOP #350409, *IPR (Initial Precision and Recovery), OPR (Ongoing Precision and Recovery) and MS (Matrix Spike sample), Spiking Procedures and Corrective Actions.*

11.5 Clients are required to send a duplicate sample early in their sampling schedule and then again for every 20 field samples collected. This duplicate is utilized in the laboratory as a Matrix Spike (MS). The MS is spiked in the same manner and with the same number of organisms as the OPR to determine the effects of the matrix on the analytical process.

11.6 Inter/intra-analyst precision is determined, at least monthly.

12.0 DATA REDUCTION, VALIDATION AND REPORTING

12.1 DATA REDUCTION

The analyst performs the data calculation functions and is responsible for the initial examination of the finished data. Data reduction steps applied to the raw data are outlined in SOP #350401, *Isolation and Identification of Cryptosporidium and/or Giardia in Water* and SOP #350406, *Data Collection and Verification for Method 1622/1623.*

12.2 VALIDATION

Guidelines for data validation are found in SOP #350406, *Data Collection and Verification for Method 1622/1623.* In general, data integrity involves reviewing all data entries and calculations for errors, reviewing all documentation to assure that sample information is correct, and that the tests have been performed appropriately and within the appropriate holding times. The secondary analyst reviews the quality of data based on the following guidelines:

- The appropriate SOP is followed
- Sample preparation is correct and complete
- Analytical results are correct and complete

12.3 REPORTING

Reporting procedures are documented in SOP #350406, *Data Collection and Verification for Method 1622/1623.* Depending on the needs of the client one or more of the following may be included: Case narrative, Chain of Custody, Internal Chain of Custody, Final Report, Raw Data, etc. When the package involves more than just QC forms, it must contain a Table of Contents and Pagination. When the package is complete, it must be reviewed first by the Primary Analyst followed by the Department Manager or second qualified analyst, and finally by the QC Department. The final review person signs that the information is complete and the package is ready for submission to the client. A copy of the final package must be kept on file.

13.0 CORRECTIVE ACTION

13.1 In the event that a nonconformance occurs in conjunction with the analytical batch, a corrective action response (CAR) must be completed. The cause of the event is stated on the form and the measures taken to correct the nonconformance clearly defined. The effectiveness of the corrective action must be assessed and noted. The CAR is kept on file by the QA Department. Corrective action procedures are documented in the SOP #350409, *IPR (Initial Precision and Recovery), OPR (Ongoing Precision and Recovery) and MS (Matrix Spike sample), Spiking Procedures.*

13.2 Required Corrective Action

13.2.1 If a spiked sample or set of samples fails to meet quality control limits

Rejection Criteria - Recoveries from the OPR fall beyond the approved QC limits: Oocyts = 22-100% and Cysts = 14-100%.

Corrective Action - Examine the spiking suspension organisms directly. To determine if the failure of the spike is due to changes in the microscope or problem with the antibody stain, re-examine the positive staining control, check Köhler illumination, and check the fluorescence and DAPI. To determine if the failure of the spike is attributable to the separation system, check the system performance by spiking a 10mL volume of reagent water with 100-500 cysts and/or oocysts and processing the sample through the IMS, staining and examination procedures. Recoveries should be greater than 70%. If the failure of the spike is attributable to the filtration/elution/concentration system, check the system performance by processing spiked reagent water according to the method and filter, stain and examine the sample concentrate. This process is performed until the cause of the failure is isolated and corrected. The sample then must be re-analyzed until acceptable results are achieved.

13.2.2 Method Blank contains positive organism when analyzed.

Rejection Criteria – The Method Blank must be free of test organisms and serves as a sterility control on the analytical system.

Corrective Action - Equipment used to process the sample may be cleaned and/or replaced. Reagents used to process the sample may be disposed of and new reagents purchased or prepared. New method blank is prepared and analyzed. This process is repeated until the method blank passes the acceptance criteria.

13.2.3 Inter/intra-analyst precision analyses are beyond ±10%.

Rejection Criteria – Results for inter and/or intra-analyst precision must be within 10% of original results.

Corrective Action - The differences are discussed between analysts until a consensus is found.

14.0 RECORD KEEPING

Record keeping is outlined in SOP #010103, *Document Control and Distribution*, SOP #030203, *Reagent Logs and Records* and SOP #030201, *Data Handling and Reporting*

15.0 QUALITY AUDITS

System and data quality audits are outlined in the ESC Quality Assurance Manual Version 8.0.

End Of Document

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SOP Revision Summary

SOP:					
Author -	Nancy Winters	**Number -**	330351	**Department -**	Volatiles
Title -	BTEX (Method 8021B, 602, SM6200C 20th) AND GASOLINE RANGE ORGANICS (Method 8015B, 8015C, 8015D) by GC (With provisions for Calif-Lo, NWTPH-Gx, OA1, WI GRO (synthetic), Wyoming LAUST Req., GRO Louisiana, GRO by OHVAP, AK101 GRO)				
Revision -	R14	**Rev. Date -**	3/23/12		

This Standard Operating Procedure has been amended to include changes required during normal business operations. These changes as defined by SOP 010103 (Document Control and Distribution) are routine modifications that will be incorporated into the SOP upon the next scheduled review.

Rev.	Date	Section	Brief Description
1	6/6/12	8.0 & 14.4	Update for Wyoming Requirements

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(see Revision History at the end of this document for more information)

TITLE: BTEX (Method 8021B, 602, SM6200C 20th) AND GASOLINE RANGE ORGANICS (Method 8015B, 8015C, 8015D) by GC (With provisions for Calif-Lo, NWTPH-Gx, OA1, WI GRO (synthetic), Wyoming LAUST Req., GRO Louisiana, GRO by OHVAP, AK101 GRO)

SOP NUMBER: 330351

Prepared by: Nancy Winters

Reviewed by: JD Gentry/Dixie Marlin

This document bears a watermark in the bottom right hand corner. The watermark is an insignia of the document's approval. The signed original is on file in the Reg. Affairs Office.

_____
Department Manager

_____
QA Department

1.0 SCOPE AND APPLICATION

STATE NOTE: For samples analyzed in conjunction with the Ohio Voluntary Action Program (VAP) please utilize SOP# 330351, Revision 13 (2/23/09).

CLIENT NOTE: For samples analyzed in conjunction for Gasoline Range Organics from Marathon/MPC LLC/SSA, please utilize ESC SOP# 330361A, except where other specific state regulatory requirements are required.

1.1 BTEXM/GRO by gas chromatograph determines the concentration of benzene, toluene, ethylbenzene, m, p & o-xylene, MTBE, and gasoline range organics (C_6 - C_{10}) (range defined in Method 8015B) in solution. All matrices, including groundwater, aqueous samples, TCLP extracts, wastewater, soils, sludge, sediments, and other solid wastes, can be analyzed by this method. Wisconsin GRO and AK101 GRO are determined by this method. Samples analyzed by the GRO-Louisiana method are quantitated using a carbon range of C_6 – C_{12}.

NOTE: Quantitation for samples from Alaska (AK101) must use GRO range from the beginning of the C_6 peak to the beginning of the C_{10} peak.

1.2 The data shown in Attachment II provides the reporting limits for analytes in clean aqueous samples for each instrument currently running this method.

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TITLE: BTEX (Method 8021B, 602, SM6200C 20th) AND GASOLINE RANGE ORGANICS (Method 8015B, 8015C, 8015D) by GC (With provisions for Calif-Lo, NWTPH-Gx, OA1, WI GRO (synthetic), Wyoming LAUST Req., GRO Louisiana, GRO by OHVAP, AK101 GRO)

1.3 An MDL study must be completed at least annually or more frequently if major instrumentation changes occur. Method Detection Limits (MDLs) are performed based on ESC SOP #030206. Updated MDL records are filed and stored in a central location within the department.

1.3.1 Limit of Detection (LOD) and Limit of Quantitation (LOQ) studies are completed at the frequency required by the TNI standard per the procedure identified in the ESC SOP #030206, *Method Detection Limits (MDL) and Limits of Detection (LOD).* Should the procedure be utilized for DOD support; then the frequency of these studies must meet the requirements of the current DOD QSM.

2.0 METHOD SUMMARY AND DEFINITIONS

2.1 Samples (except for those to be prepared by 5035A) require no preparation before analysis unless the concentration of the analyte is great enough to require a waste or serial dilution.

Samples being analyzed by 5035A, an aliquot of the methanol extract is used to prepare the necessary dilution in 5mL of DI water.

STATE NOTE: All samples analyzed for Ohio VAP must to be prepared by Method 5035A.

2.2 This SOP describes the determination of concentrations of benzene, toluene, ethylbenzene, MTBE, and m, p, & o-Xylene by PID and gasoline range organics by FID. This method uses purge and trap to determine these concentrations. The BTEXM compounds and GRO concentrations are determined by internal standard calibration using fluorobenzene as the internal standard. Sample Introduction Method: The volatile compounds are introduced into the gas chromatograph by the EPA Purge-and-Trap Method 5030, SW-846.

2.2.1 Samples are placed in vials and purged with helium gas. The purged volatile compounds are transported to a trap (Supelco Purge Trap G) that is at 40°C. The trap is rapidly heated at the end of the purge cycle to 200°C and the volatile compounds desorb to the capillary column. After passing through this column, the compounds first pass by the PID, which detects double bonds, and then by the FID, which detects compounds that burn. As they pass by these detectors, an electrical signal is transmitted to a computer or integrator and causes an electrical peak to be recorded. The area underneath these peaks can be compared to known concentrations to determine the concentrations of BTEXM compounds in the sample.

2.3 Gasoline Range Organics (GROs) - Correspond to the range of alkanes from C_6 to C_{10} and covering a boiling point range of approximately 60°C – 170°C

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2.4 Initial Calibration Verification (ICV)/Continuing Calibration Verification (CCV) – Standards prepared from the primary source that are analyzed at the beginning of each workgroup (ICV), and following every 10 samples throughout the run and at the conclusion of the sequence (CCV) to confirm that the instrument maintains calibration stability within acceptable limits

2.5 Continuing Demonstration of Capability (CDOC) – At least annual verification of analyst continued ability to perform method acceptably.

2.6 Duplicate – A second aliquot of sample that is treated the same as the original sample in order to determine the precision of the method.

2.7 Initial Demonstration of Capability (IDOC) - A demonstration of capability (DOC) must be made prior to using any analytical method and any time there is a change in instrument type, personnel or testing method. Such performance must be documented and the four preparation batches following the change in personnel must not result in the failure of any batch acceptance criteria, e.g., method blank and laboratory control sample, or the demonstration of capability must be repeated. See also Continuing Demonstration of Capability (CDOC).

2.8 Laboratory Control Sample (LCS) / Laboratory Control Sample Duplicate (LCSD) – Duplicate aliquots of a control sample of known in composition. This sample is prepared from a source that is different from the stock used to prepare the initial and continuing calibration standards. LCS/LCSD are analyzed exactly like a sample and the purpose is to determine whether the methodology is in control and whether the laboratory is capable of making accurate and precise measurements. Method precision can be determined using the results of the LCS/LCSD analysis.

2.9 Matrix Spike (MS) / Matrix Spike Duplicate (MSD) - Two aliquots of a field sample (water or soil) spiked with known quantities of specific compounds and subjected to the entire analytical procedure in order to indicate the appropriateness of the method for the matrix by measuring recovery. Method precision can be determined using the results of the MS/MSD analysis, but are subject to matrix variability issues not present in the LCS/LCSD pair.

2.10 Method Blank - An analytical control consisting of all reagents used in the analytical procedure. The method blank is used to define the level of laboratory background and reagent contamination.

2.11 Second Source Calibration Verification (SSCV) – Analytical standard run after each calibration to insure proper creation of primary calibration standards. This standard must be from independent manufacture of primary calibration standards.

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2.12 Reporting Limit (RL) – Also see Practical Quantitation Limit (PQL). Routinely the reporting limit is the lowest standard of the calibration curve. Technically, the reporting limit is the lowest level that can be reliably achieved within the established limits of precision and accuracy during routine laboratory operating conditions.

2.13 Practical Quantitation Limit (PQL) – The default reporting limit when other limits are not specified by the client or project. The PQL is usually a factor of 3-10 times the MDL.

2.14 Method Detection Limit (MDL) - The minimum concentration of a substance that can be analyzed with 99% confidence that the analyte concentration is greater than zero.

2.15 Practical Quantitation Limit (PQL) – The default reporting limit when other limits are not specified by the client or project. The PQL is usually a factor of 3-10 times the MDL.

2.16 Internal Standard (ISTD) – Analytes not expected to occur naturally in field samples that are spiked to provide a consistent basis for use in internal calibration models.

2.17 Internal Calibration – Internal standard calibration involves the comparison of instrument responses from the target compounds in the sample to the responses of specific spikes added to the sample or sample extract prior to injection.

2.18 Response Factor (RF) - The ratio of the peak area (or height) of the target compound in the sample or sample extract to the peak area (or height) of the relevant internal standard in the sample or sample extract.

2.19 Surrogate - A compound, similar to the target analytes in chemical composition and behavior, but not expected to occur naturally in field samples. Analytes are spiked by preparation/analytical personnel to assess sample extraction and analytical efficiency in each individual field sample.

2.20 Elution - The order of emergence of chemicals from the column of a chromatograph. The chemicals then typically flow into a detector of some type. Predicting and controlling the order of elution is a key aspect of column chromatographic methods and can be modified using instrument operating conditions, column selections, etc.

2.20.1 Co-elution – Peaks that are not distinctly separated or resolved by a chromatograph. Co-elution is problematic when peaks share primary and secondary mass ions making accurate quantitation questionable.

2.21 Retention Time – The expected time that it takes for a particular analyte to pass through the system (from the column inlet to the detector) under set conditions.

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2.22 External Calibration - External standard calibration involves comparison of instrument responses from the sample to the responses from the target compounds in the calibration standards. Sample peak areas (or peak heights) are compared to peak areas (or heights) of the standards. The ratio of the detector response to the amount (mass) of analyte in the calibration standard is defined as the calibration factor (CF).

2.23 Calibration Factor (CF) - The ratio of the detector response (peak areas or peak heights) to the amount (mass) of analyte in the calibration standard.

2.24 Reporting Limit Verification (RLV) – A standard analyzed following initial calibration/calibration verification at or below the analyte concentration of the routine reporting level. It is analyzed per regulatory/method requirements for drinking water analyses and various other state/national regulatory programs to verify the accuracy of field sample results at the reporting level.

2.25 Calibration Standards – Solutions of known concentrations used to create graphic representation of the relationship between the known values, such as concentrations, and instrument responses.

2.26 Linear Regression - Mathematical technique for finding the straight line that best-fits the values of a linear function, plotted on a scatter graph as data points. If a 'best fit' line is found, it can be used as the basis for estimating the future values of the function by extending it while maintaining its slope.

2.27 Limit of Detection (LOD) - A laboratory's estimate of the minimum amount of an analyte in a given matrix that an analytical process can reliably detect in their facility. The validity of the LOD shall be verified by detection of the analyte(s) in a spiked clean matrix sample in each quality system matrix. This sample shall contain the analyte at no more than 3X the MDL for single analyte tests and 4X the MDL for multiple analyte tests. This verification shall be performed on every instrument that is to be used for analysis of samples and reporting of data. The samples used for this verification must be prepared and analyzed through all steps in the analytical process used for client samples.

2.28 Limit(s) of Quantitation (LOQ) - The minimum levels, concentrations, or quantities of a target variable (e.g., target analyte) that can be reported with a specified degree of confidence. The validity of the LOQ shall be verified by successful analysis of a spiked clean matrix sample containing the analytes of concern in each quality system matrix at 1 to 2 times the claimed LOQ. A successful analysis is one where the recovery of each analyte is within the laboratory established method acceptance criteria or client data quality objectives for accuracy. The samples used for this verification must be prepared and analyzed through all steps in the analytical process used for client samples.

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3.0 HEALTH AND SAFETY

3.1 The toxicity or carcinogenicity of each reagent used in this method has not been fully established. Each chemical should be regarded as a potential health hazard and exposure to these compounds must be as low as reasonably achievable. Each laboratory is responsible for maintaining a current awareness file of OSHA regulations regarding the safe handling of the chemicals specified in this method. A reference file of material data handling sheets should also be made available to all personnel involved in the chemical analysis. Specifically, concentrated nitric and hydrochloric acids present various hazards and are moderately toxic and extremely irritating to skin and mucus membranes. Use these reagents in a fume hood whenever possible and if eye or skin contact occurs, flush with large volumes of water. Always wear safety glasses or a shield for eye protection, protective clothing and observe proper mixing when working with these reagents.

3.2 Use of this procedure requires the handling of samples and standards containing volatile organic compounds. Use of laboratory safety glasses and protective gloves are required.

3.3 For specific information regarding the toxicity of the compounds used in this procedure and other related health and safety issues including the proper storage and handling of reagents and chemicals, the analyst should consult the appropriate Material Safety Data Sheets (MSDS). These are located in a notebook at the Safety Station located in each building.

4.0 SAMPLE PRESERVATION, CONTAINERS, HANDLING, AND STORAGE

4.1 All samples must have been collected using a sampling plan that addresses the considerations of this method.

4.2 Holding Times, Storage and Container Requirements

4.2.1 Water (EPA 5030)

Aqueous samples must be collected in triplicate in 40mL vials with the pH adjusted to <2 with HCl and stored at 4°C ±2°C. Sample must be analyzed within 14 days of collection. Samples not preserved with HCl must be analyzed within 7 days of collection.

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4.2.2 Soil (EPA 5035A)

High Concentration - Collect an additional sample using the Encore sampler or equivalent.

4.2.2.1 Eject the sample into a 40mL pre-weighed vial containing 5mL of methanol. This vial can be supplied pre-weighed by the laboratory. The vial is sealed and placed in cold storage at 4°C ±2°C.

If a vial with 5mL of methanol is not available, the soil sampler is capped and placed in a foil bag. This sample must arrive at the laboratory within 48 hours. When the Encore arrives at the lab, the sample is ejected from the Encore into a pre-weighed (to 0.01g) vial containing 5mL of methanol then placed in cold storage of 4°C ±2°C.

If specially prepared vials were not available at the sampling site, a soil sample packed into a 125mL jar or brass core sample tube can be used. At the analysis site, approximately 5g of soil is removed from the sample and placed in a tared 40mL vial. 5mL of reagent water and a stir bar are added.

4.2.2.2 Collect additional duplicate aliquots of each sample in 40mL glass vials (septum sealed), 125mL glass container, brass capped core sample tube or equivalent for sample screening, dry weight determination, and any additional high concentration evaluation required.

STATE NOTE: Soil and Water samples received from the states of Missouri or Kansas may be preserved with tri-sodium phosphate and have a resulting pH of 14.

STATE NOTE: For Ohio VAP samples, Encore samplers must be collected in the field and shipped to the laboratory to be frozen within 48 hours of collection. These samples must remain frozen until analysis and do not require additional chemical preservation. The holding time for these samples remains at 14 days from the time of collection. The holding time begins at collection and ends at 14 days.

4.3 **Glassware:** All glassware must be pre-washed with detergent and rinsed with deionized water. Refer to SOP#: 030701, *Glassware Cleaning*.

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4.4 **STATE NOTE:** AK101 Soil/Sediment Collection Procedure - Soils and Sediments: Soil and sediment samples require special procedures to minimize the loss of volatile organic compounds during transit from the field to laboratory. **Please note that this sample preservation is different from SW-846 Method 8021B. The use of sodium bisulfate as a preservative is not acceptable.**

4.4.1 Soil or sediment samples must be collected into appropriately sized containers and submerged in surrogated methanol.

4.4.2 Solid samples must be collected with minimum disturbance into tared jars with a Teflon-lined septum fused to the lid. Jars should be 4oz. or larger. 25mL aliquots of methanol (includes 1.2mL of a surrogate solution at 50µg/mL) are carefully added to the undisturbed soil until the sample is submerged.

4.4.3 It is extremely important that the weight of the jar, the weight of the methanol/surrogate solution, and the weight of the sample collected be known. These must either be measured directly, or sufficient information documented so that these weights can be calculated.

4.4.4 The ratio of soil to methanol used to calculate the MDL and PQL offered in the AK101 method was 1:1 (w:w). However, absorbent, organic soils such as muskeg and tundra require a higher methanol-to-sample ratio, while beach sand may tolerate a lower ratio.

4.4.5 Soil for volatiles analysis can be collected using any coring device that minimizes soil disturbance. Any scraping, stirring, or similar activity results in a loss of volatiles during sampling. A sufficient number of samples must be collected to provide for backup in case of breakage.

4.4.6 Although it is not necessary to refrigerate all methanol preserved samples at 4° ± 2°C after collection and until analysis is complete, collected samples must be kept below 25°C.

4.4.7 A second surrogate, added to the methanol and soil mixture after sample collection, may be used in addition to, but not in place of, the surrogate with which the field methanol preservative was prepared.

4.4.8 A reagent methanol trip blank must be prepared in the same manner as the sample vials, and must contain surrogated methanol. One trip blank must be included with each shipping container and must be stored and analyzed with the field samples. Trip blank analysis is not required if all samples in a shipping container are less than the project specific cleanup level.

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4.4.9 Field blanks may be added to the sampling protocol and are prepared in the field by addition of surrogated methanol to the prepared container, as required by the Assessment Firm or the Project Manager.

4.4.10 A sample of the same soil to be analyzed for GRO must be collected into a moisture-proof container for per cent moisture determination. This sample is processed as soon as possible upon arrival at the laboratory to assure that the resulting moisture determination is representative of the preserved sample as surveyed.

4.4.11 Trip blanks, field blanks, method blanks, etc. are prepared from the same batch of solvent, reagents and vials as are used for sample preservation.

4.4.12 Twenty-eight days is the maximum holding time for soil and sediment samples collected under this section.

4.4.13 Because the jars are pre-weighed, it is extremely important that the sampler put evidence tape on the kit ONLY, or the bubble bags in which the sample bottles are shipped, and not on the individual bottles. Removal of evidence tape is extremely difficult and the additional weight biases final results. Also, the glue on the evidence tape can contribute to the volatiles concentration in the sample.

4.4.14 Trip blanks, field blanks, and bottle blanks are prepared as appropriate to meet the quality assurance goals of the project plan.

4.4.15 28 days is the maximum holding time for AK101 soil samples preserved with MeOH. 14 days is the holding time if BTEX is included.

5.0 INTERFERENCES

5.1 Matrix interference can result in samples with high concentrations of volatile organic compounds (VOC). This interference can cause the resulting peaks to not be clear and concise. This can lead to misidentification of compounds and/or poor quantitation of those compounds. This problem can be solved by diluting the sample.

5.2 Carryover from previous samples must be monitored through the use of sample blanks. When a sample with a high concentration of VOC's is followed by a low level sample, false peaks may result from carryover. Sample blanks are used to clean the instrument.

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6.0 EQUIPMENT AND SUPPLIES

The operation, cleaning and scheduled maintenance procedures prescribed by the equipment manufactures are followed as provided in the Operator's Manuals. Documentation of maintenance or system modifications is recorded in a maintenance logbook which accompanies each instrument.

6.1. Instrumentation

- Designated Instruments: VOCGC #1, #2, #3, #4, #5, #6, #7, #8, #9, #10, #11, #12, #13
- Use (method #'s): 8021B; 8015B
- Model #: HP 5890 or equivalent
- Column (type, brand, size): J & W Scientific DB VRX 75m x 0.450mm, 2.55um or equiv.
- Detector: GC FID, PID
- Software name and version: HP Chemstation G1701BA B.01.00, or equivalent
- Sample introduction system: Archon Autosampler, Encon P & T, or equivalent

6.2 Glassware

Volumetric – glassware equipped with penny head ground glass stopper. The volumetric flasks and graduated cylinders are cleaned by rinsing with methanol and laboratory reagent water. The volumetrics are dried in a low temperature oven at less than 120°C. Never use a brush or strong alkali solution to clean the volumetrics.

6.3 Glass Sample (VOA) and Standard Vials:

6.3.1 "43mL" VOA vials with a Teflon™/silicone septa and polypropylene open-top cap.

6.3.2. 8mL vials with Teflon™/silicone/Teflon™ septa and polypropylene open-top cap. (Used to store unused standards)

6.4 Miscellaneous:

6.4.1 Stainless Steel Spatula, tongue depressors.

6.4.2 Disposable aluminum drying dishes - VWR #25433-008, or equivalent

6.4.3 Teflon™-coated stir bars, 8mm x 16mm

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6.4.4 Laboratory Sand: Sand is prepared by rinsing clean, white sand with methanol and laboratory reagent water several times. The rinsed sand is baked in an oven at 175°C overnight to remove any volatiles and is then stored in the same oven. The heated laboratory sand is occasionally purged with carrier grade helium or nitrogen to remove trapped volatiles.

6.5 **Oven**: Fisher IsoTemp Forced-Air Oven with capabilities of 100°C, or equivalent

6.6 **Top-loading Balance**, capable of weighing to 0.01g, or equivalent

7.0 REAGENTS AND STANDARDS

7.1 All reagents and standards must be recorded in the appropriate preparation log and assigned a unique number. See SOP 030203, *Reagent Logs and Records*, and SOP 030230, *Standard Logger*. Additional information regarding reagent preparation can be found in the Standards Logger (Tree) digital archive system. All spiking solutions and surrogate standard solutions should be replaced at least every 6 months, or sooner, if a problem is detected unless otherwise noted.

7.2 **Reagents**

7.2.1 Nanopure water or equivalent: Distilled water is used in all blanks to assure that it contains less than the method detection limit (MDL) of all compounds of interest. The blank must be assessed to ensure that the water does not show any detection of any VOC compounds.

7.2.2 Methanol, CH_3OH (VWR #EM-MX0480-1 or equivalent) - purge and trap grade, demonstrated to be free of target analytes. Store isolated from other solvents in the designated flammables cabinet.

7.2.3 Sodium Bisulfate, $Na_2S_2O_3$ from QEC, Level 3 certified in 40mL vials, or equiv.

7.3 **Stock Standards:** Stock solutions may be prepared from pure standard materials or purchased as certified solutions. These standards are prepared in methanol. Store stock standards in vials at ≤10°C.

7.3.1 BTEXM/GRO Calibration Standard – NSI PVOC/GRO mixture UST-360-03, or equivalent is used for the BTEXM compounds, and Restek certified BTEX in unleaded gas composite Cat # 30237, or equivalent, is used for GRO. The stock standards are prepared from standards with the following components and approximate concentrations:

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Benzene	1000ug/mL
Toluene	1000ug/mL
Ethylbenzene	1000ug/mL
M & P-xylene	2000ug/mL
o-xylene	1000ug/mL
MTBE	1000ug/mL
GRO	5500ug/mL

7.3.2 Synthetic WISGRO Calibration Standard – NSI PVOC/GRO mixture UST-360-03, or equivalent, while the LCS is from Restek Cat # 30095 revised WISC PVOC/GRO mixture or equivalent. The stock standard is prepared from a standard with the following components and concentrations:

MTBE	1000ug/mL
Benzene	1000ug/mL
Toluene	1000ug/mL
Ethylbenzene	1000ug/mL
m-xylene	1000ug/mL
p-xylene	1000ug/mL
o-xylene	1000ug/mL
1,2,4-trimethylbenzene	1000ug/mL
GRO (sum of rep. comp.)	10000ug/mL
1,3,5-TMB	1000ug/mL
Napthalene	1000ug/mL

7.3.3 Louisiana GRO Calibration Standard – Restek certified BTEX in unleaded gas composite Cat # 30237 with a concentration of 5500µg/mL. The standard ranges from C_6 to C_{12} – Hexane to Dodecane.

7.3.4 BTEXM/GRO Laboratory Control Standard - Restek revised WISC PVOC/GRO mix cat# 30095 or equivalent for BTEXM and NSI Gas composite Q-4643 or equivalent for GRO: Concentrations as stated in 7.3.2.

7.3.5 GRO Retention Time Marker: Restek Cat#30483 or equivalent: The stock standard is prepared from a standard with the following concentrations:

Hexane	1000ug/mL
Decane	1000ug/mL
Dodecane	1000ug/mL

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7.4 **Intermediate ICV/CCV/LCS Standards**

7.4.1 BTEX ICV from NSI (UST-360-03) is certified BTEX in unleaded gas. The LCS is from Restek Cat#30095. Secondary dilution standards of BTEXM/GRO Standard: This intermediate standard is stored with minimal headspace in the same manner as the stock standard. 2.5mL of BTEXM solution from 7.3.1 in 50mL of methanol has the following concentrations. The GRO standard is not diluted.

Benzene	50ug/mL
Toluene	50ug/mL
Ethylbenzene	50ug/mL
M & P-xylene	100ug/mL
O-xylene	50ug/mL
MTBE	50ug/mL
GRO	5500ug/mL

(Syringe sizes needed: 5mL, 25µl)

7.4.2 WISGRO's working standard is prepared by mixing 2.5mL of synthetic WISGRO standard into 47.5mL of methanol to make a 50ppm working standard which is then used to prepare all calibration standards.

7.4.3 The working standard used for Louisiana GRO is not diluted to prepare the calibration curve.

7.5 **Calibration standards:**

Calibration standards are prepared in reagent water at a minimum of five concentration levels. The lowest standard must be at or below the RL. The calibration standards are prepared from the primary source (which is a different Lot # than the LCS) according to the instructions in 7.3. This is the intermediate stock. Use the measurements listed below and dilute each to 5mL to produce each calibration point. The concentration varies slightly with lot number.

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BTEXM/GRO

	Conc. ppm	Amt.uL	Amt.uL	Amt.uL	Amt.uL	Amt.uL	Amt.uL	Amt.uL	Amt.uL	Amt.uL
	Stock listed in 7.3.1	0.05	.1	.5	1	2.5	5	10	20	25
GRO ppm	5500	0.055 µg/mL	0.11 µg/mL	0.55 µg/mL	1.1 µg/mL	2.75 µg/mL	5.5 µg/mL	11 µg/mL	-	-
GRO Surrogate - a,a,a-TFT		200ug/L	202ug/L	204ug/L	206ug/L	208ug/L	210ug/L	212ug/L	-	-
Benzene ppb	50	0.5ug/L	1ug/L	5ug/L	10ug/L	25ug/L	50ug/L	100ug/L	200ug/L	250ug/L
Toluene ppb	50	0.5ug/L	1ug/L	5ug/L	10ug/L	25ug/L	50ug/L	100ug/L	200ug/L	250ug/L
Ethylbenzene ppb	50	0.5ug/L	1ug/L	5ug/L	10ug/L	25ug/L	50ug/L	100ug/L	200ug/L	250ug/L
m&p Xylene ppb	50	1ug/L	2ug/L	10ug/L	20ug/L	50ug/L	100ug/L	200ug/L	400ug/L	500ug/L
o Xylene ppb	50	0.5ug/L	1ug/L	5ug/L	10ug/L	25ug/L	50ug/L	100ug/L	200ug/L	250ug/L
MTBE ppb	50	0.5ug/L	1ug/L	5ug/L	10ug/L	25ug/L	50ug/L	100ug/L	200ug/L	250ug/L
BTEX Surrogate - a,a,a-TFT		200ug/L	202ug/L	204ug/L	206ug/L	208ug/L	210ug/L	212ug/L	216ug/L	218ug/L

Synthetic GRO

	Conc. ppm	Amt.uL	Amt.uL	Amt.uL	Amt.uL	Amt.uL	Amt.uL	Amt.uL	Amt.uL	Amt.uL
	Stock listed in 7.3.1	.05	.1	.5	1	2.5	5	10	20	25
GRO ppm	500			0.05 µg/mL	0.1 µg/mL	0.25 µg/mL	0.5 µg/mL	1.0 µg/mL	2.0 µg/mL	2.5 µg/mL
Benzene ppb	50	0.5ug/L	1ug/L	5ug/L	10ug/L	25ug/L	50ug/L	100ug/L	200ug/L	250ug/L
Toluene ppb	50	0.5ug/L	1ug/L	5ug/L	10ug/L	25ug/L	50ug/L	100ug/L	200ug/L	250ug/L
Ethylbenzene ppb	50	0.5ug/L	1ug/L	5ug/L	10ug/L	25ug/L	50ug/L	100ug/L	200ug/L	250ug/L
m&p Xylene ppb	50	1ug/L	2ug/L	10ug/L	20ug/L	50ug/L	100ug/L	200ug/L	400ug/L	500ug/L
o Xylene ppb	50	0.5ug/L	1ug/L	5ug/L	10ug/L	25ug/L	50ug/L	100ug/L	200ug/L	250ug/L
MTBE ppb	50	0.5ug/L	1ug/L	5ug/L	10ug/L	25ug/L	50ug/L	100ug/L	200ug/L	250ug/L

Louisiana GRO

	Conc. ppm	Amt.uL	Amt.uL	Amt.uL	Amt.uL	Amt.uL	Amt.uL	Amt.uL
	Int. Stock Conc. (from 7.3.3)	0.05	.1	.5	1	2.5	5	10
GRO ppm	5500	0.055 µg/mL	0.11 µg/mL	0.55 µg/mL	1.1 µg/mL	2.75 µg/mL	5.5 µg/mL	11 µg/mL

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7.6 **Internal standard Fluorobenzene** 100,000µg/mL - NSI Cat # Q-4187 or equivalent.

7.7 **Surrogate** Alpha, alpha, alpha-trifluorotoluene 100,000µg/mL - NSI Cat # Q-4187 or equiv.

7.8 **Surrogate/Internal standard preparation**: Commercially-prepared ααα-TFT at 100,000ug/mL and Fluorobenzene at 100,000µg/mL are purchased from NSI for use in making an internal standard/surrogate mixture. This mixture is prepared by diluting 2mL of the NSI mixture into 199mL of methanol (200mL total volume). It is injected automatically by the instrument at a rate of 1µL per 5mL purge volume. This results in a 200µg/L solution of internal standard/surrogate. Check daily to make sure that the instrument reservoir has adequate IS/Surr solution.

7.9 **Solvent:** Methanol Fisher GC Resolve A457-4 or equivalent: High res.- GC grade.

7.10 **Spike Solution (LCS/LCSD/MS/MSD):** For the LCS/LCSD, spike the LCS spike solution prepared in section 7.3.1 into duplicate aliquots of a clean matrix. For the MS/MSD, prepare the spikes using the same as LCS solution except, introduce 5µl of the solution prepared in 7.3.1 directly into separate aliquots of the selected field sample.

8.0 PROCEDURE

STATE NOTE: For samples analyzed in conjunction with the Ohio VAP program, EPA method 8015B must be used for analysis.

STATE NOTE: WYOMING QC REQUIREMENTS (Please note the difference in QC Frequency):

- One calibration check standard in 10 samples (or method-specific frequency) inside established control limits; if any are outside the limits, repeat analysis of all affected samples.
- One reagent, method or preparation blank (carried through preparation) in 10 (or per batch).
- One matrix spike in 10 (or per batch).
- One duplicate or matrix spike duplicate in 10 (or per batch).
- Internal or external standards and surrogates (where available) are used for all samples.
- As required by the method, one laboratory control sample (consists of a representative matrix spiked with a reference standard containing the target analytes) in 10 (or per batch).

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NOTE: Analysis of a spiked blank water or soil (LCS)(sodium sulfate or silica sand for organics) which is subjected to ALL extractions and cleanups with each batch of 10 samples or less is recommended to evaluate analytical system performance (CLP Statement of Work (SOW) requires analysis of an LCS).

STATE NOTE: WI GRO – An LCS/LCSD is REQUIRED in every batch for all matrices.

8.1 **Analysis Summary:** Volatile compounds are introduced into the gas chromatograph by purge and trap, via the Archon autosampler. If soil samples are high in contamination, a methanolic extraction, as described section 8.4.4 and SOP No.330760, may be necessary prior to purge and trap analysis. Soils require method 5035A for sample preparation, See SOP 330751.

8.2 **Gas Chromatography Conditions:**

8.2.1 The conditions for each instrument and column are listed in the corresponding instrument log.

8.3 **Calibration:** Method 8015, 8021B BTEXM, WI GRO, and Synthetic GRO require a five-point calibration curve. This curve must have a % RSD of <20% for each of the BTEXM/GRO compounds. In the event that RF criteria are not met, linear regression may be used. In order to use this option, the correlation coefficient of the calibration curve must be a minimum of 0.990 or better. For USACE samples the correlation coefficient must be 0.995 or better. Equal weighting factors or 1/x regressions may be used.

STATE NOTE: Linear regression is required for quantitation of **WI GRO** samples. PVOC is acceptable on average response.

STATE NOTE: AK101 GRO must have %RSD < 25%. Linear Regression is not permitted for AK101

8.3.1 BTEX/MTBE quantitation is performed using "total area vs selected peak".

8.3.2 **Retention Time Marker:** GRO by 8015, WI GRO, and Synthetic GRO quantitation is performed using "baseline to baseline" integration. The area is summed from the marker compounds of 2-methylpentane to 1,2,4-trimethylbenzene, representing C_6 to C_{10}. These markers are evaluated and RT's changed when appropriate. The instrument response attributed to the surrogate and internal standard is not included.

STATE NOTE: For Ohio VAP samples the GRO markers are analyzed daily prior to standards and QC samples.

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STATE NOTE: For NWTPH-Gx, the retention time range for gasoline integration must, at a minimum, include toluene through naphthalene. For surrogates that elute within the retention time range used for TPH integration, the analyst must subtract the area of the surrogate(s) from the total area of the TPH peak to yield the appropriate area of the petroleum product.

STATE NOTE: For AK101, the retention time range for gasoline integration must include the resolved and unresolved components that elute between and including C_6 (hexane) and C_9 (nonane) to end at the peak start time of C_{10} (decane). Quantitation must be performed using "baseline to baseline" integration.

8.3.3 **ICV:** A mid-point check standard is analyzed first. All BTEX, MTBE, WI GRO, and GRO by Method 8015 compounds must be within 15% of the actual value. When this criteria is met, a blank is run to be sure there is no carryover or instrument contamination.

STATE NOTE: AK101 GRO CCV limits are ±25% for GRO and 60-120% for surrogates.

8.3.4 A Laboratory Control Standard (LCS) is evaluated by the QC limits in Attachment III for BTEXM/GRO by Method 8015. WI GRO LCS must be within 80-120% and AK101 GRO must be within 60-120% for both soil and water. An LCS and LCSD are required with each batch. The RPD cannot exceed 20% for either matrix.

8.3.5 A Matrix Spike/Matrix Spike Duplicate must be evaluated for each matrix type. Evaluate the matrix spike and matrix spike duplicate recovery based on the QC limits in Attachment III. If for any analyte, the laboratory control standard, matrix spike, and the matrix spike duplicate are all outside the QC limits, the entire analytic batch must be reanalyzed or QC cab be reanalyzed.

8.3.6 A secondary source Calibration Verification Standard is required for AK101 GRO calibrations. This standard must be within ±25% with surrogates at 60-120%.

8.3.7 **STATE NOTE:** For MN/WI GRO samples a reporting level check standard must be analyzed after each calibration or monthly, whichever is more often, and must recover within 60-140%.

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8.4 **Gas chromatographic analysis:**

Typical Batch order for loading the autosampler when a calibration is run:

Sample/QC Type	Use
Cleanup Blank	Verify system is contamination free
Retention Marker	Verify windows for gasoline ranges. Also required to be analyzed every 24 hour for AK101 and Ohio VAP.
Calibration standard(s)	Initial 5-point calibration or single-point calibration verification. MUST be mid-point standard.
Second Source Cal. Verification (SSCV)	Second Source verification of initial calibration.
Initial Calibration Verification (ICV)	Verify initial 5-point calibration.
Laboratory Control Sample(s)	Laboratory blank, spiked with known amount(s) of analyte of interest
Matrix Spike/Matrix Spike Dup.	Sample spiked with known amount(s) of analytes of interest
Method blank	Ensure that carry over has not occurred from the calibration standard, and that the analytical system does not show contamination above the established detection limits
1 to 10 samples	Client samples
Continuing Calibration Verification (CCV)	Single-point calibration verification standard.
1 to 10 samples	Client samples

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Typical Batch order for loading the autosampler when a calibration is not run:

Sample/QC Type	**Use**
Cleanup Blank	Verify system is contamination free
Retention Marker	Verify windows for gasoline ranges. Required to be analyzed every 24 hour for AK101 and Ohio VAP.
Initial Calibration Verification (ICV)	Verify initial 5-point calibration.
Laboratory Control Sample(s)	Laboratory blank, spiked with known amount(s) of analyte of interest
Matrix Spike/Matrix Spike Dup.	Sample spiked with known amount(s) of analytes of interest
Method blank	Ensure that carry over has not occurred from the calibration standard, and that the analytical system does not show contamination above the established detection limits
1 to 10 samples	Client samples
Continuing Calibration Verification (CCV)	Single-point calibration verification standard.
1 to 10 samples	Client samples

8.4.1 Water/liquid Samples

8.4.1.1 Samples are received in 40mL vials containing HCl as a preservative. Using a gas tight syringe, 5mL is withdrawn from the vial and dispensed into another 40mL vial. The autosampler injects the sample with 1µL of internal standard/surrogate mix. The autosampler moves the vial into a heating chamber and heats the sample for 1 minute at 40°C. The sample then purges for 11min at 40°C to drive off all VOC's to the trap. The trap is desorbed for 2 minutes at 175°C before entering the column for analysis.

8.4.2 Soil/Sediment Samples (Water Purge) - Collected in soil Jar

8.4.2.1 Weigh 1 gram of soil into a 40mL vial containing a stir bar. Add 5mL of nanopure water and tighten cap. The autosampler injects the sample with 1µl of internal standard/surrogate mix. The autosampler moves the vial into a heating chamber and heats the sample for 1 minute at 40°C. The sample then purges for 11min at 40°C to drive off all VOC's to the trap. The trap is desorbed for 2 minutes at 175°C before entering the column for analysis.

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8.4.3 Soil/Sediment Samples - Collected in Encore (Encore "like") Sampling Device

8.4.3.1 The sample is collected using an Encore or Encore "like" sampling device. The device is designed to sample soil at approximately 5g. The sample is placed into a pre-weighed 40mL vial containing a stir bar and 5mL of Sodium Bisulfate, as a preservative. Weigh the vial to determine the weight of the soil. WISGRO is 25g of soil into 25mL of methanol.

Soil Sample Weight (g) = Total weight of Vial and Soil (g) - Pre - weigh value (g)

Record the determined weight of the sample and load onto the autosampler. The autosampler injects the sample with 1µL of internal standard/surrogate mix. The autosampler moves the vial into a heating chamber and heats the sample for 1 minute at 40°C. The sample then purges for 11min at 40°C to drive off all VOC's to the trap. The trap is desorbed for 2 minutes at 175°C before entering the column for analysis.

8.4.4 High Level Soil/Sediment Sample (Methanol Extraction) – Collected in Soil Jar

NOTE: Samples known to have high concentrations greater than 200ppb may be collected in a 2oz. Sample jar with no headspace.

8.4.4.1 Weigh 5g of soil sample and place in vial. Add 5mL of Methanol and shake vial for 2 minutes. Using a gas tight microsyringe, measure a maximum of 100µL of methanol extract and inject into a vial containing 5mL of water. Enter the sample multiplier as 50X. The autosampler injects the sample with 1µL of internal standard/surrogate mix. The autosampler moves the vial into a heating chamber and heats the sample for 1 minute at 40°C. The sample then purges for 11min at 40°C to drive off all VOC's to the trap. The trap is desorbed for 2 minutes at 175°C before entering the column for analysis. AK101 use 150ul of methanol extract and sample multiplier of 33.3x (Need to meet RL of 20ug/kg for Benzene. Sample is field preserved with MeOH). For PVOCGRO soil samples 25g of soil is placed into a vial with 25mL of methanol. If the weight of the soil exceeds 35g the sample is discarded. If the weight of the sample is >26g but <35g methanol is added until the volume of methanol in mL is equal to the weight of the soil in g.

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8.5 **Quantitation:**

8.5.1 Quantitation of GRO is performed by the internal standard method. The concentration of Gasoline Range Organics in the sample is determined from a summation of the total response within the range of the elution of 2-methyl pentane and 1,2,4-Trimethylbenzene, using the calibration curve. No area other than that relating to the internal standard or surrogates may be subtracted from the GRO retention time window in calculating GRO results. WISGRO is evaluated by the external standard method. WISGRO no IS/SURR areas are subtracted and range is from the beginning of MTBE and to the conclusion of Naphthalene.

8.5.2 Integration must be "baseline to baseline" as opposed to a "valley to valley". Baseline to baseline is defined here as a flat baseline drawn parallel to the x-axis of the chromatogram that includes all responses within the retention time window. The correct baseline coincides with a horizontal line drawn through the lowest point in the chromatogram before the end of the window. The lowest point may be within the window, before the window, or before the solvent front. Baseline to baseline integration does not include the solvent peak. Placement of the baseline is determined for each sample.

CLIENT NOTE: When EPA 8021B analysis is performed for samples analyzed in conjunction with Marathon/MPC LLC/SSA, confirmation is performed using GC/MS by EPA 8260B.

8.6 For acceptance criteria and corrective actions, see sections 10.0 & 11.0.

9.0 DATA ANALYSIS AND CALCULATIONS

9.1 Internal Calibration Equations:

$$RF = \frac{[A_s][C_{is}]}{[A_{is}][C_s]}$$

where:
A_s = Peak area (or height) of the analyte or surrogate.
A_{is} = Peak area (or height) of the internal standard.
C_s = Concentration of the analyte or surrogate, in µg/L.
C_{is} = Concentration of the internal standard, in µg/L.

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- Percent Relative Standard Deviation (%RSD)

$$\overline{RF} = \frac{\sum_{i=1}^{n} RF_i}{n} \qquad SD = \sqrt{\frac{\sum_{i=1}^{n} (RF_i - \overline{RF})^2}{n-1}} \qquad RSD = \frac{SD}{\overline{RF}} x100\%$$

where:
RSD = Relative standard deviation.
RF = Mean of 5 initial RFs for a compound.
SD = Standard deviation of average RFs for a compound.

- Percent Difference

$$\frac{RFv - RF}{RF} * 100\%$$

where:
RF = Average response factor from initial calibration.
RFv = Response factor from current verification check standard.

- Percent Drift

$$\%Difference = \frac{C_0 - C_1}{C_1} X100$$

where:
C_1 = compound standard concentration
C_0 = measured concentration using selected quantitation method

9.2 Linear calibration model:

$$y = mx + b$$

where: y = Response A_X for External Standard
x = Concentration C_X for External Standard
m = Slope
b = Intercept

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- Slope (m):

$$m = \frac{[(Swx_iy_i * Sw) - (Swx_i * Swy_i)]}{[(Sw * Swx_i^2) - (Swx_i * Swx_i)]}$$

- Intercept (b):

$$b = y_{AVE} - (m * (x_{AVE}))$$

- Correlation Coefficient (r):

$$r = \frac{[(Sw * Swx_iy_i) - (Swx_i * Swy_i)]}{\sqrt{\{[(Sw * Swx_i^2) - (Swx * Swx_i)] * [(Sw * Swy_i^2) - (Swy_i * Swy_i)]\}}}$$

- Coefficient of Determination (r^2):

$$r^2 = r * r$$

Where: n = number of x, y pairs
x_i = individual values for the independent variable
y_i = individual values for the dependent variable
w = weighting factor, for equal or no weighting w = 1
x_{AVE} = average of the x values
y_{AVE} = average of the y values
S = the sum of all the individual values

9.3 External Calibration Equations:

- The calibration factor for each standard can be calculated:

$$CF = \frac{A_s}{C_s}$$

where: A_s - Average Peak Area over the number of peaks used for quantitation
C_s – Concentration of the analyte in the standard.

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- The average (or mean) calibration factor ($\overline{CF}$) is calculated:

$$\overline{CF} = \frac{\sum_{i=1}^{n} CF_i}{n}$$

where: CF_i – Calibration Factor for each level of the calibration curve
n – number of standards analyzed in the calibration curve

- The standard deviation (SD) of the calibration is determined:

$$SD = \sqrt{\frac{\sum_{i=1}^{n} (CF_i - \overline{CF})^2}{n-1}}$$

where: $\overline{CF}$ – Average Calibration Factor for the calibration curve
CF_i – Calibration Factor for each level of the calibration curve
n – number of standards analyzed in the calibration curve

- The Percent Relative Standard Deviation for each analyte in the curve is determined:

$$RSD = \frac{SD}{\overline{CF}} \times 100$$

where: $\underline{SD}$ – Standard Deviation for each analyte
$\overline{CF}$ – Average calibration factor for the specific analyte

9.4 Percent Difference for daily calibration curve verification:

$$\% \text{ Difference} = \frac{\overline{CF} - CF_v}{\overline{CF}} \times 100$$

where: $\underline{CF_v}$ – Calibration Factor from the calibration verification standard
$\overline{CF}$ – Average (or mean) calibration factor from the initial calibration curve

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9.5 LCS/ICV/CCV Percent Recovery (%R):

$$\%\,R = \frac{\text{Measured concentration}}{\text{Actual concentration}} \times 100$$

9.6 Matrix Spike Recoveries ($\%R_{MS/MSD}$):

$$\%R_{MS/MSD} = \frac{O_i - O_s}{T_i} X 100$$

where: O_i = observed sample concentration with the spike added
O_s = the observed value for the sample without the spike
T_i = True value of the spike added

9.7 Relative Percent Difference (%RPD):

$$RPD = \frac{\text{Value 1} - \text{Value 2}}{\left(\frac{\text{Value 1} + \text{Value 2}}{2}\right)} \times 100$$

9.8 AK101 Moisture Correction: In order to report results for volatiles analysis of samples containing significant moisture (>10%) content on an "as received" basis, the calculated concentration needs to be corrected using the total solvent/water mixture volume represented as Vt. This total solvent/water volume is calculated as follows:

$$\mu L \text{ solvent/water } V_t = \left[\frac{mL \text{ of solvent} + (\%\text{ moisture} \times g \text{ of sample})}{100}\right] \times 1000\ \mu L/mL$$

10.0 QUALITY CONTROL AND METHOD PERFORMANCE

10.1 All analysts must meet the qualifications specified in SOP 030205, *Technical Training and Personnel Qualifications* before approval to perform this method. Analysts must complete an initial demonstration of proficiency before being approved to perform this method. Continuing proficiency must be demonstrated using proficiency testing, laboratory control sample analysis and/or MDL studies. Method performance is assessed per analyst. Updated method performance records are filed and stored in a central location within the department.

10.2 Use the designated Run log to record batch order and standards/reagents used during analysis. See SOP 030201, *Data Handling and Reporting*.

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10.3 Initial Calibration – Initial calibration curves must meet the criteria found in the following table. One concentration of the calibration standards must be at or below the RL. The remaining concentration should encompass the linear working range of the instrument. In most cases, %RSD or linear regression is acceptable. When using linear regression, equal weighting factors or 1/x regressions may be used.

Analytical Method	Min. # of Calibration Standards Required	Initial Calibration Acceptance Criteria	
		%RSD	Linear Correlation Coefficient
EPA 8015B, 8015C, 8015D	5	≤20%	≥0.990
EPA 8021B	5	≤20%*	≥0.990*
WI PVOC	5	≤20%*	≥0.990*
WI GRO	5	NA	≥0.990
SM 6200	5	≤20%	≥0.994
NWTPH-Gx	5	≤20%	≥0.990
Synthetic GRO	5	≤20%	≥0.990
EPA 602	3	≤10%	≥0.990
OA1	3	≤20%	≥0.995
AK101	3	<25%	NA

* required for each target analyte being reported for this method.
NA indicates that this process cannot be used for this method.
NOTE: For USACE samples the correlation coefficient must be ≥0.995.

10.4 Initial Calibration Verification (ICV)/Continuing Calibration Verification (CCV) – Before beginning a sample run, a midpoint check standard (ICV) is analyzed initially to ensure accurate instrument calibration. Continuing calibration verification (CCV) must be checked after every 10 samples and at the conclusion of the sequence by using a midpoint check standard. Acceptance criteria for the specific methods are listed in the table below. If the criterion is exceeded, the 10 samples preceding the CCV are unacceptable and must be re-analyzed. In order for any sample to have valid results, acceptable QC must bracket it.

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Analytical Method	Continuing Calibration Acceptance Criteria
EPA 8015B, 8015C, 8015D	± 20% of Expected Value
EPA 8021B	± 20% of Expected Value
WI PVOC	± 15% of Expected Value
WI GRO	± 20% of Expected Value
SM 6200	± 30% of Expected Value
NWTPH-Gx	± 20% of Expected Value
Synthetic GRO	± 15% of Expected Value
EPA 602	± 30% of Expected Value
OA1	± 20% of Expected Value
AK101	± 25% of Expected Value

10.5 Method Blank – Method Blanks contain reagent water that are analyzed following successful calibration and/or verification to ensure that the analytical system is free from interferences prior to the analysis of field samples. The acceptance criteria for all method blanks is <1/2 RL.

STATE NOTE: For samples analyzed in conjunction with AK101, blank subtraction is not permitted. Blanks should be reported by value for data quality assessment.

STATE NOTE: For samples analyzed in conjunction with Ohio VAP, when target analyte concentrations are above the reporting limit in the blank, all field samples must be re-prepared and re-analyzed, if sufficient sample volume was submitted by the client, prior to flagging the data report.

NOTE: Additional blanks may be submitted with client batches to verify that no cross contamination occurs during shipping of samples and there is no contamination contributed from the sampling equipment. Additional blanks may also be analyzed to ensure that the analytical system remains clean following the analysis of highly contaminated samples.

10.6 Matrix Spike/Matrix Spike Duplicate - are run every 20 samples. Acceptance criteria are in Attachment III. For Synthetic GRO, the recovery must be between 50 and 100%. The RPD must not exceed 25%. The analyst also verifies that the spikes are at the appropriate levels. Spiking levels correspond to the midpoint of the calibration curve. Acceptance criteria for BTEXM/GRO are listed in Attachment III. If the spike recovery does not meet criteria, verify matrix interference and apply qualifiers.

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10.7 Laboratory Control Sample (LCS)/Laboratory Control Sample Duplicate (LCSD) – An LCS and LCSD are required with each batch.and evaluated using the QC limits in Attachment III for BTEXM/GRO by Method 8015. Levels correspond to the midpoint of the calibration curve.

STATE NOTE: WI GRO LCS must be within 80-120% for both soil and water. The RPD cannot exceed 20% for either matrix.

STATE NOTE: AK101 GRO LCS must be within 60-120% for both soil and water. An LCS and LCSD are required with each batch. The RPD cannot exceed 20% for either matrix. Surrogates in AK101 must meet 60% to 120% recovery in Blanks, LCS, and LCSD.

10.8 Surrogates - must be assessed for all samples and QC in the batch. Alpha, Alpha, Alpha - TFT recovery must be within acceptance criteria listed in Attachment III.

STATE NOTE: The WI PVOC Surrogate must be within >80% for both soil and water and are analyzed from the PID only.

STATE NOTE: Surrogates in AK101 must meet 60% to 120% recovery in Blanks, LCS, and LCSD. Surrogates in field samples must meet 50-150% for both soil and water. For ease of analysis, the control limits used by the laboratory as found in Attachment III, exceed the method required limits, but allow for running these samples in conjunction with other TPH analyses.

STATE NOTE: Surrogates in NWTPH-Gx must meet 50-150% for both soil and water. For ease of analysis, the control limits used by the laboratory as found in Attachment III, exceed the method required limits, but allow for running these samples in conjunction with other TPH analyses.

10.9 Internal standard - IS fluorobenzene, response must be within acceptance limits for all samples and quality control samples. The internal standard response must be within 50% - 200% of the response of the calibration verification standard.

CLIENT NOTE: For Marathon/Speedway (MPC LLC/SSA), the internal standard area counts for all calibration standards, QC samples and field samples for quantitation must not change by a factor of greater than -50% to +130%.

10.10 Dilutions - All sample analytical results must be below the high standard of the calibration curve.

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10.11 IDOC's - The analyst must demonstrate proficiency in performing the analysis as outlined in SOP No.030205, *Technical Training and Personnel Qualifications*. Method proficiency must be re-demonstrated anytime a major method modification is made, a major software revision is added, or a major instrument modification is made.

STATE NOTE: Wisconsin GRO requires analysis of five replicates for initial demonstration of capability. Waters must be analyzed at a concentration of 100µg/L, with recoveries falling between 80-120% of the known concentration and the RSD must be <20% to be acceptable. Soils must be analyzed at a concentration of 10mg/kg, with recoveries falling between 75-120% of the known concentration and the RSD must be <20% to be acceptable.

10.12 Retention time windows - are calculated over a 72-hr period by taking the average RT of each compound in the ICV and calculating ±3 SD from this average. This is the retention time window. Retention time windows can vary between instruments.

STATE NOTE: Wisconsin GRO requires verification of the retention time window at the beginning of each data and whenever a new GC column is installed. This can be accomplished as part of the calibration verification.

10.13 Manual Integration – All manual integrations must comply with the requirements found in ESC SOP# 030215, *Manual Integration Procedure*. Before and after integrations must be available for review by the secondary data reviewer.

11.0 DATA VALIDATION AND CORRECTIVE ACTION

11.1 All data must undergo a primary review by the analyst. The analyst must check the performance of the initial calibration, mid-point check standard and continuing calibrations to ensure that they meet the criteria of the method. The analyst should review any sample that has quantifiable compounds and make sure that they have been confirmed, if needed. The analyst must also verify that reported results are derived from quantitation between the required RL and the highest standard of the initial calibration curve. All calculations must be checked (any dilutions, %solids, etc.). Data must be checked for the presence or absence of appropriate flags. Comments should be noted when data is flagged.

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TITLE: BTEX (Method 8021B, 602, SM6200C 20th) AND GASOLINE RANGE ORGANICS (Method 8015B, 8015C, 8015D) by GC (With provisions for Calif-Lo, NWTPH-Gx, OA1, WI GRO (synthetic), Wyoming LAUST Req., GRO Louisiana, GRO by OHVAP, AK101 GRO)

11.2 All data must undergo a second analyst review. The analyst checking the data must check the performance of the initial calibration, mid-point check standard and continuing calibrations to ensure that they meet the criteria of the method.

11.2.1 The analyst should look at any sample that has quantifiable compounds and check the integration.

11.2.2 All calculations must be checked.

11.2.3 All surrogate recoveries must be checked to see if they are within limits.

11.2.4 Blanks must be clean of all interfering peaks.

11.2.5 Quality control criteria should be checked for the LCS, LCSD, MS, and MSD.

11.2.6 Data must be checked for the presence or absence of appropriate flags. Comments should be noted when data is flagged.

11.2.7 See SOP# 030201, *Data Handling and Reporting*.

11.2.8 See SOP# 030208, *Corrective Action*.

11.3 Initial calibration - If the initial calibration does not meet the criteria for acceptance using response/calibration factors, then linear regression can be utilized, as long as the correlation coefficient meets the necessary criteria. If the linear regression criteria cannot be met, additional corrective actions are required. Standards must be reviewed and re-prepared, if necessary. Instrument maintenance may also be required, including column clipping/replacement, source cleaning, etc. When corrective actions have been completed, the instrument must be re-calibrated and the acceptance criteria must be met for the analytes of interest prior to the analysis of any field samples.

11.4 Initial/Calibration Check Standard (ICV/CCV) - When the initial or continuing calibration verification is beyond the acceptance criteria, the analysis must be terminated and corrective action must be taken to determine the cause of the problem. Corrective actions include: re-analysis of the ICV/CCV once. If the failure persists, additional corrective actions include: instrument maintenance, re-preparing the calibration standard, re-calibration of the instrument. Samples analyzed between the last passing calibration standard and the calibration standard that is out of control must be re-analyzed.

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11.5 Method Blank – If the method blank shows any detectable amount greater than ½ the RL, the laboratory performance is assumed to be out of control and the problem must be corrected. Corrective actions include: re-analysis once. If the failure persists, re-extract the entire batch of samples, if submitted sample volume permits.

STATE NOTE: For samples analyzed in conjunction with Ohio VAP, when target analyte concentrations are above the reporting limit in the blank, all field samples must be re-prepared and re-analyzed, if sufficient sample volume was submitted by the client, prior to flagging the data report.

11.6 Matrix Spike/Matrix Spike Duplicate - Assess that matrix spike/matrix spike duplicates were analyzed at required frequency, and that results are within acceptance criteria. Spike failure results in the use of a "J" or "V" flag. If a "J" flag is used, it is followed by the appropriate number, which further explains the failure concerning high or low response. The "V" flag is used to indicate that the sample concentration was too high to accurately evaluate the spike recovery. Acceptance criteria for BTEX/MTBE and GRO by Method 8015 are listed in Attachment III. For Synthetic GRO, the recovery must be between 50 – 100%. The RPD must not exceed 25%.

11.7 Laboratory Control Sample (LCS)/Laboratory Control Sample Duplicate (LCSD) – A Laboratory Control Sample (LCS) is run every 20 samples. Acceptance criteria for BTEX/MTBE and GRO by Method 8015 are listed in Attachment III. Levels correspond to the midpoint of the calibration curve.

STATE NOTE: WI GRO LCS must be within 80 - 120% for both soil and water. An LCS and LCSD is required with each batch. The RPD cannot exceed 20% for either matrix. Failure of the LCS results in a required of all samples within the batch.

STATE NOTE: AK101 GRO LCS must be within 60-120% for both soil and water with RPD not exceeding 20%. Surrogates in AK101 must meet 60% to 120% recovery in laboratory control samples – Blanks, LCS, and LCSD.

If the control does not perform within the ranges listed in Attachment III or current control ESC limits, the laboratory performance is assumed to be out of control and the problem must be corrected. Corrective action can include re-analysis, if instrument malfunction is suspected, or re-preparation and re-analysis of the entire batch, if the failure is suspected as either extraction or sample related.

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11.8 Surrogates - If the recovery is not within limits stated in Attachment II or ESC current control limits, confirm that there are no errors in the calculations, surrogate solutions and standards. Check the instrument performance. Examine the chromatograms for interfering peaks and integrated areas. Re-calculate the data and/or re-analyze the field sample if any of the above checks reveal a problem. When permitted, flag the data "J1" (surrogate high) or "J2" (surrogate low).

11.8.1 High recoveries may be due to co-eluting matrix interference: examine the sample chromatogram.

11.8.2 Low recoveries may be due to the sample matrix.

STATE NOTE: If field samples are analyzed in conjunction with the Ohio VAP program, surrogate outliers in batch QC samples, including the blank, LCS/LCSD, MS/MSD require re-extraction of the entire batch, if sufficient volume has been submitted by the client and an obvious matrix interferent is not present.

STATE NOTE: The surrogate for WI PVOC must recovery >80% for both matrices.

11.9 Internal standard - The internal standard area counts must be monitored for all CCVs. ISTDs must recover within 50% to 200% of the area counts from the internal standard area counts of the midpoint standard of the most recent initial calibration sequence. If any internal standard response is beyond the acceptable recovery, corrective action is required. Corrective action can take to form of checking the original calculations to ensure accuracy, re-analysis of the CCV to verify initial results, instrument maintenance (i.e. column clipping or changing, inlet liner cleaning/replacement, etc.) or re-calibration.

The internal standard responses and retention times in the check calibration standard must be evaluated immediately after or during data acquisition. If the retention time for any internal standard changes by more than 30 seconds from the last calibration verification, the chromatographic system must be inspected for malfunctions and corrections must be made, as required. When corrections are made, re-analysis of the CCV or a complete re-calibration is necessary, depending on the impact of the correction on the analytical system.

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Internal standards must be monitored for each sample. ISTDs in samples must meet the –50% to +200% criteria when compared to the ISTDs in the daily CCV or mid-level of the calibration curve, on 12h shifts when full calibration is performed. Possible corrective actions include: if instrument malfunction is suspected, or re-preparation and re-analysis, if the failure is suspected as either extraction or sample related. If the sample has an obvious matrix interferent and the internal standard recovery is greater than 200%, the sample can be diluted (if acceptable reporting limits can be achieved) to minimize the interference or the sample must be re-extracted and re-analyzed to confirm the original results.

11.10 Data that does not meet acceptable QC criteria may be acceptable for use in certain circumstances.

11.10.1 If a method blank contains an amount of target analyte, but all samples are non-detected, the data may be reported with a “B3” flag. If a method blank contains an amount of target analyte, but the samples contain analyte at a level that is 10 times the level present in the method blank, the data may be reported with a “B” flag.

11.10.2 If the MS/MSD fails in an initial analysis and again upon re-analysis, the data is released with an appropriate qualifier as the failure is accepted as matrix related.

11.10.3 If a calibration verification standard is above the acceptable QC criteria and all samples being bracketed are below the reporting limit, the data is acceptable based on a high calibration bias with undetectable levels in the field samples. Any positive samples require re-analysis.

11.10.4 If a sample duplicate is above the acceptable range for the RPD and the sample concentration is <5X the RL, then the value can be flagged with a “P1” qualifier indicating that the RPD calculation is not applicable at that concentration.

STATE NOTE: For all samples analyzed from Minnesota, the reporting limit must be verified at least monthly, with each new initial calibration, or when there has been significant change to the instrument (column replacement, cleaning source, etc.) whichever is more frequent. The reporting limit verification can be performed by either re-injecting the low standard or by re-processing the low standard that was analyzed in the calibration curve. The reporting limit verification (RLV) must recovery within ±40% of the expected concentration. If this criteria is not met, the RLV may be re-analyzed once, instrument maintenance can be performed, a higher concentration standard can be injected, or a new calibration curve must be generated. If a higher concentration standard is utilized, the reporting limit must be raised to the higher level verified.

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12.0 POLLUTION PREVENTION AND WASTE MANAGEMENT

12.1 The EPA requires that laboratory waste management practice to be conducted consistent with all applicable federal and state laws and regulations. Excess reagents, samples and method process wastes must be characterized and disposed of in an acceptable manner. See *ESC Waste Management Plan.*

12.2 See SOP #030302, *Pollution Prevention*.

13.0 METHOD MODIFICATIONS/CLARIFICATIONS

13.1 No technical modifications have been made. Provisions for additional QC and specific variations have been added.

13.2 Modifications to this method are noted in the body of the text as state notes. Compliance analyses performed in conjunction with specific state requirements must be performed as noted within the specific state(s) note listed.

13.3 Adjustments to the concentrations of standards/spiking solutions, standards providers, and quality control are subject to change to better meet client/project/regulatory needs or to improve laboratory method performance.

14.0 REFERENCES

14.1 Test Methods for Evaluating Solid Waste, EPA SW-846, *Method 8015B* (Rev. 2, Dec. 1996)

14.2 Test Methods for Evaluating Solid Waste, EPA SW-846, *Method 8021B* (Rev. 2, Dec. 1996)

14.3 *Modified GRO Method for Determining Gasoline Range Organics,* WISCONSIN DNR, Sept. 1995

14.4 A2LA – (R211) *Wyoming Storage Tank Remediation Testing Laboratory Accreditation Program* (5/10/11), http://www.a2la.org/requirements/17025_WY_STR.pdf

14.5 40 CFR Part 136, Appendix A to Part 136 -- Methods for Organic Chemical Analysis of Municipal and Industrial Wastewater, Method 602 -- Purgeable Aromatics

14.6 Standard Methods for the Examination of Water and Wastewater, 20th Edition, *Method 6200C*, Purge and Trap Capillary-Column Gas Chromatographic Method.

14.7 Iowa Laboratory Certification: Method OA-1 - *Method for Determination of Volatile Petroleum Hydrocarbons (Gasoline),* Rev. 7/27/93.

14.8 California LUFT Field Manuals, October 1989, *Total Petroleum Hydrocarbons (TPH) Analysis, Gasoline and Diesel*.

14.9 State of Alaska, Dept. of Env. Conservation, Contaminated Sites Laboratory Approval Memorandum, Soil Moisture Corrected Reporting by EPA Method 8000C, Feb. 2008.

14.10 States of Oregon and Washington, NWTPH-Gx: *Volatile Petroleum Products Method for Soil and Water*

14.11 State of Alaska Methods: AK101 version 4/08/02.

14.12 Test Methods for Evaluating Solid Waste, EPA SW-846, *Method 8015C* (Rev. 3, Feb., 2007)

14.13 Test Methods for Evaluating Solid Waste, EPA SW-846, *Method 8015D* (Rev. 4, June, 2003)

14.14 Test Methods for Evaluating Solid Waste, EPA SW-846, *Method 8000B* (Rev. 2, Dec. 1996)

14.15 Test Methods for Evaluating Solid Waste, EPA SW-846, *Method 8000C* (Revision 3, March, 2003)

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Attachment I: Revision History

Current Version:

Version	Date	Description of Revisions
14	3/23/12	Technical and Quality Review and update. Added sections 1.3.1, 2.14 through 2.28, 10.13, and state/client notes in sections 1.0, 8.3.2, 8.5.2, and 11.10; Revised Attachments II and III and sections 1.2, 2.4, 7.1, 7.5, 7.8, 8.3, 9.1 through 9.8, 10.3 through 10.12, 11.1 through 11.9, 12.1, 14.7, 14.9, and 14.10; Incorporated previous minor revisions.

Superseded Versions:

This document supersedes the following:

Version	Date	Description of Revisions
0	8/94	Origination
1	7/95	
2	12/28/98	
3	9/1/99	
4	8/22/00	
5	11/1/01	
6	4/29/02	
7	4/23/03	
8	11/3/03	
9	4/14/04	
10	8/15/06	
11	11/30/07	Technical and Quality Review and update.
12	1/10/08	Addition of Section 4.5.4 - AK101 requirements.
13	2/23/09	Clarification of spike solutions in section 7.9; addition of state notes; inclusion of calculations for average response factors, linear calibration and correlation coefficient; addition of corrective actions in section 11.3 through 11.8; Clarifications in sections 12.0 & 13.0. Ohio VAP approved 2/23/09.

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Attachment II: Routine Reporting Limits*

Compound	RL SOIL (mg/Kg) 1g sample size	RL Water (mg/L)
Benzene	0.0025	0.0005
Toluene	0.025	0.005
Ethylbenzene	0.0025	0.0005
M & P Xylenes	0.0050	0.0010
O Xylenes	0.0025	0.0005
MTBE	0.0250	0.005
GRO	0.5	0.10
Compound	**RL methanol (mg/Kg) extract by 5035A**	**RL Sodium bisulfate (mg/Kg)**
Benzene	0.025 (AK101 0.020)	0.0005
Toluene	0.25	0.005
Ethylbenzene	0.025	0.0005
M & P Xylenes	0.050	0.0010
O Xylenes	0.025	0.0005
MTBE	0.250	0.005
GRO	5.0	0.10

**See section 13.3.*

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Attachment III: QC ACCEPTANCE CRITERIA*

Compound	LCS Control Limits %	LCS RPD Limits %	MS Control Limits %	MS RPD Limits %
Benzene	71-121 (water) 74-123 (soil)	13 (water) 13 (soil)	64-117 (water) 62-116 (soil)	17 (water) 18 (soil)
Toluene	71-125 (water) 79-120 (soil)	17 (water) 12 (soil)	70-119 (water) 64-120 (soil)	15 (water) 14 (soil)
Ethylbenzene	76-115 (water) 73-124 (soil)	17 (water) 13 (soil)	73-114 (water) 61-118 (soil)	16 (water) 17 (soil)
m&p Xylenes	73-124 (water) 74-124 (soil)	16 (water) 13 (soil)	65-124 (water) 60-121 (soil)	24 (water) 16 (soil)
o Xylene	76-118 (water) 78-117 (soil)	14 (water) 12 (soil)	68-120 (water) 64-118 (soil)	25 (water) 16 (soil)
MTBE	65-129 (water) 56-134 (soil)	23 (water) 13 (soil)	60-127 (water) 57-128 (soil)	22 (water) 23 (soil)
GRO	76-114 (water) 73-124 (soil)	17 (water) 13 (soil)	57-120 (water) 54-117 (soil)	16 (water) 17 (soil)
Wisconsin GRO	80-120 (water) 80-120 (soil)	20 (water) 20 (soil)	80-120 (water) 80-120 (soil)	20 (water) 20 (soil)
AK101 GRO	60-120 (water) 60-120 (soil)	20 (water) 20 (soil)		
Louisiana GRO	70-130 (water) 70-130 (soil)	20 (water) 20 (soil)	70-130 (water) 70-130 (soil)	20 (water) 20 (soil)
a,a,a-TFT (Surr) AK101 Laboratory Samples	60-120 (FID) 60-120 (PID)			
a,a,a-TFT (Surr) AK101 Field Samples	60-120 (FID) 60-120 (PID)			
a,a,a-TFT (Surr) WI GRO/PVOC	>80% (FID) >80% (PID)			
a,a,a-TFT (Surr)	68-124 (FID water) 80-123 (PID water) 66-124 (FID soil) 75-119 (PID soil)			

*Control Limits are calculated semi-annually and are subject to change (except for Wisconsin GRO, AK101 GRO and Louisiana GRO which are method defined).

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Environmental Science Corporation
SOP/Document REVISION FORM

02/06/07 R.1.0

SOP/DOC# 330351 Current revision date & number: R14 3/23/12

Procedure/Method : BTEX (Method 8021B, 602, SM6200C 20th) AND GASOLINE RANGE ORGANICS (Method 8015B, 8015C, 8015D) by GC (With provisions for Calif-Lo, NWTPH-Gx, OA1, WI GRO (synthetic), Wyoming LAUST Req., GRO Louisiana, GRO by OHVAP, AK101 GRO)

Date	Analyst	Section	Revision	Reason*	Approvals	
					Supervisor	QA
6/6/12	DLM	8.0	Revise state note regarding Wyoming batch requirement of 10 samples.	WY STR Update	JD6	QAH
			Wyoming updated their batch requirement to ≤20 field samples on 5/10/11.			
		14.4	Update to reflect: R211 – Specific Requirements: Wyoming Storage Tank			
			Remediation Testing Laboratory Accreditation Program, May 10, 2011			

*Comments:

STATE NOTE: WYOMING QC REQUIREMENTS:

- One calibration check standard in 20 samples (or method-specific frequency) inside established control limits; if any are outside the limits, repeat analysis of all affected samples.
- One reagent, method or preparation blank (carried through preparation) in 20 (or per batch).
- One matrix spike in 20 (or per batch).
- One duplicate or matrix spike duplicate in 20 (or per batch).
- Internal or external standards and surrogates (where available) are used for all samples.
- As required by the method, one laboratory control sample (consists of a representative matrix spiked with a reference standard containing the target analytes) in 20 (or per batch).

TITLE: DIESEL RANGE ORGANICS/TOTAL PETROLEUM HYDROCARBONS (C_{10} TO C_{28}) BY GAS CHROMATOGRAPHY With #2 Diesel Fuel (EPA METHODS 8015B/C/D)

SOP NUMBER: 330350A

Prepared by: Judy Morgan

Reviewed by: Chris Johnson/Kelly Fox/Dixie Marlin

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_____
Department Manager

_____
QA Department

1.0 SCOPE AND APPLICATION

STATE NOTE: For samples analyzed in conjunction with the Ohio Voluntary Action Program (VAP) please utilize SOP# 330352, Revision 13 (2/19/09).

1.1 The diesel range organics (DRO) method is designed to determine the concentration of diesel range organics in water and soil using calibration performed with #2 diesel fuel standards. The "diesel range" corresponds to an alkane range of C_{10}-C_{28} and includes mid-range petroleum products such as diesel or fuel oil. Reporting limits are based on 100ug/mL of diesel in the extract and are as follows:

Reporting Limits (RL)

Ground water & Wastewater	0.10 mg/L
Soil & Sediment	4.0 mg/Kg
Waste (TCLP) (100→1)	1.0 mg/L

1.2 Dilutions are performed as necessary to place sample quantitation within the linear range of the calibration curve. This is equivalent to a range from 100ug/mL to 10,000ug/mL of diesel fuel in the extract.

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TITLE: DIESEL RANGE ORGANICS/TOTAL PETROLEUM HYDROCARBONS (C_{10} TO C_{28}) BY GAS CHROMATOGRAPHY With #2 Diesel Fuel (EPA METHODS 8015B/C/D)

1.3 An MDL study must be completed at least annually or more frequently if major instrumentation changes occur. Method Detection Limits (MDLs) are performed based on ESC SOP #030206. Updated MDL records are filed and stored in a central location within the department.

1.3.1 Limit of Detection (LOD) and Limit of Quantitation (LOQ) studies are completed at the frequency required by the TNI standard per the procedure identified in the ESC SOP #030206, *Method Detection Limits (MDL) and Limits of Detection (LOD).* Should the procedure be utilized for DOD support; then the frequency of these studies must meet the requirements of the current DOD QSM.

2.0 METHOD SUMMARY AND DEFINITIONS

2.1 Either one liter of water is extracted using EPA 3510, 100mL of water using EPA 3510 Reduced Volume (RV), 40mL of water using Large Volume Injection (LVI) by EPA 3511, 25 grams of soil using EPA 3550 (sonication) or 12.5 grams of soil using EPA 3546 (microwave) is spiked with a surrogate compound and extracted with methylene chloride. The extract is dried and concentrated to a volume of up to 5.0mL (RV or LVI), 1.0mL (water and sonication) or 0.5mL (microwave). One to 50uL of the extract is injected into a capillary column gas chromatograph equipped with a flame ionization detector (FID). Quantitation is performed by comparing the total chromatographic area to the response of a diesel standard. All chromatographic peaks eluting within the retention time windows determined by the appropriate carbon range will be considered. The required standard for this method is #2 diesel fuel. If bulk product is available from the sampling site, a calibration curve can be analyzed using the site-specific reference.

2.2 Initial Calibration Verification (ICV)/Continuing Calibration Verification (CCV) – Standards prepared from the primary source that are analyzed at the beginning of each workgroup (ICV), and following every 10 samples throughout the run and at the conclusion of the sequence (CCV) to confirm that the instrument maintains calibration stability within acceptable limits.

2.3 Continuing Demonstration of Capability (CDOC) – At least annual verification of analysts continued ability to perform method acceptably.

2.4 Initial Demonstration of Capability (IDOC) - A demonstration of capability (DOC) must be made prior to using any analytical method and any time there is a change in instrument type, personnel or testing method. Such performance must be documented and the four preparation batches following the change in personnel must not result in the failure of any batch acceptance criteria, e.g., method blank and laboratory control sample, or the demonstration of capability must be repeated. See also Continuing Demonstration of Capability (CDOC).

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2.5 Method Detection Limit (MDL) - The minimum concentration of a substance that can be analyzed with 99% confidence that the analyte concentration is greater than zero.

2.6 Laboratory Control Sample (LCS) / Laboratory Control Sample Duplicate (LCSD) – Duplicate aliquots of a control sample of known in composition. This sample is prepared from a source that is different from the stock used to prepare the initial and continuing calibration standards. LCS/LCSD are analyzed exactly like a sample and the purpose is to determine whether the methodology is in control and whether the laboratory is capable of making accurate and precise measurements. Method precision can be determined using the results of the LCS/LCSD analysis.

2.7 Matrix Spike (MS) / Matrix Spike Duplicate (MSD) - Two aliquots of a field sample (water or soil) spiked with known quantities of specific compounds and subjected to the entire analytical procedure in order to indicate the appropriateness of the method for the matrix by measuring recovery. Method precision can be determined using the results of the MS/MSD analysis, but are subject to matrix variability issues not present in the LCS/LCSD pair.

2.8 Method Blank - An analytical control consisting of all reagents used in the analytical procedure. The method blank is used to define the level of laboratory background and reagent contamination.

2.9 Second Source Calibration Verification (SSCV) – A mid-point or low standard made from a secondary standard that is not used to construct the calibration curve. The SSCV is used to represent the calibration accuracy of the instrument and must perform within method stated criteria.

2.10 Reporting Limit (RL) – Also see Practical Quantitation Limit (PQL). Routinely the reporting limit is the lowest standard of the calibration curve. Technically, the reporting limit is the lowest level that can be reliably achieved within the established limits of precision and accuracy during routine laboratory operating conditions.

2.11 Practical Quantitation Limit (PQL) – The default reporting limit when other limits are not specified by the client or project. The PQL is usually a factor of 3-10 times the MDL.

2.12 Surrogate - A compound, similar to the target analytes in chemical composition and behavior, but not expected to occur naturally in field samples. Analytes are spiked by preparation/analytical personnel to assess sample extraction and analytical efficiency in each individual field sample.

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2.13 Elution - The order of emergence of chemicals from the column of a chromatograph. The chemicals then typically flow into a detector of some type. Predicting and controlling the order of elution is a key aspect of column chromatographic methods and can be modified using instrument operating conditions, column selections, etc.

2.13.1 Co-elution – Peaks that are not distinctly separated or resolved by a chromatograph. Co-elution is problematic when peaks share primary and secondary mass ions making accurate quantitation questionable.

2.14 Retention Time – The expected time that it takes for a particular analyte to pass through the system (from the column inlet to the detector) under set conditions.

2.15 External Calibration - External standard calibration involves comparison of instrument responses from the sample to the responses from the target compounds in the calibration standards. Sample peak areas (or peak heights) are compared to peak areas (or heights) of the standards. The ratio of the detector response to the amount (mass) of analyte in the calibration standard is defined as the calibration factor (CF).

2.16 Calibration Factor (CF) - The ratio of the detector response (peak areas or peak heights) to the amount (mass) of analyte in the calibration standard.

2.17 Reporting Limit Verification (RLV) – A standard analyzed following initial calibration/calibration verification at or below the analyte concentration of the routine reporting level. It is analyzed per regulatory/method requirements for drinking water analyses and various other state/national regulatory programs to verify the accuracy of field sample results at the reporting level.

2.18 Calibration Standards – Solutions of known concentrations used to create graphic representation of the relationship between the known values, such as concentrations, and instrument responses.

2.19 Linear Regression - Mathematical technique for finding the straight line that best-fits the values of a linear function, plotted on a scatter graph as data points. If a 'best fit' line is found, it can be used as the basis for estimating the future values of the function by extending it while maintaining its slope.

2.20 Limit of Detection (LOD) - A laboratory's estimate of the minimum amount of an analyte in a given matrix that an analytical process can reliably detect in their facility. The validity of the LOD shall be verified by detection of the analyte(s) in a spiked clean matrix sample in each quality system matrix. This sample shall contain the analyte at no more than 3X the MDL for single analyte tests and 4X the MDL for multiple analyte tests. This verification shall be performed on every instrument that is to be used for analysis of samples and reporting of data. The samples used for this verification must be prepared and analyzed through all steps in the analytical process used for client samples.

2.21 Limit(s) of Quantitation (LOQ) - The minimum levels, concentrations, or quantities of a target variable (e.g., target analyte) that can be reported with a specified degree of confidence. The validity of the LOQ shall be verified by successful analysis of a spiked clean matrix sample containing the analytes of concern in each quality system matrix at 1 to 2 times the claimed LOQ. A successful analysis is one where the recovery of each analyte is within the laboratory established method acceptance criteria or client data quality objectives for accuracy. The samples used for this verification must be prepared and analyzed through all steps in the analytical process used for client samples.

3.0 HEALTH AND SAFETY

3.1 The toxicity or carcinogenicity of each reagent used in the laboratory has not been fully established. Each chemical should be regarded as a potential health hazard and exposure to these compounds should be as low as reasonably achievable. Each laboratory is responsible for maintaining a current awareness file of OSHA regulations regarding the safe handling of the chemicals specified in this method. A reference file of material data handling sheets should also be made available to all personnel involved in the chemical analysis. Specifically, concentrated nitric and hydrochloric acids present various hazards and are moderately toxic and extremely irritating to skin and mucus membranes. Use these reagents in a fume hood whenever possible and if eye or skin contact occurs, flush with large volumes of water. Always wear safety glasses or a shield for eye protection, protective clothing and observe proper mixing when working with these reagents.

4.0 SAMPLE PRESERVATION, CONTAINERS, HANDLING, AND STORAGE

4.1 All samples must have been collected using a sampling plan that addresses the considerations of this method.

4.2 Water samples are collected in a 1 Liter amber bottle with Teflon lined caps for traditional EPA 3510C extractions or in a 100mL amber bottle with Teflon lined caps for 3510RV extraction. LVI samples are collected in 40mL amber vials with Teflon lined caps. Water samples are preserved to a pH of <2 with concentrated HCl. Soils are collected in a glass jar with a Teflon™ lined cap. The samples are stored at 4 ± 2°C from the time of collection until the time of extraction. Extraction must be performed on waters within 7 days and soils within 14 days. All analyses must take place within 40 days of extraction.

4.3 Sample extracts shall be stored in appropriately sized vials with Teflon™ lined closures (screw or crimp top) at 4 ± 2°C.

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4.4 All glassware is cleaned as soon as possible after use by rinsing with the last solvent used. This is followed by detergent washing with hot water, and rinses with tap water and organic-free reagent water. Drain the glassware and dry in an oven at 130°C for several hours or rinse with methanol and drain. Store dry glassware in a clean environment. See SOP 030701, *Glassware Cleaning*.

5.0 INTERFERENCES

5.1 Interferences can be caused by the following:

5.1.1 Contaminated solvents or reagents

5.1.2 Sample processing hardware or glassware

5.1.3 Contaminated carrier gas

5.1.4 GC parts, column surfaces or detectors

5.1.5 Sample matrix

5.1.6 Other organic compounds; including chlorinated hydrocarbons, phenols and phthalate esters are measurable.

5.2 Method interferences are reduced by washing all glassware with hot, soapy water and then rinsing it with tap water, carbon filtered water, methanol, and methylene chloride. See SOP No. 030701, *Glassware Cleaning*.

5.3 High purity reagents such as pesticide grade methylene chloride must be used to minimize interference problems.

5.4 Common Chemical Contaminants:

*General organic compound*s: Animal and vegetable oil and grease, chlorinated hydrocarbons, phenols, and phthalate esters are measurable under the conditions of this method. Compounds eluting within the chromatographic retention window will be included in the diesel range organic results. If excessive interferences are noted, it may be necessary to utilize extract clean-up procedures such as those specified in SW-846.

Phthalate Esters: Special precautions must be taken to avoid contamination by phthalate esters. Phthalate esters are common plasticizers, frequently found in labware and supplies. Some of the phthalate peaks will fall within the retention window and be included in the quantitation of the diesel range organics. Care must be exercised to minimize the presence of phthalates by avoiding the use of plastics wherever possible.

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High molecular weight compounds: Samples containing high molecular weight compounds may cause residual instrument contamination. A solvent blank (injection of pure solvent) should be analyzed after such a sample to ensure that the chromatograph system is free from interferences before proceeding with additional sample analyses. To reduce carryover, the chromatography column may also require an extended bake-out to remove the high molecular weight material.

5.5 Contamination by carryover can occur whenever high-level and low-level samples are sequentially analyzed. Whenever an unusually concentrated sample is encountered, it must be followed by an analysis of a solvent blank to check for cross-contamination.

6.0 EQUIPMENT AND SUPPLIES

6.1 Syringes: (Use the following brands or equivalent)

VWR Cat #	Syringe Size
60361-136	10µL
60376-230	25µL
60376-220	100µL
60376-558	1mL

6.2 Volumetric Flasks, "Class A", with ground glass stoppers, various sizes, ranging from 10mL to 100mL.

6.3 Gas Chromatography

6.3.1 Instrumentation:

- Instrument ID: SVGC #7 or #16/LVI/RV: SVGC #21 or #25
- Model #: HP 6890 or HP7890
- Heating elements - Restek – GC Racer
- Column (type, brand, size): ZEBRON ZB-5, RTX 15m - 5 x 0.25 x 0. 5 or 30m equivalent
- Detector: FID
- Gases used (grade and supplier): Air – medical, H_2 – 5.0, and/or N_2 all supplied by AirGas
- Autosampler Syringes used (brand, size, type): Agilent 10uL up to 100uL
- Temperature programs can be found in each instruments maintenance log.

For further information on which instruments run which methods please see our QC department for MDL studies on each instrument.

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7.0 REAGENTS AND STANDARDS

7.1 All reagents and standards must be recorded in the appropriate preparation log and assigned a unique number. See SOP 030203, *Reagent Logs and Records*, and SOP 030230, *Standard Logger*. Additional information regarding reagent preparation can be found in the Standards Logger (Tree) digital archive system. All spiking solutions and surrogate standard solutions should be replaced at least every 6 months, or sooner if a problem is detected unless otherwise noted.

7.2 Reagent water: Carbon filtered de-ionized water

7.3 Methylene Chloride: Burdick and Jackson Pesticide grade - VWR Cat # BJ300-4 or equivalent

7.4 Stock Standard Solution: Pre-made NSI environmental UST145TP 50000ug/mL or equivalent. See table below for calibration standards.

7.4.1 Prepare an intermediate standard at 10000ppm for use in diluting calibration standards. Dilute 1mL of Stock Diesel Fuel #2 to a total of 5mL of methylene chloride. The final concentration is 10,000ug/mL.

Calibration Curve Standard Preparation In Methylene Chloride

Conc. Diesel (ppm)/ OTP (ppm)	Volume (µL) of Diesel Intermediate (10,000ppm)	Volume (µL) of OTP Intermediate (1000ppm)	Final Volume (mL)
100/5	10	5	1.0
200/10	20	10	1.0
50020	50	20	1.0
1000/25	100	25	1.0
2000/40	200	40	1.0
3000	300	-	1.0
5000	500	-	1.0
10000	1000	-	1.0

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7.4.2 For LVI or RV, prepare an intermediate standard at 200ppm for use in diluting calibration standards. Dilute 40uL of stock Diesel Fuel #2 to a total of 10mL of methylene chloride.

Calibration Curve Standard Preparation in Methylene Chloride (LVI CALIBRATION CURVE)

Conc. Diesel (ppm)/ OTP (ppm)	Volume (µL) of Diesel Intermediate (200ppm)	Volume (µL) of OTP Intermediate (20ppm)	Final Volume (mL)
2/0.1	10	5	1.0
4/0.2	20	10	1.0
10/0.4	50	20	1.0
20/0.5	100	25	1.0
40/0.8	200	40	1.0
60	300	-	1.0
100	500	-	1.0
200	1000	-	1.0

7.5 Custom DROPORT Standard Solution: Pre-made NSI environmental Q-4028 9000ug/mL or equivalent. See table below for calibration standards.

DROPORT Calibration Curve Standard Preparation In Methylene Chloride

Conc. Of Custom DRO (ppm)/ OTP (ppm)	Volume (µL) of Custom DRO (9000 ppm)	Volume (µL) of OTP Intermediate (1000 ppm)	Final Volume (mL)
90/5	10	5	1.0
180/10	20	10	1.0
450/20	50	20	1.0
900/40	100	40	1.0
1800/50	200	50	1.0
4500	500	-	1.0
9000	1000	-	1.0

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7.6 ICV & CCV Standard Preparation in Methylene Chloride

Conc. Diesel (ppm)	Volume (µL) from 50000 ppm	OTP Volume (µL) from 1000 ppm	Final Cal Std Volume (mL)
3000	600	20	10.0

LVI Conc. Diesel (ppm)	Volume (µL) from 3000ppm DRO CCV	OTP Volume (µL) from 1000 ppm	Final Cal Std Volume (mL)
60ppm	200	-	10.0

Conc. DROPORT Diesel (ppm)	Volume (µL) from 9000 ppm	OTP Volume (µL) from 10000 ppm	Final Cal Std Volume (mL)
1800	1000	25	5.0

Concentration levels are subject to change depending on instrument with the exception of the low and high concentrations.

7.7 Stock Laboratory Control Sample/SSCV: 7,500 ug/mL diesel #2. Working solution is made up at 1,500ug/mL in acetone. Transfer the stock standard solution into a Teflon-sealed screw cap bottle. Store away from light with minimal headspace at -10°C to -20°C.

7.8 Surrogate Standard: Ortho-Terphenyl (OTP), Ultra Scientific IST-480 (or equivalent), 10000ug/mL in methylene chloride. Store refrigerated. Do not store above 35°C. Surrogates are added to the field samples during the extraction process.

7.8.1 Surrogate (OTP) Intermediate standard: Dilute 0.5mL of Stock OTP to a total of 5mL of methylene chloride. The final concentration is 1,000ug/mL.

7.8.2 LVI/RV Surrogate (OTP) Intermediate standard: Dilute 20uL of Stock OTP (10,000 ppm) up to 10 mL with $MeCl_2$. The final concentration is 20ug/mL.

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7.9 Carbon Number Marker Compounds – the regulatory state sets quantitation ranges based on carbon number. Table 7.9 lists the compounds used as marker compounds for various carbon number ranges. The marker compounds standard is purchased from Ultra Scientific, Catalog No. SFL-601.

Table 7.9: Carbon Number Marker Compounds

Carbon Number	Compound	Approximate Retention Time (min.)[1]
8	n-octane	0.37
10	n-Decane	0.83
12	n-Dodecane	1.25
14	n-Tetradecane	1.63
16	n-Hexadecane	1.99
18	n-Octadecane	2.33
Surrogate	o-terphenyl	2.45
20	n-Eicosane	2.64
22	n-Docosane	2.93
24	n-Tetracosane	3.19
26	n-Hexacosane	3.45
28	n-Octacosane	3.71
30	n-Triacontane	3.95
32	n-Dotricontane	4.19
34	n-Tetratricontane	4.42
36	n-Hexatriacontane	4.65
38	n-Octatriacontane	4.85
40	n-Tetracontane	5.05

[1] *Approximate retention times. Actual retention times are verified during instrument calibration. These RT's are subject to change and are here as a representation only. These RT's will be different from instrument to instrument.*

8.0 PROCEDURE

NOTE: Waters are extracted according to ESC SOP #330702, *Separatory Funnel Liquid-Liquid Extraction (EPA Method 3510)*, or SOP #330743, *Solid Phase Extraction (EPA Method 3535)*. Soil samples are extracted using SOP #330705, *Sonication Extraction (EPA Method 3550) or* SOP #330707, *Microwave Extraction (EPA Method 3546)*. Large volume injection samples are extracted according to ESC SOP #330709, *Micro-Extraction Procedure for Diesel Range Organics* (EPA Method 3511). Reduced volume injection samples are extracted according to ESC SOP #330702B (EPA Method 3510C). Refer to the specific SOP for additional direction regarding sample preparation.

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8.1 **Gas Chromatography**

Before starting an analytical sequence, an initial check and maintenance of the instrument must be performed

8.1.1 Example conditions: Set H_2 column flow to 5mL/minute. Column temperature starts at:

60°C ramps to 350°C on CTM A68
80°C ramps to 350°C on racer
80°C Holds at 0.25min
Ramps 240°C at 90°C/min
280°C at 80°C/min
325°C at 70°C/min
350°C at 60°C/min
Hold at 350°C for 0.66min

The FID temperature should be at 320°C and the injector temperature should be set at 300°C.

Check Injection Logs for individual instrument conditions. Each instrument has optimum conditions and programs that are set for the method. If a new instrument is installed, then follow method recommended conditions listed above.

8.2 **Calibration**

8.2.1 Blanks - Before beginning calibration or analysis, run at least 1 methylene chloride blank to ensure that the instrument does not have any contamination from previous use. If contamination is observed, run additional methylene chloride blanks to clean and verify that the analytical system is ready to use in field sample analysis.

8.2.2 Retention Marker Standard - Before any new calibration curve is analyzed, a marker standard comprising of the appropriate markers (see Table 7.9) for the specific analysis requested is analyzed to set calibration range and retention times.

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8.2.3 Initial Calibration - Load the autosampler with the calibration standards. Calibrate the GC with an initial eight-point calibration using the commercial diesel standard (Section 7.4). Tabulate the area response of the diesel standard. The ratio of the peak response to the amount injected, defined as the calibration factor (CF), can be calculated for the standard at each concentration. If the percent relative standard deviation (%RSD) is <20% over the working range of all standards, linearity through the origin can be assumed and the calibration factor can be used in place of a calibration curve. If the %RSD is beyond 20%, then linear calibration may be employed. The correlation coefficient for linear calibration must be 0.990 or better (USACE requires 0.995 or better). See section 9.1 and 9.2 for calculations.

STATE NOTE: For DRO sample in OH, the total range of C_{10}- C_{34} is evaluated and the response given for the total is inserted into the 2 separate ranges for calibration.

8.2.4 Integration - For each standard analyzed, set the data system to begin integrating peaks after the solvent front and 0.05 minutes before the apex of the C_{10} peak. Stop the integration after upper limit of the retention time of the C_{28} peak. The peak integration parameters should be set to integrate to the baseline such that the area in the unresolved complex is included. Valley-to-valley integration is not permitted.

STATE NOTE: For DRO samples from Ohio, the integration should begin at 0.05min. before the apex of the C_{10} peak to 0.05min. after the apex of the C_{20} peak with the second range beginning at 0.05min. after the apex of the C_{20} peak to 0.05min. after the apex of the C_{34} peak.

STATE NOTE: For DRO- CA the integration should begin at 0.05min. before the apex of the C_{10} peak to 0.05min and after the apex of the C_{22} peak

STATE NOTE: For DRO- IN the integration should begin at 0.05min. before the apex of the C_{10} peak to 0.05min and after the apex of the C_{28} peak

CLIENT NOTE: For DROPORT the ranges are broken down to C_{10} - C_{20} and C_{20} - C_{28}. And further into C_{10} - C_{12}, C_{12} - C_{16}, C_{16} - C_{21}, and C_{21} - $C_{28.0}$

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8.2.5 Second Source Calibration Verification (SSCV) – Following each initial calibration, a SSCV must be analyzed to ensure the accuracy of the standard solutions used to perform instrument calibration. The SSCV is analyzed at 1500ppm and is prepared using the commercial diesel standard (Section 7.6). If the response for this standard varies from the predicated response by more than ±20%, a new calibration curve must be prepared. See section 9.4 for the equation to calculate recovery.

8.2.6 Calibration Verification - The appropriate retention time and working calibration factor or linear calibration curve must be verified at the beginning of each 12 hour work shift, by the injection of a retention time marker and a mid-point continuing calibration verification (CCV) standard. A 3,000ug/mL diesel standard, or 60ug/mL diesel standard is used for this standard. See section 9.6 for the equation to calculate percent difference. If the response for this standard varies from the predicated response by more than ±15%, a new calibration curve must be prepared.

STATE NOTE: For DRO-CA, CCV criteria is ±10% for the petroleum range.

8.3 Retention Time Windows:

8.3.1 Before establishing windows, be certain that the GC system is within optimum operating conditions. Make three injections of the method standard throughout the course of a 72 hour period. Serial injections over less than a 72 hour period results in retention time windows that are too tight.

8.3.2 Calculate the standard deviation of the three absolute retention times for the surrogate standard.

8.3.2.1 The retention time window for individual peaks is defined as plus or minus three times the standard deviation of the absolute retention time for each component.

8.3.2.2 In those cases where the standard deviation for a particular analyte is zero, ±0.05 min is the retention time window.

8.3.3 Retention time windows must be calculated for the surrogate standard on each GC column and whenever a new GC column is installed. Retention time information must be recorded in the Instrument log. The instrument log must reflect the date that the retention time windows are calculated and the dates of the standards used to calculate the windows. The data is retained by the laboratory and is traceable through instrument logs.

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8.4 Gas Chromatograph Analysis:

Typical Batch order for loading the autosampler:

Sample/QC Type	Use
Solvent Blank (minimum of 1)	Verify system is contamination free
Retention Time Marker	Prior to calibration or calibration verification and every 12/24 hours – depending on method
Calibration standard(s)/SSCV	Initial 8-point calibration followed by a Second Source Calibration Verification.
Initial Calibration Verification (ICV)	Verify initial 8-point calibration.
Method blank	Ensure that carry over has not occurred from the calibration standard, and that the analytical system does not show contamination above the established detection limits
Laboratory Control Sample(s)	Extracted laboratory blank, spiked with known amount(s) of analyte of interest
1 to 10 samples	Client samples
Continuing Calibration Verification (CCV)	Single-point calibration verification standard.
Solvent Blank	Verify system is contamination free
1 to 10 samples	Client samples
Continuing Calibration Verification (CCV)	Single-point calibration verification standard.
Solvent Blank	Verify system is contamination free

8.4.1 Samples are analyzed by GC/FID. Injection volumes are 1- 2uL using the conditions established in 8.1. Injection volumes for LVI/RV are 50uL using the conditions established in 8.1.

8.4.2 If initial calibration has been performed, verify the retention time then the continuing acceptability and accuracy of the calibration by analysis of a mid-point (3,000ug/mL diesel standard/60ug/ml diesel standard). The mid-point standard must also be run once after every ten samples and at the end of each sequence, or once every 12 hours (whichever is more frequent).

8.4.3 CCV Criteria - Calculate the percent difference of the calibration factor from the mean calibration factor. If the calibration factors have a percent difference of >15%, the instrument must be re-calibrated.

STATE NOTE: For DRO-CA, CCV criteria is +10% for the petroleum range.

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8.4.4 Baseline Subtraction - A methylene chloride blank must be run every 10 samples (or every 12 hours, whichever comes first) to determine the area generated by normal baseline bleed under the conditions prevailing in the 24 hour period. This area is generated by projecting a horizontal baseline between the retention times observed for C_{10} and C_{28}. This area is subtracted from the DRO area generated in the same manner for the samples. Methylene chloride blanks should also be run after samples suspected of being highly concentrated to prevent carryover.

8.4.5 If any field sample extract concentration exceeds the linear range of the instrument, the extract must be diluted and re-analyzed.

8.5 **Chromatogram appearance**

8.5.1 Chromatogram Interpretation - The analyst should generate a value for both diesel range organics and diesel or other products. Identification of diesel or other products is performed by comparing the retention times and patterns of the peaks in the sample chromatogram to those of the peaks in the standard chromatogram. The experience of the analyst weighs heavily in the interpretation of the chromatogram. Quantitation of the diesel range organics is based on summation of all peaks eluting between n-decane and n-octacosane in the most recent retention time marker.

Baseline Subtraction - Because the chromatographic conditions employed for DRO analysis can result in significant column bleed and a resulting rise in the baseline, it is appropriate to perform a subtraction of the column bleed from the area of the DRO chromatogram. In order to accomplish this subtraction, a methylene chloride blank should be analyzed during each 12-hour analytical shift during which samples are analyzed for DROs. The area of this chromatogram is measured in the same fashion as is used for samples by projecting a horizontal baseline across the retention time range for DROs. This area is then subtracted from the area measured for the sample and the difference in areas is used to calculate the DRO concentration,

8.5.2 Reporting Non-Diesel Detections - Other organic compounds, including chlorinated hydrocarbons, phenols, and phthalates are measurable by this method and will be reported as diesel range organics when they fall within the determined carbon range window. A comment will be made in the data report about the presence of non-diesel materials that appear in the diesel range.

NOTE: Although the retention time window definition (n-decane to n-octacosane) introduces a bias (55 to 75% for diesel), it improves precision and reduces interferences from non-diesel range components.

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8.5.3 Additional Identification and Special Qualifiers - If standards are available, additional products can be qualitatively identified using this method. These products could include aviation fuel, kerosene, fuel oil #4 and #6, turpentine, and creosote. Additional products that elute outside the diesel range (C_{10} - C_{28}) may be qualitatively identified, but cannot be quantitated using this method. These products include gasoline, lubricating oils, and silicone oils. See the following table.

Table 8.5: Qualifiers

Qualifier	Description/Use
Y0	Significant peaks were detected outside of the hydrocarbon range defined by the method.
Y1	This sample most closely matches the laboratory standard for Diesel
Y2	This sample most closely matches the laboratory standard for #6 Fuel Oil
Y3	This sample most closely matches the laboratory standard for Hydraulic Fluid
Y4	This sample most closely matches the laboratory standard for Motor Oil
Y5	This sample has responded in the Diesel range, however it does not appear to be a hydrocarbon product.
Y6	This sample has responded in the Oil range, however it does not appear to be a hydrocarbon product.

9.0 DATA ANALYSIS AND CALCULATIONS

9.1 External Calibration Equations:

- The calibration factor for each standard can be calculated:

$$CF = \frac{A_s}{C_s}$$

where: A_s - Average Peak Area over the number of peaks used for quantitation
C_s – Concentration of the analyte in the standard.

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- The average (or mean) calibration factor ($\overline{CF}$) is calculated:

$$\overline{CF} = \frac{\sum_{i=1}^{n} CF_i}{n}$$

where: CF_i – Calibration Factor for each level of the calibration curve
n – number of standards analyzed in the calibration curve

- The standard deviation (SD) of the calibration is determined:

$$SD = \sqrt{\frac{\sum_{i=1}^{n} (CF_i - \overline{CF})^2}{n-1}}$$

where: $\overline{CF}$ – Average Calibration Factor for the calibration curve
CF_i – Calibration Factor for each level of the calibration curve
n – number of standards analyzed in the calibration curve

- The Percent Relative Standard Deviation for each analyte in the curve is determined:

$$RSD = \frac{SD}{\overline{CF}} \times 100$$

where: SD – Standard Deviation for each analyte
$\overline{CF}$ – Average calibration factor for the specific analyte

9.2 Linear calibration model:

$$y = mx + b$$

where: y = Response A_X for External Standard
x = Concentration C_X for External Standard
m = Slope
b = Intercept

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- Slope (m):

$$m = \frac{[(Swx_iy_i * Sw) - (Swx_i * Swy_i)]}{[(Sw * Swx_i^2) - (Swx_i * Swx_i)]}$$

- Intercept (b):

$$b = y_{AVE} - (m * (x_{AVE}))$$

- Correlation Coefficient (r):

$$r = \frac{[(Sw * Swx_iy_i) - (Swx_i * Swy_i)]}{\sqrt{\{[(Sw * Swx_i^2) - (Swx * Swx_i)] * [(Sw * Swy_i^2) - (Swy_i * Swy_i)]\}}}$$

- Coefficient of Determination (r^2):

$$r^2 = r * r$$

Where: n = number of x, y pairs
x_i = individual values for the independent variable
y_i = individual values for the dependent variable
w = weighting factor, for equal or no weighting w = 1
x_{AVE} = average of the x values
y_{AVE} = average of the y values
S = the sum of all the individual values

9.3 Sample Quantitation: calculations are performed by the HP Enviroquant software, according to the following:

$$\text{Concentration} = \frac{\text{(ug/mL from curve) X (Volume of extract in mL) X (dilution factor)}}{\text{(sample amount extracted in mL or grams)}}$$

9.4 LCS/ICV/CCV Percent Recovery (%R):

$$\%R = \frac{\text{Measured concentration}}{\text{Actual concentration}} \times 100$$

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9.5 Matrix Spike Recoveries (%$R_{MS/MSD}$):

$$\%R_{MS/MSD} = \frac{O_i - O_s}{T_i} X 100$$

where: O_i = observed sample concentration with the spike added
O_s = the observed value for the sample without the spike
T_i = True value of the spike added

9.6 Relative Percent Difference (%RPD):

$$RPD = \frac{Value\ 1 - Value\ 2}{\left(\frac{Value\ 1 + Value\ 2}{2}\right)} \times 100$$

10.0 QUALITY CONTROL AND METHOD PERFORMANCE

10.1 All analysts must meet the qualifications specified in SOP 030205, *Technical Training and Personnel Qualifications* before approval to perform this method. Analysts must complete an initial demonstration of proficiency before being approved to perform this method. Continuing proficiency must be demonstrated using proficiency testing, laboratory control sample analysis and/or MDL studies. Method performance is assessed per analyst. Updated method performance records are filed and stored in a central location within the department.

10.2 Use the designated Run log to record batch order and standards/reagents used during analysis. See SOP 030201, *Data Handling and Reporting*.

10.3 Batches:

Batches are defined as sets of 1 - 20 samples. Batch analysis must include the following: 1 retention time marker every 12/24 hours as required by the applicable method, 1 method blank for every 12 hours or 10 samples, 1 Laboratory Control Sample (LCS), 1 Initial Calibration Verification (ICV), 1 Matrix Spike/Spike Duplicate (MS/MSD), 1 Continuing Calibration Verification (CCV) every 10 samples, 1 CCV at end of run, 1 surrogate control sample. All batch information must be maintained in the preparation documentation assigned to the department.

10.4 Retention Time Marker - The marker standard comprising of the appropriate carbon range markers (see Table 7.9) is analyzed prior to calibration or calibration verification and every 12/24 hours (per specific method) to ensure accurate quantitation for the diesel range addressed in this procedure.

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10.5 Initial Calibration - Run a 5 to 8 point initial calibration curve, using the primary source standards each time major instrument maintenance occurs, or if the CCV does not meet acceptance criteria. The percent relative standard deviation for the initial calibration curve must be <20%. If linear calibration is used, the correlation coefficient must be 0.990 or better (USACE requires 0.995 or better).

10.6 Surrogate Control Sample - After successful calibration, analyze a surrogate control sample. This standard is also the reagent blank sample and is analyzed with every analytical batch or sequence. The surrogate recovery should be within the established limits of 50-150% recovery and the sample should not have Diesel Range Organics above 1/2 of the reporting limit.

10.7 Second Source Calibration Verification (SSCV) – Following each initial calibration, a SSCV must be analyzed to ensure the accuracy of the standard solutions used to perform instrument calibration. The response must be within ±20% of the expected concentration.

10.8 ICV/CCV - Run a mid-point Initial Calibration Verification (ICV) using the primary source standards on a daily basis before sample analysis. Also run a CCV every 10 samples during an analytical sequence. The ICV/CCV must recovery within ±15% of the expected value. The concentration of the CCVs is changed routinely to verify the entire calibration range. A successful CCV is required at the end of the analytical sequence. All samples must be bracketed by calibration verification standards that meet the acceptance criteria.

STATE NOTE: For DRO-CA, CCV criteria is +10% for the petroleum range.

10.9 Method Blank - A Method Blank is analyzed with every batch of 20 samples. The quantitation of diesel range organics must be <½ RL,

10.10 LCS/LCSD - A Laboratory Control Sample (LCS)/Laboratory Control Sample Duplicate (LCSD) pair is run with every 20 samples. The accuracy of the LCS and LCSD must be within 50-150% and the RPD must be ≤20%.

10.11 Surrogate - Calculate the surrogate standard recovery in each sample. The accuracy of the surrogate must be within 50-150%.

10.11.1 High recoveries may be due to co-eluting matrix interference: examine the sample chromatogram.

10.11.2.Low recoveries may be due to the sample matrix.

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10.12 RLV – The reporting limit verification when analyzed must recover within ±50% of the target concentration for the standard.

STATE NOTE: For all samples analyzed from Minnesota, the reporting limit must be verified at least monthly. The reporting limit verification (RLV) must recovery within ±40% of the expected concentration. If this criteria is not met, the RLV may be re-analyzed once, instrument maintenance can be performed or a higher concentration standard can be analyzed. If a higher concentration standard is utilized, the reporting limit must be raised to the higher level verified.

10.13 MS/MSD - A Matrix Spike (MS)/Matrix Spike Duplicate (MSD) pair is run with every 20 samples. The accuracy of the MS and MSD must be within 50-150% and the RPD must be ≤20%.

10.14 Any sample analyte responses that are beyond the linear range of the calibration curve must be diluted and re-analyzed.

10.15 Manual Integration – All manual integrations must comply with the requirements found in ESC SOP# 030215, *Manual Integration Procedure*. Before and after integrations must be available for review by the secondary data reviewer.

10.16 Field blanks, duplicates, and additional quality control samples may be recommended for specific sampling programs. Matrix spikes should use the spike levels specified for laboratory control samples. (1500 ug/mL diesel)

10.17 Control limits must be established for both precision and accuracy. Control Limits must be the average recovery ±3 standard deviations (SD). The Warning Limits must be the average recovery ±2SD. See SOP 030207, *Quality Control Charting and Tracking*.

11.0 DATA VALIDATION AND CORRECTIVE ACTION

11.1 All data must undergo a primary review by the analyst. The analyst must check the performance of the initial calibration, mid-point check standard, and continuing calibrations to ensure that they meet the criteria of the method. The analyst should review any sample that has quantifiable compounds and make sure that they have been confirmed, if needed. The analyst must also verify that reported results are derived from quantitation between the RL and the highest standard of the initial calibration curve. All calculations must be checked (any dilutions, %solids, etc.). Data must be checked for the presence or absence of appropriate flags. Comments should be noted when data is flagged.

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11.2 All data must undergo a second analyst review. The analyst checking the data must check the performance of the initial calibration, mid-point check standard, and continuing calibrations to ensure that they meet the criteria of the method.

11.2.1 The analyst should look at any sample that has quantifiable compounds and check the integration.

11.2.2 All calculations must be checked.

11.2.3 All surrogate recoveries must be checked to see if they are within limits.

11.2.4 Blanks must be clean of all interfering peaks.

11.2.5 Quality control criteria should be checked for the LCS, MS, and MSD.

11.2.6 Data must be checked for the presence or absence of appropriate flags. Comments should be noted when data is flagged.

11.2.7 See SOP #030201, *Data Handling and Reporting*.

11.2.8 See SOP# 030208, *Corrective Action*.

11.3 Initial calibration - If the initial calibration does not meet the criteria for acceptance using calibration factors, then linear regression can be utilized, as long as the correlation coefficient meets the necessary criteria. If the linear regression criteria cannot be met, additional corrective actions are required. Standards must be reviewed and re-prepared, if necessary. Instrument maintenance may also be required, including injection port cleaning, column clipping/replacement, etc. When corrective actions have been completed, the instrument must be re-calibrated and the acceptance criteria must be met for the analytes of interest prior to the analysis of any field samples.

11.4 Method Blank - If the blank shows any detectable amount greater than ½ the RL, the laboratory performance is assumed to be out of control and the problem must be corrected. Corrective actions include: re-analysis once. If the failure persists, re-extract the entire batch of samples, if submitted sample volume permits. If acceptable to the client, the data may be flagged with a B when the analyte concentration in the field sample is 10 times greater than the analyte contained in the blank.

STATE NOTE: For samples analyzed in conjunction with Ohio VAP, when target analyte concentrations are above the reporting limit, samples must be re-extracted and re-analyzed, if sufficient sample volume was submitted by the client, prior to flagging the data report.

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11.5 Initial/Calibration Check Standard (ICV/CCV) - When the initial or continuing calibration verification is out of the acceptance criteria, the analysis must stop and corrective action must be taken to determine the cause of the problem. Corrective actions include: re-analysis of the ICV/CCV once. If the failure persists, additional corrective actions include: instrument maintenance, re-preparing the calibration standard, re-calibration of the instrument. Samples analyzed between the last passing calibration standard and the calibration standard that is out of control must be re-analyzed.

11.6 Laboratory Control Sample/Laboratory Control Sample Duplicate (LCS/LCSD) - If the control does not perform within the ranges listed in Attachment II, or current control ESC limits, the laboratory performance is assumed to be out of control and the problem must be corrected. Corrective action can include re-analysis, if instrument malfunction is suspected, or re-preparation and re-analysis of the entire batch, if the failure is suspected as either extraction or sample related.

11.7 Surrogates - If the recovery is not within limits stated in Attachment II or ESC current control limits, confirm that there are no errors in the calculations, surrogate solutions and standards. Check the instrument performance. Examine the chromatograms for interfering peaks and integrated areas. Re-calculate the data and/or re-analyze the extract if any of the above checks reveal a problem. Re-extract and re-analyze the sample if none of the above are a problem or flag the data "J1" (surrogate high) or “J2” (surrogate low).

11.7.1 High recoveries may be due to co-eluting matrix interference: examine the sample chromatogram.

11.7.2 Low recoveries may be due to the sample matrix.

11.8 RLV – If the RLV does not meet the acceptance criteria, the RLV may be re-analyzed once, instrument maintenance can be performed, a higher concentration standard can be injected, or a new calibration curve must be generated. If a higher concentration standard is utilized, the reporting limit for the field samples must be elevated to the higher level verified.

11.9 Second Source Calibration Verification (SSCV) – If the SSCV does not meet the accuracy requirement, the initial calibration standards must be reviewed and re-prepared, if necessary. Instrument maintenance may also be required, including injection port cleaning, column clipping/replacement, etc. When corrective actions have been completed, the instrument must be re-calibrated and the acceptance criteria must be met for the analytes of interest prior to the analysis of any field samples.

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11.10 MS/MSD – If the spike and spike duplicate do not meet the criteria listed in section 10.13, or current ESC quality control acceptance criteria, the sample must be flagged as possible matrix interference.

11.10.1 Spike failure that result in the use of a "J" flag followed by the appropriate number, which further explains the failure concerning high or low bias

11.11 Data that does not meet acceptable QC criteria may be acceptable for use in certain circumstances.

11.11.1 If a method blank contains an amount of target analyte, but all samples are non-detected, the data may be reported with a “B3” flag. If a method blank contains an amount of target analyte, but the samples contain analyte at a level that is 10 times the level present in the method blanks, the data may be reported with a “B” flag.

STATE NOTE: The Ohio VAP program does not accept data released using the 10X criteria for method blank contamination as noted in section 11.11.1.

11.11.2 If the MS/MSD fails (recovery less than 30% or greater than 150% and/or RPD greater than 30%) in an initial analysis and again upon re-analysis, the data is released with an appropriate qualifier as the failure is accepted as matrix related.

11.11.3 If a calibration verification standard is above the acceptable QC criteria and all samples being bracketed are below the reporting limit, the data is acceptable based on a high calibration bias with undetectable levels in the field samples. Any positive samples require re-analysis.

11.11.4 If the surrogate exhibits high recovery in the field samples and the target analytes in the field samples are below the reporting limit, the data may be released with a J1 qualifier indicating the high bias. If the QC samples (LCS, LCSD, MS, MSD) exhibit a high bias in the surrogate and the field samples are below the reporting limit for the target analyte, the data may be released with a J1 qualifier.

11.11.5 If the target analyte spiked in the quality control samples (LCS, LCSD, MS, MSD) exhibits high recovery and the target analytes in the field samples are below the reporting limit, the data may be released with a J4 qualifier indicating the high bias.

11.11.6 If the target analyte spiked into the QC pair (LCS/LCSD, MS/MSD) exhibit acceptable recoveries, but high calculated RPD values for precision, and the target analytes in the field sample are flagged with a J3 for the precision beyond acceptable quality control limits.

11.11.7 Sample results can be qualified and possible bias is narrated per the ESC SOP# 030201, *Data Handling*.

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STATE NOTE: If field samples are analyzed in conjunction with the Ohio VAP program, surrogate outliers in batch QC samples, including the blank, LCS/LCSD, MS/MSD require re-extraction of the entire batch, if sufficient volume has been submitted by the client and an obvious matrix interferent is not present.

12.0 POLLUTION PREVENTION AND WASTE MANAGEMENT

12.1 The EPA requires that laboratory waste management practice to be conducted consistent with all applicable federal and state laws and regulations. Excess reagents, samples and method process wastes must be characterized and disposed of in an acceptable manner. See *ESC Waste Management Plan.*

12.2 See SOP #030302, *Pollution Prevention*.

13.0 METHOD MODIFICATIONS/CLARIFICATIONS

13.1 No major method modifications have been made. ESC has added the ability to further identify DRO responses by employing the use of specific standards to provide compound information, if the DRO response is atypical from actual diesel.

13.2 Modifications to this method are noted in the body of the text as state notes. Compliance analyses performed in conjunction with specific state requirements must be performed as noted within the specific state(s) note listed.

13.3 ESC uses a GC Racer (from Restek) or the A68 Door (from LTM) to assist the GC oven to have a faster ramp.

13.4 EPA 3510 RV: The reduction of the size of the field sample used in this procedure is performed in accordance with section 7.1 of the published EPA method. The reduction in volume extracted along with analysis of the resulting extract using large volume injection (up to 250uL can be injected with the LVI injection port) on each GC allows for low detection limits in line with those obtained using a 1L extraction and the 1-2uL injection. Complete method validation is performed for each determinative method prior to utilizing the reduced volume extraction. This validation is maintained by the Regulatory Affairs Department and is regularly verified using LCS/LCSD, MDL studies and DOCs.

13.5 EPA 3510 RV: Extractions are performed using solvent volumes of 6mL, then 6mL and then 6mL, to accommodate the100mL sample volume; rather than using 60mL of solvent three times. The sample volumes used in this procedure, when compared to the field sample volume being extracted, remain consistent with the ratios present in the published method.

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14.0 REFERENCES

14.1 USEPA "SW-846 Test Methods for Evaluating Solid Waste", Methods 8000B, 8000C, 8015B, 8015C, 8015D, 3510C, 3511, 3546, and 3550C.

14.2 California - Leaking Underground Fuel Tank Field Manual: Guidelines For Site Assessment, Cleanup, and Underground Storage Tank Closure, October 1989, State of California Leaking Underground Fuel Tank Task Force

14.3 California - Leaking Underground Fuel Tank Field Manual: Guidelines For Site Assessment, Cleanup, and Underground Storage Tank Closure (Draft), October 2010, State of California Water Resources Control Board.

14.4 Indiana - RISC Technical Guide – Approved February 15, 2001. Chapter 8 “Total Petroleum Hydrocarbons”, Table 7.1.

14.5 Ohio – Ohio Administrative Code, 1301:7-9-13 (H)(1)(c) Table 1.

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Attachment I: Revision History

Current Version:

Version	Date	Description of Revisions
2	7/5/12	Technical and Quality Review and update. Revised Attachment II and sections 2.1, 2.2, 2.5, 2.9, 4.2, 6.3.1, 7.5, 7.6, 7.7, 8.0 (note), 8.2.2, 8.2.6, 8.4, 9.1, 9.2, 9.4 through 9.6, 10.3, 10.4, 10.7, 10.9, 11.1, 11.8, 12.1 and 14.1; Added sections 1.3.1, 2.12 through 2.21, 5.5, 7.5, 10.12 through 10.15, 11.9 through 11.11, 13.4 through 13.5 and 14.2 through 14.5; Added state notes in sections 8.2.3, 8.2.4, Deleted sections 7.4 through 7.6.

Superseded Versions:

This document supersedes the following:

Version	Date	Description of Revisions
0	4/14/04	Origination
1	8/17/09	Technical and Quality Review and update.

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TITLE: DIESEL RANGE ORGANICS/TOTAL PETROLEUM HYDROCARBONS (C_{10} TO C_{28}) BY GAS CHROMATOGRAPHY With #2 Diesel Fuel (EPA METHODS 8015B/C/D)

Attachment II: ESC Tech Note to SOP 330350A R2

DRO, DROOH, DROCA, DROIN

Test Name (Parameter Name)	Alkane Range
DRO (TPH GC/FID High Fraction)	$C_{10} – C_{28}$
DROOH	$C_{10} – C_{20}$; $C_{20} – C_{34}$
DROCA	$C_{10} – C_{22}$
DROIN	$C_8 – C_{28}$

Standard (PPM)	Limits (PPM)
DRO 3000 / 60 (85 - 115%)	2550 – 3450 / 51 – 69
OTP 20 / 0.4 (50 – 150%)	10 – 30 / 0.2 – 0.6
Routine Reporting Limits:	
Soil	4.0 mg/Kg
Water (Groundwater/Waste water)	0.1 mg/L
Waste (TCLP)	1.0 mg/L

LCS/D Spike (PPM)	Range with Multiplier (PPM)
Soil Spike 60 (50 - 150%)	Soil Spike (Multiplier = 0.04) 30 - 90
Water Spike 1.5 (50 - 150%)	Water Spike (Multiplier = 0.001) 0.75 – 2.25
OTP Surrogate Spike (PPM)	**Range with Multiplier (PPM)**
Soil Spike 0.8 (50 - 150%)	Soil Spike (Multiplier = 0.04) 0.4 – 1.2
Water Spike 0.02 (50 - 150%)	Water Spike (Multiplier = 0.001) 0.01 – 0.03

	Calibration
%RSD	<20%
Linear Correlation Coefficient	>0.990 (>0.995 for USACE)
SSCV	+20%

[1]$MeCl_2$ Subtraction must be performed for all ranges
[2]Blank must be below ½ RL

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DROPORT

Test Name (Parameter Name)	Alkane Range
DROPORT (TPH C_{10}-C_{20}, C_{20}-C_{28})	C_{10} – C_{28}
(TPH >C_{20} - C_{28})	C_{20} – C_{28}
(TPH >C_{10} – C_{12})	C_{10} – C_{12}
(TPH >C_{12} – C_{16})	C_{12} – C_{16}
(TPH >C_{16} – C_{21})	C_{16} – C_{21}
(TPH >C_{21} – C_{28})	C_{21} – C_{28}

Standard (PPM)	Limits (PPM)
DROPORT 1800 (85 - 115%)	1530 - 2070
OTP 50 (50 – 150%)	25 – 75
Routine Reporting Limits:	
Soil	4.0 mg/Kg
Water (Groundwater/Waste water)	0.1 mg/L
Waste (TCLP)	1.0 mg/L

LCS/D Spike (PPM)	Range with Multiplier (PPM)
Soil Spike 60 (50 - 150%)	Soil Spike (Multiplier = 0.04) 30 - 90
Water Spike 1.5 (50 - 150%)	Water Spike (Multiplier = 0.001) 0.75 – 2.25
OTP Surrogate Spike (PPM)	**Range with Multiplier (PPM)**
Soil Spike 0.8 (50 - 150%)	Soil Spike (Multiplier = 0.04) 0.4 – 1.2
Water Spike 0.02 (50 - 150%)	Water Spike (Multiplier = 0.001) 0.01 – 0.03

Calibration	
%RSD	<20%
Linear Correlation Coefficient	>0.990 (>0.995 for USACE)
SSCV	+20%

[1]$MeCl_2$ Subtraction must be performed for all ranges
[2]Blank must be below ½ RL
[3]DROPORTC is a DROPORT sample with silica gel cleanup

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SOP Revision Summary

SOP:					
Author -	Michael Jacobs	**Number -**	330363	**Department -**	Volatiles
Title -	VOLATILE ORGANIC COMPOUNDS BY GC/MS (EPA 8260B, 8260C, 624 AND SM6200B 20TH)				
Revision -	R17	**Rev. Date -**	3/12/12		

This Standard Operating Procedure has been amended to include changes required during normal business operations. These changes as defined by SOP 010103 (Document Control and Distribution) are routine modifications that will be incorporated into the SOP upon the next scheduled review.

Rev.	Date	Section	Brief Description
1	10/10/12	8.3.1.4	Clarification of section and inclusion of SC state note
2	12/14/12	8.2.7.3 & 10.1	Inclusion of SC requirements

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SOP Revision Summary

SOP:					
Author -	Michael Jacobs	**Number -**	330363	**Department -**	Volatiles
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Revision -	R17	**Rev. Date -**	3/12/12		

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Rev.	Date	Section	Brief Description
1	10/10/12	8.3.1.4	Clarification of section and inclusion of SC state note

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(see Revision History at the end of this document for more information)

TITLE: VOLATILE ORGANIC COMPOUNDS BY GC/MS (EPA 8260B, 8260C, 624 AND SM6200B 20TH)

SOP NUMBER: 330363

PREPARED BY: Michael Jacobs

REVIEWED BY: JD Gentry/Dixie Marlin

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_____
Department Manager

_____
QA Department

1.0 SCOPE AND APPLICATION

STATE NOTE: For samples analyzed in conjunction with the Ohio Voluntary Action Program (VAP) please utilize SOP# 330363, Revision 16 (1/30/09).

1.1 This standard operating procedure is used to determine volatile organic compounds in a variety of matrices. This SOP is designed for EPA methods 8260B, 624, Standard Method 6200B, GRO, or similar volatile GC/MS analyses. This procedure is applicable to nearly all kinds of samples, regardless of water content, including ground water, aqueous sludge, caustic liquors, acid liquors, waste solvents, oily wastes, mousses, tars, fibrous wastes, polymeric emulsions, filter cakes, spent carbons, spent catalysts, soils and sediments. The compounds that can be determined using this SOP are listed in Attachment III, which contains a list of the typical primary and secondary ions used in determining these compounds.

1.2 Reporting Limits (RLs) are listed in the Attachment II. Compounds routinely analyzed by this method and their typical reporting limits are included in the following table (subject to change).

1.3 An MDL study must be completed at least annually or more frequently if major instrumentation changes occur. Method Detection Limits (MDLs) are performed based on ESC SOP #030206. Updated MDL records are filed and stored in a central location within the department.

1.3.1 Limit of Detection (LOD) and Limit of Quantitation (LOQ) studies are completed at the frequency required by the TNI standard per the procedure identified in the ESC SOP #030206, *Method Detection Limits (MDL) and Limits of Detection (LOD).* Should the procedure be utilized for DOD support; then the frequency of these studies must meet the requirements of the current DOD QSM.

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TITLE: VOLATILE ORGANIC COMPOUNDS BY GC/MS (EPA 8260B, 8260C, 624 AND SM6200B 20TH)

2.0 METHOD SUMMARY AND DEFINITIONS

2.1 Volatile organic compounds (VOCs) are determined from a 5mL sample withdrawn from a sealed 40mL vial. For water samples analyzed for low levels of analytes using Method 5030 (SOP 330752), the entire vial is placed into the instrument autosampler. The autosampler purges 5mL of sample and adds 1µL of surrogate standards and internal standards. An inert gas is bubbled through a sparger needle inserted into the sample. The purged volatile components then travel via a transfer line to a sorbent trap. When purging is complete, the trap is rapidly heated. The trap is backflushed with a helium carrier gas, to transport the desorbed sample components into a gas chromatographic (GC) column. The GC column separates and carries the components to a mass spectrometer (MS) or a specific detector, depending on the determinative method selected.

2.2 For other samples, Method 5035 (SOP 330751), volatile organic compounds are determined from a 5g sample combined with 5mL reagent water.

2.3 Initial/Continuing Calibration Verification (ICV/CCV) – Analytical standard run at the beginning of every 12 hours shift to verify the stability of the established initial calibration of the analytical system.

2.4 Continuing Demonstration of Capability (CDOC) – At least, annual verification of analyst continued ability to perform method acceptably.

2.5 Duplicate – A second aliquot of a field sample analyzed using identical preparation and analytical procedures. Analysis of a sample duplicate monitors precision associated with laboratory procedures.

2.6 Initial Demonstration of Capability (IDOC) - A demonstration of capability (DOC) must be made prior to using any analytical method and any time there is a change in instrument type, personnel or testing method. Such performance must be documented and the four preparation batches following the change in personnel must not result in the failure of any batch acceptance criteria, e.g., method blank and laboratory control sample, or the demonstration of capability must be repeated. See also Continuing Demonstration of Capability (CDOC).

2.7 Laboratory Control Sample (LCS) / Laboratory Control Sample Duplicate (LCSD) – Duplicate aliquots of a control sample of known in composition. This sample is prepared from a source that is different from the stock used to prepare the initial and continuing calibration standards. LCS/LCSD are analyzed exactly like a sample and the purpose is to determine whether the methodology is in control and whether the laboratory is capable of making accurate and precise measurements. Method precision can be determined using the results of the LCS/LCSD analysis.

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2.8 Matrix Spike (MS) / Matrix Spike Duplicate (MSD) - Two aliquots of a field sample (water or soil) spiked with known quantities of specific compounds and subjected to the entire analytical procedure in order to indicate the appropriateness of the method for the matrix by measuring recovery. Method precision can be determined using the results of the MS/MSD analysis, but are subject to matrix variability issues not present in the LCS/LCSD pair.

2.9 Method Blank - An analytical control consisting of all reagents used in the analytical procedure. The method blank is used to define the level of laboratory background and reagent contamination.

2.10 Method Detection Limit - The minimum concentration of a substance that can be analyzed with 99% confidence that the analyte concentration is greater than zero.

2.11 Reporting Limit (RL) – Also see Practical Quantitation Limit (PQL). Routinely the reporting limit is the lowest standard of the calibration curve. Technically, the reporting limit is the lowest level that can be reliably achieved within the established limits of precision and accuracy during routine laboratory operating conditions.

2.12 Practical Quantitation Limit (PQL) – The default reporting limit when other limits are not specified by the client or project. The PQL is usually a factor of 3-10 times the detection limit.

2.13 Second Source Calibration Verification (SSCV) – A mid-point or low standard made from a secondary standard that is not used to construct the calibration curve. The SSCV is used to represent the calibration accuracy of the instrument and must perform within method stated criteria.

2.14 Surrogate - A compound which is similar to the target analytes in chemical composition and behavior and not expected to occur naturally in field samples that is spiked by preparation/analytical personnel to assess sample preparation and analytical efficiency in each individual field sample.

2.15 Poor Performer – An analyte having a lower mean recovery and higher standard deviation resulting in wider LCS control limits. These analytes can display erratic behavior in the analytical system.

2.16 Limit of Detection (LOD) - A laboratory's estimate of the minimum amount of an analyte in a given matrix that an analytical process can reliably detect in their facility. The validity of the LOD shall be verified by detection of the analyte(s) in a QC sample in each quality system matrix. This QC sample shall contain the analyte at no more than 3X the MDL for single analyte tests and 4X the MDL for multiple analyte tests. This verification shall be performed on every instrument that is to be used for analysis of samples and reporting of data. The QC samples used for this verification must be prepared and analyzed through all steps in the analytical process used for client samples.

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2.17 Limit(s) of Quantitation (LOQ) - The minimum levels, concentrations, or quantities of a target variable (e.g., target analyte) that can be reported with a specified degree of confidence. The validity of the LOQ shall be verified by successful analysis of a QC sample containing the analytes of concern in each quality system matrix at 1 to 2 times the claimed LOQ. A successful analysis is one where the recovery of each analyte is within the laboratory established method acceptance criteria or client data quality objectives for accuracy. The QC samples used for this verification must be prepared and analyzed through all steps in the analytical process used for client samples.

3.0 HEALTH AND SAFETY

3.1 The toxicity or carcinogenicity of each reagent used in the laboratory has not been fully established. Each chemical should be regarded as a potential health hazard and exposure to these compounds should be as low as reasonably achievable. Each laboratory is responsible for maintaining a current awareness file of OSHA regulations regarding the safe handling of the chemicals specified in this method. A reference file of material data handling sheets should also be made available to all personnel involved in the chemical analysis. Specifically, concentrated nitric and hydrochloric acids present various hazards and are moderately toxic and extremely irritating to skin and mucus membranes. Use these reagents in a fume hood whenever possible and if eye or skin contact occurs, flush with large volumes of water. Always wear safety glasses or a shield for eye protection, protective clothing and observe proper mixing when working with these reagents.

3.2 Glycol ethers are suspected carcinogens. All solvent handling should be performed in a hood while using proper protective equipment to minimize exposure to liquid and vapor. Minimum personal protection includes the use of laboratory safety glasses, a lab coat or apron, and protective gloves.

4.0 SAMPLE PRESERVATION, CONTAINERS, HANDLING, AND STORAGE

4.1 All samples must have been collected using a sampling plan that addresses the considerations of this method.

4.2 Volatile analysis for water and sodium bisulfate preserved soil samples must be completed within 14 days from the time of sample collection. Water samples that are not chemically preserved must be analyzed within 7 days. It is also an ESC requirement that water samples with 2-chloroethylvinyl ether (2-CEVE), as a compound of interest, be collected unpreserved and analyzed within 7 days. It has been shown that the acid preservative reacts with the 2-CEVE, which could result in false negative reporting of 2-CEVE in samples. Unpreserved soil samples must be analyzed within 48 hrs from the time of collection or otherwise frozen at $\leq$-7°C. High-level soil samples collected in Encore™ or equivalent type sampling devices must be placed in vials of methanol according to Method 5035 (SOP# 330751).

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STATE NOTE: The State of South Carolina and the Ohio VAP requires that all soil samples must be collected and analyzed using Method 5035. Samples must be preserved within 48 hours from the time of collection, if collected in Encore™ type sampling devices. The holding time for soil samples preserved with methanol or sodium bisulfate is 14 days from the time of collection. Non-Preserved South Carolina VOC's require 7-day Holding Time.

4.3 Aqueous samples must be collected in at least duplicate in 40mL vials with 0.008% $Na_2S_20_3$ per liter if residual chlorine is present. Sample kits can be configured to request additional vials per client request. The pH must be adjusted to <2 with HCl. Soil samples must be collected by one of the following: 1) A 4oz. soil jar filled with soil with zero headspace, 2) Two 5g samples preserved in the field with 5mL $NaHS0_4$ to a pH<2 and one 5g sample preserved in the field with methanol (for high level analysis) or 3) A 5g or 25g sample collected in an Encore or equivalent type sampling device and frozen in the laboratory within 48 hours from the time of collection.

For all soil samples, a 4oz. soil jar should also be collected to determine percent solids. All samples and extracts must be shipped and stored at <6°C.

STATE NOTE: Soil and Water samples received from the states of Missouri or Kansas may be preserved with tri-sodium phosphate and will have a resulting pH of 14.

STATE NOTE: For Ohio VAP samples, Encore samplers must be collected in the field and shipped to the laboratory to be frozen within 48 hours of collection. These samples will remain frozen until analysis and do not require additional chemical preservation. The holding time for these samples remains at 14 days from the time of collection. The holding time begins at collection and ends at 14 days.

STATE NOTE: For Alaska samples, when using a water miscible solvent (e.g. methanol) to extract soil volatile organic compounds (VOC), the adjustment of solvent volume for soil moisture content must be performed. Significant soil moisture can add to a pronounced dilution when performing methanol extractions. The potential under reporting of volatile concentrations is more pronounced as the percent moisture content increases. See section 9.9 for the calculation needed.

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5.0 INTERFERENCES

5.1 Major sources of contamination are volatile materials in the laboratory and impurities in the inert purging gas and in the sorbent trap. The use of non-polytetrafluoroethylene (PTFE) thread sealants, plastic tubing, or flow controllers with rubber components must be avoided since such materials out-gas organic compounds which will be concentrated in the trap during the purge operation.

5.2 Analyses of reagent blanks provide information about the presence of contaminants. When potential interfering peaks are noted in blanks, the analyst should change the purge gas source and regenerate the molecular sieve purge gas filter. Subtracting blank values from sample results is not permitted.

5.3 Interfering contamination may occur when a sample containing low concentrations of volatile organic compounds is analyzed immediately after a sample containing high concentrations of volatile organic compounds. After analysis of a sample containing high concentrations, one or more instrument blanks must be analyzed to check for cross contamination.

5.3.1 This interference may be prevented by rinsing the purging apparatus and sample syringes with portions of organic-free reagent water between samples.

5.3.2 For samples containing large amounts of water soluble materials, suspended solids, high boiling compounds or high concentrations of compounds being determined, it may be necessary to wash the purging device with a soap solution, rinse it with organic-free reagent water, and then dry the purging device in an oven at 105°C.

5.3.3 In extreme situations, the whole purge and trap device may require dismantling and cleaning.

5.3.4 Screening of the samples prior to purge and trap GC/MS analysis is highly recommended to prevent contamination of the system. This is especially true for soil and waste samples. Screening may be accomplished with an automated headspace technique. by SW-846 Method 3820, Hexadecane Extraction and Screening of Purgeable Organics), or screening of 5mL of sample using an HNU or equivalent portable PID.

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5.4 Special precautions must be taken to avoid contamination when analyzing for methylene chloride. The analytical and sample storage area must be isolated from all atmospheric sources of methylene chloride. Otherwise, random background levels will result. Since methylene chloride will permeate through PTFE tubing, all gas chromatography carrier gas lines and purge gas plumbing must be constructed from stainless steel or copper tubing. Laboratory clothing worn by the analyst must be clean since clothing previously exposed to methylene chloride fumes during liquid/liquid extraction procedures can contribute to sample contamination.

5.5 Samples can be contaminated by diffusion of volatile organics (particularly methylene chloride and fluorocarbons) through the septum seal into the sample during shipment and storage. A trip blank prepared from organic-free reagent water and carried through the sampling and handling protocol can serve as a check on such contamination. A storage blank must be analyzed weekly to check for cross contamination of samples while samples are stored in the volatiles laboratory walk-in cooler. The storage blank is prepared from organic-free water and is placed in the cooler for a period of one week. After a week, it is analyzed to verify that no contamination of client samples has taken place due to contamination in the storage unit. Analysis of a storage blank must be performed for both the volatiles laboratory walk-in coolers.

5.6 This procedure 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. Volatile water-soluble compounds can be included in this analytical technique. However, for the more soluble compounds, quantitation limits are approximately 50 times higher due to poor purging efficiency. Such compounds include low-molecular-weight halogenated hydrocarbons, aromatics, ketones, nitriles, acetates, acrylates, ethers, and sulfides.

5.7 Soil samples that contain carbonate minerals (either from natural sources or applied as an amendment) may effervesce upon contact with the acidic preservative solution in the low concentration sample vial. If a large amount of effervescent gas is generated, the sample may lose a significant amount of volatile analytes. If a sample effervesces, an unpreserved sample will be collected to eliminate volatiles loss whenever possible. The holding time for unpreserved VOC samples is seven days, rather than 14 days.

5.8 An analyst may re-analyze any sample if instrumentation or human error is suspected. This includes all QC samples, which can only be re-analyzed twice. If failure continues, instrument maintenance must be performed and/or the instrument must be re-calibrated.

5.9 Glassware must be scrupulously cleaned. All glassware must be cleaned per EPA protocol, as stated in SOP # 030701, *Glassware Cleaning*.

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TITLE: VOLATILE ORGANIC COMPOUNDS BY GC/MS (EPA 8260B, 8260C, 624 AND SM6200B 20TH)

6.0 EQUIPMENT AND SUPPLIES

The operation, cleaning, and scheduled maintenance procedures, as prescribed by the equipment manufacturer, are followed as provided in the Operator's Manuals. Documentation of maintenance or system modifications is recorded in a maintenance logbook which accompanies each analytical system.

6.1 Instrumentation: All instrumentation meets or exceeds EPA method requirements.

- Use (method #'s): 8260, 624
- Model #: HP 5890, or equivalent
- Column (type, brand, size): J & W Scientific DB VRX 75 m x 0.450 mm, 2.55 micron, or equivalent.
- Detector: MS 5972 or better.
- Software name and version: HP Chemstation G1701BA B.01.00, or equivalent
  Sample introduction system: Archon Autosampler, Encon P & T, or equivalent

6.2 Glassware must be scrupulously cleaned. All glassware must be cleaned per EPA protocol, as stated in SOP # 030701, *Glassware Cleaning*. Rinsing with methanol and laboratory reagent water cleans the volumetric flasks and graduated cylinders. The volumetric flasks are dried in a low temperature oven at less than 120°C and never cleaned with a brush or strong alkali solution.

6.3 The carrier gas used for volatiles analysis is Helium-5.0 grade.

6.4 Syringes used for preparing the calibration curve and preparing samples and sample dilutions are Hamilton brand (or equivalent). Syringe sizes used are 0.50µL, 10µL, 25µL, 50µL, 100µL, 250µL, 1mL, and 5mL.

6.5 Glass Sample (VOA) and Standard Vials:

6.5.1 40mL VOA vials with a Teflon™/silicone septa and polypropylene open-top cap.

6.5.2 8mL vials with Teflon™/silicone/Teflon™ septa and polypropylene open-top cap. (Used to store unused standards)

6.6 Miscellaneous:

6.6.1 Stainless Steel Spatula or wooden tongue depressor.

6.6.2 Disposable aluminum drying dishes - VWR #25433-008, or equivalent

6.6.3 Teflon™-coated stir bars, 8mm x 16mm

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6.6.4 Laboratory Sand: Sand is prepared by rinsing clean, white sand with methanol and laboratory reagent water several times. The rinsed sand is baked in an oven at 175°C overnight to remove any volatiles and is then stored in the same oven. The heated laboratory sand is occasionally purged with carrier grade helium or nitrogen to remove trapped volatiles.

6.7 Oven: Fisher IsoTemp Forced-Air Oven with capabilities of 100°C, or equivalent

6.8 Top-loading Balance, capable of weighing to 0.01g, or equivalent.

7.0 REAGENTS AND STANDARDS

7.1 All reagents and standards must be recorded in the appropriate preparation log and assigned a unique number. See SOP 030203, *Reagent Logs and Records*, and SOP 030230, *Standard Logger*. Additional information regarding reagent preparation can be found in the Standards Logger (Tree) digital archive system. All spiking solutions and surrogate standard solutions should be replaced at least every 6 months, or sooner if a problem is detected unless otherwise noted.

7.2 Laboratory water created by reverse osmosis/DI filtration evaluated to .055uS/cm to ensure purity. Laboratory water is used in all blanks to assure that it contains less than the minimum detection limit (MDL) of all compounds of interest. The blank must be assessed to ensure that the water does not show any detection of any VOC compounds. If volatile compounds are detected in the blank above the MDL all samples associated with this blank must be flagged.

7.3 Methanol, CH_3OH – purge-and-trap grade, demonstrated to be free of analytes. Store apart from other solvents.

7.4 Sodium Bisulfate, $Na_2S_2O_3$ - QEC Level 3 Certified, or equivalent

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7.5 STOCK SOLUTIONS – Calibration and LCS

- Stock calibration solutions must be purchased as certified solutions.
- Certificates must be kept on file.
- All Stock standards must be stored below -10°C
- All non-gas stock standards must be replaced after six months, or sooner, if check standards indicate a problem.
- Both gas and liquid standards must be monitored closely by comparison to the initial calibration curve and by comparison to a second source ICV.
- Gas intermediate/secondary standards must be replaced weekly, or sooner, if comparison to check standards indicates a problem.
- Non-gas intermediate/secondary standards must be replaced after six months, or sooner, if comparison to check standards indicates a problem.

7.5.1 CALIBRATION - Primary source standards is used to prepare the initial 5-point calibration curve (additional levels may be used as needed), the initial calibration verification (ICV), and continuing calibration verification (CCV) standard. The ICV and CCV are analyzed to verify the initial calibration and are prepared using the primary source standards used to produce the calibration curve. See Section 8.2.6 through 8.2.8 for the instrument preparation of the calibration standards. When primary standards are consumed, new standards must meet the same QC criteria as the consumed standards. Stock standards must be stored below -10° C and have a six-month holding time once opened. The expiration date of the diluted standards must not exceed the expiration date of the stock standards from which they are prepared. Once diluted, the standard must be replaced weekly.

ICV Mix

The ICV solution is prepared in methanol in a 50mL volumetric flask by adding:

Manufacturer	Product	Cat. #	Amount added (mL)	Final Conc. (ppm)
NSI	8260 Custom Mix 1	Q-4146	5	50
NSI	8260 Custom Mix 2	Q-4147	5	250
Absolute	n-Hexane	70962	2.5	50
Ultra	1+2 Methylnaphthalenes	Q-4260A	2.5	50
SPEX	Acrolein	Q-3835	2.5	250
SPEX	AZ fuel additive 4 comps.			
Ultra	8260 Gases Mix	CUS-5661	25	50

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GRO ICV Mix

The GRO ICV solution is prepared in methanol in a 10mL volumetric flask by adding:

Manufacturer	Product	Cat. #	Amount added (mL)	Final Conc. (ppm)
Restek	TX TPH matrix spike mix	Cat 31484	1	1000

AP9/Oxygenates ICV Mix

The GRO ICV solution is prepared in methanol in a 10mL volumetric flask by adding:

Manufacturer	Product	Cat. #	Amount added (mL)	Final Conc. (ppm)
NSI	Custom AP9 Standard	Q-4973	3.125	5

7.5.1.1 For soil/water autosamplers (5mL), a dilution of 10X is required for the ICV mix. The solution is stored in 3mL aliquots in zero headspace vials. The storage temperature is below -10° C.

7.5.2 LABORATORY CONTROL STANDARDS - The standard list of target LCS compounds are those compounds listed in Attachment VIII. Secondary source standards must be used to prepare the laboratory control standard (LCS) and the matrix spike and their duplicates. These standards are purchased from a different vendor or the primary vendor can supply different lot numbers, if a separate vendor is not available. The standard is at a concentration near the mid-level calibration standard. Stock standards must be stored at or below -10°C and have a six month holding time once opened, except the ICV gases which have 1 week holding time. Once diluted, the standard must be replaced weekly.

LCS & Matrix Spikes

Prepare the LCS/LCSD in methanol in a 50mL volumetric flask as follows. A separate source or separate lot number is used for standard verification. The standard list of target LCS compounds are those compounds listed in Attachment VIII.

Manufacturer	Product	Cat. #	Amount added (mL)	Final Conc. (ppm)
Restek	8260 Custom Mix 1	Q-4146	5	50
NSI	8260 Custom Mix 2	Q-4147	5	250
Absolute	n-Hexane	70962	2.5	50
Ultra	1+2 Methylnaphthalenes	Q-4260A	2.5	50
SPEX	Acrolein	Q-3835	2.5	250
Ultra	8260 Gases Mix	CUS-5661	25	50

GRO LCS

The GRO LCS is prepared in methanol in a 10mL volumetric flask by adding:

Manufacturer	Product	Cat. #	Amount added (mL)	Final Conc. (ppm)
Ultra	GRO-MO Mix	Q-4208	1	1000

AP9/Oxygenates LCS

The AP9/Oxygenates LCS is prepared in methanol in a 25mL volumetric flask by adding:

Manufacturer	Product	Cat. #	Amount added (mL)	Final Conc. (ppm)
Ultra	Custom AP9 Standard	CUS-9324	3.125	5

The LCS/LCSD must be prepared in the appropriate matrix (organic-free reagent water, or purified solid) depending upon the matrix within the analytical batch; and contain all of the method target analytes. A subset of the method target analytes could be used based on the project specific requirements.

7.5.2.1 For soil/water autosamplers (5mL), a dilution of 10X is required. The solution is stored in 3mL aliquots in zero headspace vials. The storage temperature is at or below -10° C.

STATE NOTE: South Carolina and the USACE require that all target analytes are present and evaluated in the LCS.

7.6 Surrogate standard stock solutions must be purchased as certified solutions. Certificates must be kept on file. Stock standards must be stored at or below -10° C and have a six month holding time, once opened. Surrogate spiking solutions are purchased from NSI as custom standard Q-4185, or equivalent, at 20,000ug/mL, which contains both internal standards and surrogate compounds. This solution is then diluted by 100X to obtain a 200ug/mL working solution.

7.6.1 The following are ESC designated volatiles' analysis surrogates:

- Toluene-d8
- 4-Bromofluorobenzene
- Dibromofluoromethane
- ααα-Trifluorotoluene

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7.7 Internal standard stock solutions must be purchased as certified solutions. Certificates must be kept on file. Stock standards must be stored at or below -10° C and have a six month holding time, once opened. [Internal standard and surrogate standard – NSI custom MS/IS/SS mix Q-4185, or equivalent]

7.7.1 The following are ESC designated volatiles' analysis internal standards:

- 1,4-Difluorobenzene
- 2-Bromo-1chloropropane
- 1,4-Dichlorobenzene-d4
- Pentafluorobenzene

7.8 4-Bromofluorobenzene (BFB) standard - The BFB in the custom internal standard mix is used to verify mass spectrometer tuning. Since internal standards and surrogates are added to all samples and standards, BFB is included as part of our initial calibration and calibration verification standards. Certificates of analysis must be kept on file. Stock standards must be stored at or below -10° C and have a six month holding time, once opened.

7.9 Matrix spike (MS) standard - Stock standards must be stored at or below -10°C and have a six month holding time, once opened. Once diluted, the standard must be replaced weekly.

7.9.1 The matrix spike standard is prepared from the stock standard in Section 7.5.

7.9.2 The spike should be at a mid-level of the calibration range. Some contracts may require a site-specific concentration.

7.9.3 Standard spiking practice requires the use of ALL TARGET ANALYTES as specified in Attachment VIII and must be evaluated against the current control limits.

Project Specific Requirements (Non-South Carolina Projects): Individual projects may specify required spike compounds. In addition to any project specific requirements, the following table lists the minimum required compounds that must be included in the spike solution.

Minimum Spiking Compounds for Project Specific Requirements
• 1,1-Dichloroethene
• Trichloroethene
• Chlorobenzene
• Toluene
• Benzene
• n-Hexane (when requested as a target analyte)

All compounds in the spike solution must be evaluated for acceptable recovery. In the absence of established control limits, default recovery limits are 70 - 130%.

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7.9.4 **STATE NOTE:** OHIO VAP SPIKES: Matrix spikes are evaluated using in house limits for only those compounds listed in 7.9.3.

8.0 PROCEDURE

Analysis Summary**:** Volatile compounds are introduced into the gas chromatograph by purge and trap, via the Archon autosampler, as described on Section 2. If soil samples are high in contamination, a methanolic extraction, SOP No.330760, may be necessary prior to purge and trap analysis. Soils require method 5035 for sample preparation, See SOP 330751.

8.1 Chromatographic conditions: All changes in analytical conditions are listed in the Maintenance Log.

8.1.1 Typical conditions for each instrument and column are listed below:

Inlet	off
Detector	200°C
Oven Equib. Time:	0.50 minutes
Oven Max	240°C
Init Temp	45°C hold 1.0 minute
Ramp	20°C/min to 240 hold 1.0 minute

8.1.2 Typical conditions for each autosampler are listed below:

Heating sample	1 minute at 40°C
Purge	11 minutes at 40°C
Desorb	1 minutes at 250°C
Bake	2 minutes at 260°C

8.1.3 Typical condition for each MS detector are listed below:

Electron energy – 70 volts (nominal)
Mass range – 35 to 300 amu
Scan time – 1.2 sec/scan
Manifold vacuum – 3 x 10^{-6} torr

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8.2 **Initial calibration**

8.2.1 TUNING - Each GC/MS system must be hardware-tuned (1µL = 50ng) with BFB to meet the criteria listed below. The mass-spectrometer must meet acceptable BFB sensitivity criteria before analysis can begin. The instrument must be tuned every 12 hours for 8260B, 8260C, and 6200B. BFB tuning for method 624 is every 24 hours.

BFB Key Ions and Ion Abundance Criteria	
Mass	Ion Abundance Criteria
50	15.0-40.0% of mass 95
75	30.0-60.0% of mass 95
95	base peak, 100% relative abundance
96	5.0-9.0% of mass 95
173	< 2.0% of mass 174
174	> 50.0% of mass 95
175	5.0-9.0% of mass 174
176	> 95.0%, but less than 101% of mass 174
177	5.0-9.0% of mass 176

8.2.2 Calibration Curve – General Criteria

- A minimum of 5-point calibration is performed using the primary standards listed in Section 7.5.1. Additional levels may be included to better meet project or client requirements. Regardless of the specific number, the calibration levels analyzed should correspond to a range of concentrations expected to be found in samples, without exceeding the working range of the GC/MS system.
- A calibration point must be analyzed at or below the reporting limit. The concentration of the lowest calibration standard analyzed should be at least 3-5 times the MDL. The instrument response must be distinguishable from the instrument background noise. The signal to noise ratio is the magnitude of the signal strength detected by the mass spectrometer relative to the magnitude of the background noise of the instrument. Instrument conditions must be optimized before the analysis of a calibration curve to minimize background effects.

STATE NOTE: A reporting level standard must be run after calibration is complete. This standard is required by the state of North Carolina and is used to verify the low end of the calibration curve.

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STATE NOTE: When analyzing samples from MN, the reporting limit must be verified with each calibration or at least monthly. Verification can be performed by re-quantitation of the low calibration standards using the newly updated calibration curve or by analyzing a separate reporting level standard following calibration curve update. This standard must recover ±40% of the expected concentration. If the criteria is not met, a higher level standard may be re-quantitated or analyzed; however the reporting limit must be amended to reflect the increased concentration of the standard utilized. Analytes known to be poor performers are dealt with on a case-by-case basis.

- The highest standard must not exceed the linear range of the detector. The concentration of the highest standard must produce a response, which does not cause the MS detector to become saturated. The highest concentration used in the calibration curve must allow the analyte to meet the calibration requirements outlined in Sections 8.2.6 through 8.2.8.
- When using Method 5035, SOP #330751, the calibration curve must be prepared in the same solutions used to preserve the field samples.
- **EPA 8260C NOTE:** The method of linear regression analysis has the potential for a significant bias to the lower portion of a calibration curve, while the relative percent difference and quadratic methods of calibration do not have this potential bias. When calculating the calibration curves using the linear regression 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 calibration standard back into the curve. It is not necessary to reanalyze a low concentration standard, rather the data system can recalculate the concentrations as if it were an unknown sample. The recalculated concentration of the low calibration point should be within ±30% of the standard's true concentration.
- The method reference spectra must be updated from the mid-point of each calibration.

STATE NOTE: For Ohio VAP samples, when soil samples are received pre-preserved with sodium bisulfate, calibration standards must also include sodium bisulfate at the same concentration. It is preferred that samples are not chemically preserved in the field and are instead capped and shipped to the laboratory to be frozen with 48 hours of sampling.

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8.2.3 Calibration Levels for single analytes

8.2.3.1 Soil Samples - Soil samples are analyzed with a heated purge in the soil chamber of the Archon, or equivalent autosampler. The calibration curve is generated by injecting the following volumes of ICV Mix (See Section 7.5.1) into 5mL of water containing 1g sodium bisulfate.

Calibration Curve - GC/MS Soil (into 5mL water)	
Intermediate solution volume (µL)	Concentration of standard (ppb)
0.05	0.5
0.1	1
0.2	2
0.5	5
1	10
2.5	25
5.0	50
10.	100
20.	200

AP9/0xygenates Calibration Curve - GCIMS Soil (into 5mL of water)	
Intermediate solution volume (µL)	Concentration of standard (ppb)
1	1
2.5	2.5
5	5
7.5	7.5
10	10
12.5	12.5
15	15
17.5	17.5
20.	20

Note 1: When analyzing soil samples by the low-concentration method (Section 8.7.1), the calibration standards must be heated to 40°C ± 1°C prior to purging.

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Note 2: Injections should be performed from the lowest to the highest standards with a cleanup injected after the highest standard and followed by the secondary source standard to verify the initial calibration curve.

8.2.3.2 Water Samples - Water samples are run with a heated purge using the Archon, or equivalent autosampler. The calibration curve is generated by injecting the following volumes of ICV Mix (See Section 7.5.1.) into 5mL of water.

Calibration Curve - GC/MS Water (into 5mL water)	
Intermediate solution volume (µL)	Concentration of standard (ppb)
0.05	0.5
0.1	1
0.2	2
0.5	5
1.0	10
2.5	25
5.0	50
10.	100
20.	200

AP9/Oxygenates Calibration Curve – GC/MS water (into 5mL of water)	
Intermediate solution volume (µL)	Concentration of standard (ppb)
1	1
2.5	2.5
5	5
7.5	7.5
10	10
12.5	12.5
15	15
17.5	17.5
20.	20

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8.2.3.3 The Centurion autosampler, or equivalent, transfers 5mL of sample to the concentrator for purging and requires full VOA vials. The sampler requires 50mL of standard prepared and analyzed using the ICV Mix in Section 7.5.1.

Calibration Curve - GC/MS Water (into 50mL water)	
Intermediate solution volume (µL)	Concentration of standard (ppb)
0.5	0.5
1	1
2	2
5	5
10	10
25	25
50	50
100	100
200	200

AP9/Oxygenates Calibration Curve – GC/MS Water (into 50mL water)	
Intermediate solution volume (µL)	Concentration of standard (ppb)
10	1
25	2.5
50	5
75	7.5
100	10
125	12.5
150	15
175	17.5
200	20

8.2.4 Calibration Levels for GRO by MS

8.2.4.1 Soil Samples - Soil samples are analyzed with a heated purge in the soil chamber of the Archon, or equivalent autosampler. The calibration curve is generated by injecting the following volumes of ICV Mix (See Section 7.5.1.) into 5mL of water containing 1g sodium bisulfate.

Calibration Curve - GC/MS Soil (into 5mL water)	
Intermediate solution volume (µL)	Concentration of standard (ppm)
2	0.4
5	1
10	2
20	4
25	5
35	7
50	10
100	20

8.2.5 Internal Standards and Surrogates – Soil/Water

The autosampler adds 1µL of the IS/surrogate mix to each sample. The addition of 1µL of the surrogate spiking/internal standard solution to 5mL of sample is equivalent of 40µg/L of each surrogate standard. Internal standard and surrogate standard are contained within the same spiking mix. Internal Standards are listed Section 7.7.1 and Surrogates are listed in Section 7.6.1.

Tabulation of the Internal Standards

Tabulate the area response of the characteristic ions (see Attachment III) against each compound's concentration and each internal standard concentration. Then calculate the response factor (RF) for the quantifying ion of each compound relative to the appropriate internal standard according to the calculation provided in Section 9.1. The internal standards used should permit most of the compounds of interest in a chromatogram to have retention times of 0.80 to 1.20, relative to one of the internal standards. The average RF must be calculated and recorded for each compound.

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8.2.6 System Performance Check Compounds (SPCCs) – Soil/Water

A system performance check must be made before the calibration curve can be used. The minimum relative response factor for volatile SPCCs are as follows:

Chloromethane	0.10
1,1-Dichlorethane	0.10
Bromoform	0.10
Chlorobenzene	0.30
1,1,2,2-Tetrachloroethane	0.30

These compounds are typically used to check compound instability and to check for degradation caused by contaminated lines or active sites in the system. Examples of these occurrences are:

Compound	**Effect on stability**
Chloromethane	This compound is the most likely compound to be lost if the purge flow is too fast.
Bromoform	This compound is one of the compounds most likely to be purged very poorly if the purge flow is too slow. Cold spots and/or active sites in the transfer lines may adversely affect response. Response of the quantitation ion (m/z 173) is directly affected by the tuning of BFB at ions m/z 174/176. Increasing the m/z 174/176 ratio relative to m/z 95 may improve bromoform response.
1,1,2,2-Tetrachlorethane and 1,1-Dichloroethane	Contaminated transfer lines degrade these compounds in purge-and-trap systems. Active sites in trapping materials also can cause problems.

Adjust the purge gas (helium) flow rate to 25-40mL/min on the purge-and-trap device. Optimize the flow rate to provide the best response for chloromethane and bromoform. Excessive flow rate reduces chloromethane response, whereas insufficient flow reduces bromoform response.

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8.2.7 **EPA 8260B:** Response Factors (RF's) & Calibration Check Compounds (CCC's) - Soil/Water

Using the RF's for the initial calibration curve from Section 8.2.2, calculate and record the percent relative standard deviation (%RSD) for all compounds. Calculate the percent RSD as in Section 9.2. Linearity can be assumed if the RSD criteria is met, thus allowing quantitation calculations to be performed using RF.

8.2.7.1 CCC Criteria - The %RSD for each individual CCC must be less than 30%. The CCCs are:

1,1-Dichloroethene	Toluene
Chloroform	Ethylbenzene
1,2-Dichloropropane	Vinyl chloride

8.2.7.2 Target Analytes and other Non-CCC's - The RSD must meet the following criteria -

<15% RSD for all 8260B Target Analytes
<35% RSD for all 624 Target Analytes
<20% RSD for all KSGRO Samples
<15% RSD for n-Hexane
<15% RSD for 6200 Analytes
<15% appendix 9 Analytes
<10% RSD for all 601/602 Target Analytes

Compounds not meeting the RSD requirement may be considered for linear regression as stated in 8.2.7.3

8.2.7.3 Linear Regression - Criteria

When any compound does not meet the calibration criteria for RF, the analyst must use linear regression. If linear regression is used, it must be noted on the data (preferably on the CCV RF report), next to the affected compound. It must also meet correlation coefficient criteria of 0.99 or better. USACE requires correlation coefficient criteria of 0.995 or better.

Linear regression is achieved by plotting the instrument response versus the concentration of the standards. The resulting regression line must not be forced through the origin and the origin must not be included as a calibration point.

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Linear regression may not be used for compounds that exceed 40% RSD except for the poor performers listed in section 8.3.4. Compound calibration with a %RSD >40 is considered out of control and must be re-calibrated or not used. The %RSD for the poor performers cannot exceed 50%.

For linear regression calculations, see section 9.11.

8.2.7.4 Calibration Corrective Action

When the RSD exceeds 15% or linear regression criteria could not be met, plot and inspect the calibration data for abnormal chromatographic responses. The inspection may indicate analytical problems, including errors in standard preparation, the presence of active sites in the chromatographic system, analytes that exhibit poor chromatographic behavior, etc.

If calibration criteria are not met, then one of the following options must be applied to the GC/MS initial calibration:

8.2.7.4.1 Adjust the instrument and/or perform instrument maintenance and re-analyze the calibration standards until the RSD of the calibration meets criteria.

8.2.7.4.2 Narrow the calibration range until the response is linear. If the low standard is below the estimated quantitation limit (i.e., for the poor purgers in a commercially available prepared standard mix), then this standard may be dropped. Re-calculate the RSD without the low standard to determine if the RSD meets the QC limit. If the lowest standard is dropped, the reporting limit could require a change. Check with the supervisor to determine if a point can be removed and not affect reporting limits requirements.

Compounds that are very soluble in water generally are poor purgers. The ketones, vinyl acetate, acrolein, and acrylonitrilie fall into this category.

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8.2.8 **EPA 8260C:** Response Factors (RF's)

8.2.8.1 Calibration Curve Criteria - Calculate the mean response factor and the relative standard deviation (RSD) of the response factors for each target analyte using the following equations. The RSD should be less than or equal to 20% for each target analyte. It is also recommended that a minimum response factor for the most common target analytes as noted in Attachment VII, be demonstrated for each individual calibration level as a means to ensure that these compounds are behaving as expected. In addition, meeting the minimum response factor criteria for the lowest calibration standard is critical in establishing and demonstrating the desired sensitivity. Due to the large number of compounds that may be analyzed by this method, some compounds will fail to meet this criteria. For these occasions, it is acknowledged that the failing compounds may not be critical to the specific project and therefore they may be used as qualified data or estimated values for screening purposes.

8.2.8.2 When the RSD exceeds 20% or linear regression criteria could not be met, plot and inspect the calibration data for abnormal chromatographic responses. The inspection may indicate analytical problems, including errors in standard preparation, the presence of active sites in the chromatographic system, analytes that exhibit poor chromatographic behavior, etc.

8.3 Calibration Verification

8.3.1 Calibration Verification for EPA Methods 8260B, 624 and SM 6200B:

8.3.1.1 SSCV's

After a successful calibration, a Second Source Calibration Verification (SSCV) must be analyzed to verify the calibration. This standard must be made from a second source, preferable from a different vendor than the calibration standards. The second source calibration standard must perform within following criteria:

CCC and SPCC compounds	± 30%
Other compounds (non-poor performers)	± 40%
Poor Performers (8.3.2)	in-house LCS limits

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8.3.1.2 CCC's

The curve must be verified initially by a calibration standard known as the Initial Calibration Verification standard (ICV). In addition, the same standard is analyzed every 12 hours for continuing calibration verification (CCV). This standard is prepared at or near the mid-point of the calibration curve. A maximum of 20% criteria would be expected for CCC analytes (Listed in Section 8.2.7.1) and n-Hexane when requested as a target analyte.

Compounds on average response factor use % difference,

$$\% \text{ Difference} = (RF_v - Rf_{ave}) / Rf_{ave} \times 100\%$$

Compounds on regression fit model use percent drift,

% Drift = (Calculated conc - Theoretic conc) / Theoretic conc x 100%

Criteria for both is $\leq$20%.

8.3.1.3 SPCC's

The SPCC's must have a minimum response factor as stated in Section 8.2.6. If these criteria are exceeded, then corrective action is required.

8.3.1.4 All Target Analytes and Non-CCC's

When analyzing 8260B0 and 624 concurrently, target analytes and non-CCC compounds must meet the criteria established in 624 (Attachment VI) for all analytes on the 624 list. For analytes not on the 624 list, all target analytes (except for the poor performers, as listed below) and non-CCC's must meet a maximum of 40% drift from the calibration curve. The analyst evaluates all analytes carefully and the experience of the analyst weighs heavily when determining the usability of the data.

Poor performers are allowed a maximum of 50% drift from the calibration curve. See section 8.3.3 for a listing of poor performing analytes.

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8.3.2 Calibration Verification for EPA Method 8260C:

8.3.2.1 SSCV's

After a successful calibration, a Second Source Calibration Verification (SSCV) must be analyzed to verify the calibration. This standard must be made from a second source, preferable from a different vendor than the calibration standards. The second source calibration standard must perform within following criteria:

All compounds	± 30%
Poor Performers (8.3.3)	in-house LCS limits

8.3.2.2 Target Analytes

The curve must be verified initially by a calibration standard known as the Initial Calibration Verification standard (ICV). In addition, the same standard is analyzed every 12 hours for continuing calibration verification (CCV). This standard is prepared at or near the mid-point of the calibration curve. A maximum of 20% criteria would be expected for all target analytes and n-Hexane when requested as a target analyte.

If the percent difference or percent drift for a compound is less than or equal to 20%, then the initial calibration for that compound is assumed to be valid. Due to the large numbers of compounds that may be analyzed by this method, some compounds will fail to meet the criteria. If the criterion is not met (i.e., greater than 20% difference or drift) for more than 20% of the compounds included in the initial calibration, then corrective action must be taken prior to the analysis of samples. In cases where compounds fail, they may still be reported as non-detects if it can be demonstrated that there was adequate sensitivity to detect the compound at the applicable quantitation limit. For situations when the failed compound is present, the concentrations must be reported as estimated values.

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8.3.3 Poor Performers:

The poor performers are as follows:

Poor Performing Compounds

Propene	2-Chloroethylvinyl Ether
Dichlorodifluoromethane	Acrolein
Carbon Disulfide	Vinyl acetate
Bromomethane	trans-1, 4-dichloro-2-butene
Chloroethane	Alcohols (Ethanol, TBA, TAA, ETBA, TBF, Butanol)
1,3-Butadiene	Iodomethane.
2,2-Dibromo-3-chloropropane	Naphthalene
1- Methylnaphthalene	2-Butanone
2- Methylnaphthalene	2-Hexanone
Acetone	4-Methyl-2-pentanone
Pentachloroethane	Cyclohexanone

8.3.4 Laboratory Control Standard (LCS): A laboratory control sample (LCS) should be included with each analytical batch. The LCS consists of an aliquot of a clean (control) matrix similar to the sample matrix and of the same weight or volume. The LCS is spiked with the same analytes at the same concentrations as the matrix spike, when appropriate. When the results of the matrix spike analysis indicate a potential problem due to the sample matrix itself, the LCS results are used to verify that the laboratory can perform the analysis in a clean matrix. Also note the LCS for water sample matrices is typically prepared in organic-free reagent water similar to the continuing calibration verification standard. In these situations, a single analysis can be used for both the LCS and continuing calibration verification.

- QC Limits are in Attachment VIII, LCS and MS/MSD Criteria.
- If the stated criteria are exceeded, then corrective action is required.
- See Section 11.6.1, on marginal excedences.

STATE NOTE: All **South Carolina** LCS responses must be within 70 – 130%. NO FAILURES are acceptable, qualifiers cannot be used. FAILURES require a batch re-analysis. See Section 10 for QC evaluation.

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8.3.5 Internal Standard Evaluation

8.3.5.1 When a calibration is performed at the beginning of an analytical run: The internal standard areas must be evaluated against the mid-point of the curve. Samples are analyzed within a 12-hour window; the internal standards of those samples are evaluated against mid-point of the curve. Then a CCV is analyzed, this is compared to the mid-point of the initial calibration curve. Addition samples are analyzed within a 12-hour window; the internal standards of those samples are evaluated against the previous acceptable CCV.

8.3.5.2 When an analytical run is started using a passing ICV (which is compared against the initial calibration mid-point to verify the calibration curve): Samples are analyzed within a 12-hour window, the internal standards of those samples are evaluated against the daily ICV. Then a CCV is analyzed, this is compared to the mid-point of the curve. Additional samples are analyzed within a 12-hour window; the internal standards of those samples are evaluated against the previous acceptable CCV.

8.4 Gas chromatographic analysis:

8.4.1 Typical sequence order for loading the autosampler with calibration:

Sample/QC Type	Use
Cleanup Blank	Verify system is contamination free
BFB Tune	Tuning criteria
Calibration standard(s)	Initial volatiles calibration and 7-point for GRO (if analyzed)
Second Source Calibration Verification (SSCV)	Verify initial calibration with second source.
Laboratory Control Sample/ Laboratory Control Sample Duplicate	Laboratory blank, spiked with known amount(s) of analyte of interest
Matrix Spike/Matrix Spike Dup.	Sample spiked with known amount(s) of analytes of interest
Method blank	Ensure that carry over has not occurred from the calibration standard and that the analytical system does not show contamination above the established detection limits
12-hour window	Client samples
Continuing Calibration Verification (CCV)	Single-point calibration verification standard, if needed.
12-hour window	Client samples

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8.4.2 Typical sequence order for loading the autosampler with no calibration:

Sample/QC Type	Use
Cleanup Blank	Verify system is contamination free
BFB Tune	Tuning criteria
Initial Calibration Verification (ICV)	Verify initial calibration.
Laboratory Control Sample/ Laboratory Control Sample Duplicate	Laboratory blank, spiked with known amount(s) of analyte of interest
Matrix Spike/Matrix Spike Dup.	Sample spiked with known amount(s) of analytes of interest
Method blank	Ensure that carry over has not occurred from the calibration standard and that the analytical system does not show contamination above the established detection limits
12-hour window	Client samples
Continuing Calibration Verification (CCV)	Single-point calibration verification standard, if needed.
12-hour window	Client samples

8.5 GC/MS Analysis -- Water Samples

8.5.1 Screening the sample prior to purge-and-trap analysis provides guidance on whether sample dilution is necessary and prevents contamination of the purge-and-trap system. Three screening techniques that can be used are the headspace sampler, using a gas chromatograph (GC) equipped with a photo ionization detector (PID) in series with an electrolytic conductivity detector (ELCD) (SW-846 Method 3810), extraction of the sample with hexadecane and analysis of the extract on a GC with a FID and/or an ECD (SW-846 Method 3820), and screening of 5mLs of sample using an HNU or equivalent portable PID.

8.5.2 All samples and standard solutions must be allowed to warm to ambient temperature before analysis.

8.5.3 Set up the GC/MS system as outlined in Section 8.1.

8.5.4 BFB tuning criteria and GC/MS calibration criteria must be met (Section 8.2.1) before analyzing samples.

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8.5.5 Rinse a 5mL syringe by filling and discarding 3 times with distilled water. Open the sample and quickly remove a 5mL aliquot. Immediately replace the sample cap and deposit the aliquot into a clean/baked 40mL autosampler vial. Cap quickly and tightly.

All samples analyzed for USACE must be run using the autosampler prep technique. VOA vials for USACE samples must not be opened prior to analysis to minimize target analyte loss.

After the sample has been loaded on the instrument, check the pH of the sample using the remaining sample in the VOA vial. Use universal pH paper and record the sample pH to the nearest whole pH unit. Samples not passing the pH requirements are flagged with a "T2" qualifier.

8.5.6 Sample Dilution -- When necessary, samples can be diluted before purging. This can be performed in a clean/baked 40mL vial that has been filled with 5mL of measured reagent water. The sample is measured through the use of an appropriate microliter syringes.

8.5.7 Compositing samples prior to GC/MS analysis – Site or project-specific requirements may require compositing of samples, which is performed according to the instructions below. Compositing of samples is only performed at the request of the client.

8.5.7.1 Add 5mL of each sample (up to 5 samples are allowed) to a 25mL glass syringe. Practice special precautions to maintain zero headspace in the syringe. Larger volumes of a smaller number of samples may be composited, as long as equal volumes of each sample are used.

8.5.7.2 The samples must be cooled to 4°C or less during composition to minimize the loss of volatiles. Sample vials may be placed in a tray of ice to prevent volatile loss during this process.

8.5.7.3 Mix each vial well. Using the 25mL syringe, draw out a 5mL aliquot of sample.

8.5.7.4 Once all the aliquots have been combined in the syringe, invert the syringe several times to mix the aliquots. The sample is now ready to be analyzed.

8.5.7.5 If less than five samples are being used for compositing, a smaller syringe may be used, provided ample volume is obtained for analysis.

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NOTE: Samples are not routinely composited, however, if site-specific requirements state procedures for compositing samples, the laboratory makes every effort to comply with those requirements.

8.5.8 Surrogate/Internal Standards – The autosampler adds 1uL of the IS/surrogate mix to each sample. The addition of 1µL of the surrogate spiking/internal standard solution to 5mL of sample is equivalent of 40µg/L of each surrogate standard. Internal standard and surrogate standard are contained within the same spiking mix.

8.5.9 Refer to SOP 330752 EPA 5030B Purge and Trap for Aqueous Samples and SOP 330751 EPA 5035 for additional information on sample introduction.

8.5.10 If the initial analysis of a sample or a dilution of the sample has a concentration of analytes that exceeds the initial calibration range, the sample must be re-analyzed at a higher dilution. All dilutions must keep the response of the major constituents (previously saturated peaks) in the upper half of the linear range of the curve. Secondary ion quantitation is allowed only when there are sample interferences with the routinely quantitated primary ion. When a sample is analyzed that has saturated the detector, the samples following must be analyzed for contamination. If any sample shows contamination, they must be re-analyzed.

8.6 GC/MS Analysis -- Water-miscible liquids

8.6.1 Water-miscible liquids are analyzed as water samples after first diluting them at least 50-fold with laboratory water.

8.6.2 Initial and serial dilutions can be prepared by pipetting a known amount of the sample to a 50mL volumetric flask and diluting to volume with organic-free reagent water. Transfer immediately to a clean/baked 40mL vial using a 5mL syringe.

8.6.3 Alternatively, prepare dilutions directly in a clean/baked 40mL vial filled with organic-free reagent water by adding at least 1µL, but not more than 2500µL of liquid sample. The sample is ready for addition of internal and surrogate standards. Proceed with Section 8.5.8.

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8.7 GC/MS Analysis -- Sediment/soil and waste samples

These samples may contain percent quantities of purgeable organics that will contaminate the purge-and-trap system, and require extensive cleanup and instrument downtime. The screening of samples is highly recommended. Screening data should be used in conjunction with site-specific DQOs, if known, to determine whether to use the low-concentration method (0.005 - 1 mg/Kg) or the high-concentration method (>1mg/Kg).

8.7.1 Low-concentration method -- This is designed for samples containing individual purgeable compounds of <1 mg/Kg. It is limited to sediment/soil samples and waste that is of a similar consistency (granular and porous). The low-concentration method is based on purging a heated sediment/soil sample mixed with organic-free reagent water containing the surrogate and internal standards. All QC samples and standards are to be analyzed under the same conditions as the samples, using 5g of clean sand or clean soil.

STATE NOTE: This option cannot be used for OH VAP or South Carolina samples. Please refer to SOP 330751 that addresses Method 5035 for sample preparation.

8.7.1.1 Use a 5g sample if the expected concentration is <0.1mg/Kg, or a 1g sample for expected concentrations between 0.1 and 1mg/Kg.

8.7.1.2 The GC/MS system must be set up as in Sections 8.1 and 8.2. This must be done prior to the preparation of the sample to avoid loss of volatiles from standards and samples. Both the initial and daily calibration standards (Sections 8.2 and 8.3) must be heated to 40°C purge temperature. Refer to Method 5035 (SOP #330751) for additional instructions for 8260B soil analysis.

8.7.1.3 The sample consists of the entire contents of the sample container. Do not discard any supernatant liquids. Mix the contents (using slow but precise movement to limit the loss of volatiles) of the sample container with a narrow metal spatula. Weigh the amount determined in Section 8.7.1.1 into a tared purge device. Note and record the actual weight to the nearest 0.1 g.

8.7.1.4 Add distilled water to the purging vial, which contains the weighed amount of sample, and place the vial in the purge-and-trap system.

NOTE: Prior to the placement of the vial, the procedures in Sections 8.7.1.4 must be performed rapidly and without interruption to avoid loss of volatile organics. These steps must be performed in a laboratory free of solvent fumes.

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8.7.2 High-concentration method -- The method is based on extracting the sediment/soil with methanol. A waste sample is either extracted or diluted, depending on its solubility in methanol. Wastes (i.e., petroleum and coke wastes) that are soluble in methanol are diluted. An aliquot of the extract is added to organic-free reagent water containing surrogate and internal standards. This is purged at ambient temperature. All samples with an expected concentration of >1.0 mg/Kg must be analyzed by this method.

STATE NOTE: This method is not suitable for samples from South Carolina, North Carolina or Indiana. South Carolina does not recognize the practices in sections 8.7.2.1 or 8.7.2.2. 5035 must be used for all high-level soil samples, see SOP 330751.

8.7.2.1 The sample (for volatile organics) consists of the entire contents of the sample container. Do not discard any supernatant liquids. Mix the contents (using slow but precise movement to limit the loss of volatiles) of the sample container with a narrow metal spatula. For sediment/soil and solid wastes that are insoluble in methanol, weigh 5g (wet weight) of sample into a tared 40-mL vial. Use a top-loading balance. Note and record the actual weight to 0.1g. For waste that is soluble in methanol, tetraglyme, or PEG, weigh 5g (wet weight) into a 40mL vial.

8.7.2.2 Add 5mL Methanol. Shake well for 2 minutes. See SOP 330751.

NOTE: Sections 8.7.2.1 and 8.7.2.2 must be performed rapidly and without interruption to avoid loss of volatile organics. These steps must be performed in a laboratory free from solvent fumes.

8.7.2.3 The GC/MS system must be set up as in Sections 8.1.

8.7.2.4 If a screening procedure was followed, use the estimated concentration to determine the appropriate volume. If the sample was submitted as a high-concentration sample, start with 100µL.

8.7.2.5 In a clean/baked vial filled with reagent water, inject the corresponding aliquot of methanol extract. Immediately cap and place in the autosampler. The autosampler adds 1uL of the IS/surrogate mix to all of the samples.

8.7.2.6 Proceed with the analysis as outlined in Sections 8.5.9-8.5.10. Analyze all blanks on the same instrument as that used for the samples. The standards and blanks must also contain 100µL of the dilution solvent to simulate the sample conditions.

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8.7.2.7 For a matrix spike in the high-concentration of sediment/soil samples, Add a 100µL aliquot of this extract to 5mL of organic-free reagent water for purging (as per Section 8.7.2.6) in a clean/baked 40mL VOA vial and add 20µL spiking solution, 1µL internal and surrogate standard solution (IS/Surr solution added by autosampler).

8.8 Qualitative Identification

The qualitative identification of compounds determined by this method is based on retention time, and on comparison of the sample mass spectrum, after background correction, with characteristic ions in a reference mass spectrum. The reference mass spectrum must be generated by the laboratory using the conditions of this method. All hits must be visually compared to the reference spectrum for confirmation. The characteristic ions from the reference mass spectrum are defined to be 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 criteria below are met.

8.8.1 The intensities of the characteristic ions of a compound maximize in the same scan or within one scan of each other. Selection of a peak by a data system target compound search routine where the search is based on the presence of a target chromatographic peak containing ions specific for the target compound at a compound-specific retention time is accepted as meeting this criterion.

8.8.2 The RRT of the sample component is within + 0.06 RRT units of the RRT of the standard component.

8.8.3 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%.)

8.8.4 Structural isomers that produce very similar mass spectra are identified as individual isomers if they have sufficiently different GC retention times. Sufficient GC resolution is achieved if the height of the valley between two isomer peaks is less than 25% of the sum of the two peak heights. Otherwise, structural isomers are identified as isomeric pairs.

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8.8.5 Identification is hampered when sample components are not resolved chromatographically and produce mass spectra containing ions contributed by more than one analyte. When gas chromatographic peaks obviously represent more than one sample component (i.e., a broadened peak with shoulder(s) or a valley between two or more maxima), appropriate selection of analyte spectra and background spectra are important. Examination of extracted ion current profiles of appropriate ions can aid in the selection of spectra, and in qualitative identification of compounds. When analytes co-elute (i.e., only one chromatographic peak is apparent), the identification criteria can be met, but each analyte spectrum contains extraneous ions contributed by the co-eluting compound.

8.8.6 TIC's - Tentatively Identified Compounds

Periodically, clients may request additional identification of compounds that are not normally calibrated. This identification is limited to the compounds in the current mass spectral library employed by ESC.

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 is determined by the type of analyses being conducted. At the client request, when serving the role of QA (or referee) laboratory, tentatively identified compounds (TICs) must always be reported. Guidelines for making tentative identification are:

(1) Relative intensities of major ions in the reference spectrum (ions >10% of the most abundant ion) should be present in the sample spectrum.
(2) The relative intensities of the major ions should agree within 30% to be consistent with target compound list identification. (Example: For an ion with an abundance of 50% in the standard spectrum the corresponding sample ion abundance must be between 20 and 80%).
(3) Molecular ions present in the reference spectrum should be present in the sample spectrum.
(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.
(5) Ions present in the reference spectrum but not in the sample spectrum must 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.

Computer generated library search routines must not use normalization routines that would misrepresent the library or unknown spectra when compared to each other. Only after visual comparison of sample with the nearest library searches does the mass spectral interpretation specialist assign a tentative identification.

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8.9 Quantitative Analysis

8.9.1 When a compound has been identified, the quantitation of that compound is based on the integrated abundance from the EICP of the primary characteristic ion. Quantitation is accomplished using the internal standard technique, as described in Section 9. The internal standard used must be the one nearest the retention time of that of a given analyte.

8.9.2 Sediment/soil samples are reported on a dry weight basis, while sludge and wastes are reported on a wet weight basis. The percent dry weight of the sample (see Section 9.7) must be reported along with the data in either instance. At ESC, the dry weight conversion calculations for sample reporting are performed by the LIMS system. [Dry weight only when requested]. The LIMS Final Client Report represents the reporting basis as either wet weight or dry weight, depending upon the calculation used.

9.0 DATA ANALYSIS AND CALCULATIONS

9.1 Internal Calibration Equations:

$$RF = \frac{[A_s][C_{is}]}{[A_{is}][C_s]}$$

where:
A_s = Peak area (or height) of the analyte or surrogate.
A_{is} = Peak area (or height) of the internal standard.
C_s = Concentration of the analyte or surrogate, in µg/L.
C_{is} = Concentration of the internal standard, in µg/L.

Percent Relative Standard Deviation (%RSD)

$$\overline{RF} = \frac{\sum_{i=1}^{n} RF_i}{n} \qquad SD = \sqrt{\frac{\sum_{i=1}^{n} (RF_i - \overline{RF})^2}{n-1}} \qquad RSD = \frac{SD}{\overline{RF}} x100\%$$

where:
RSD = Relative standard deviation.
RF = Mean of 5 initial RFs for a compound.
SD = Standard deviation of average RFs for a compound.

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Percent Difference

$$\frac{RFv - RF}{RF} * 100\%$$

where:
RF = Average response factor from initial calibration.
RFv = Response factor from current verification check standard.

Percent Drift

$$\%Difference = \frac{C_0 - C_1}{C_1} X100$$

where:
C_1 = compound standard concentration
C_0 = measured concentration using selected quantitation method

9.2 Linear calibration model:

$$y = mx + b$$

where: y = Response A_X for External Standard
x = Concentration C_X for External Standard
m = Slope
b = Intercept

Slope (m):

$$m = \frac{[(Swx_i y_i * Sw) - (Swx_i * Swy_i)]}{[(Sw * Swx_i^2) - (Swx_i * Swx_i)]}$$

Intercept (b):

$$b = y_{AVE} - (m * (x_{AVE}))$$

Correlation Coefficient (r):

$$r = \frac{[(Sw * Swx_i y_i) - (Swx_i * Swy_i)]}{\sqrt{\{[(Sw * Swx_i^2) - (Swx * Swx_i)] * [(Sw * Swy_i^2) - (Swy_i * Swy_i)]\}}}$$

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Coefficient of Determination (r^2):

$$r^2 = r * r$$

Where: n = number of x, y pairs
x_i = individual values for the independent variable
y_i = individual values for the dependent variable
w = weighting factor, for equal or no weighting w = 1
x_{AVE} = average of the x values
y_{AVE} = average of the y values
S = the sum of all the individual values

NOTE: As an alternative to calculating mean response factors and applying the RSD test, use the GC/MS data system software or other available software to generate a linear or second linear order regression calibration curve by plotting A/A(is) vs. Q(x). Either equal weighting factors or 1/x regressions may be used.

9.3 Concentration of Target Analytes in Water and Water-Miscible Waste

$$\text{Concentration}(ug/L) = \frac{(A_x)(I_s)(D)}{(A_{is})(\text{ave.RF})(V_s)}$$

where:

A_x = Area (or height) of the peak for the analyte in the sample.
A_{is} = Area (or height) of the peak for the internal standard.
I_s = Mass (amount) of the internal standard in the concentrated sample extract (ng). This is not just the mass injected into the instrument, but the total mass of internal standard in the concentrated extract.
D = Dilution factor, if the sample or extract was diluted prior to analysis. If no dilution was made, D = 1. The dilution factor is always dimensionless.
ave.RF = Mean response factor from the initial calibration.
V_s = Volume of the aqueous sample extracted or purged (mL). If units of liters are used for this term, multiply the results by 1000.

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9.4 Concentration of Target Analytes in Sediment/Soil, Sludge, and Waste

9.4.1 High-concentration procedure

$$\text{Concentration}(ug/L) = \frac{(A_x)(I_s)(V_t)}{(A_{is})(\text{ave.RF})(V_i)(W_s)}$$

where:
A_x, I_s, A_{is}, ave.RF are the same as in water and water-miscible waste above.
Vt = Volume of total extract (μL) (use 10,0000 μL or a factor of this when dilutions are made).
Vi = Volume of extract added (μL) for purging.
Ws = Weight of sample extracted or urged (g). The wet weight or dry weight may be used, depending upon the specific applications of the data.

9.4.2 Low-concentration procedure

$$\text{Concentration}(ug/L) = \frac{(A_x)(I_s)(V_t)}{(A_{is})(\text{ave.RF})(V_i)(W_s)}$$

where:

A, I_s, A_{is}, RF are the same as in water and water-miscible waste above.
V_t = Volume of total extract (μL) (use 10,0000 μL or a factor of this when dilutions are made).
V_i = Volume of extract added (μL) for purging.
W_s = Weight of sample extracted or urged (g). The wet weight or dry weight may be used, depending upon the specific applications of the data.

9.4.3 Soil Weight determination with Methanol (samples received with MeOH).

$$\text{SoilWeight} = \text{VialTotalWeight}(\text{vial},\text{soil},\text{MeOH}) - \text{TareVialWeight} - \text{MeOHWeight}.$$

9.5 Percent Dry Weight

$$\%\text{DryWeight} = \frac{\text{g of dry sample}}{\text{g of sample}} X100$$

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9.6 LCS/ICV/CCV Percent Recovery (%R):

$$\%R = \frac{\text{Measured concentration}}{\text{Actual concentration}} \times 100$$

9.7 Matrix Spike Recoveries ($\%R_{MS/MSD}$):

$$\%R_{MS/MSD} = \frac{O_i - O_s}{T_i} X 100$$

where: O_i = observed sample concentration with the spike added
O_s = the observed value for the sample without the spike
T_i = True value of the spike added

9.8 Relative Percent Difference (%RPD):

$$RPD = \frac{\text{Value 1 - Value 2}}{\left(\frac{\text{Value 1 + Value 2}}{2}\right)} \times 100$$

9.9 In order to report results for volatiles analysis of samples containing significant moisture (>10%) content on an "as received" basis, the calculated concentration needs to be corrected using the total solvent/water mixture volume represented as Vt. This total solvent/water volume is calculated as follows:

$$\mu L \text{ solvent/water } V_t = \left[\frac{mL \text{ of solvent} + (\% \text{ moisture} \times g \text{ of sample})}{100}\right] \times 1000 \ \mu L/mL$$

9.10 For required method performance criteria, see section 10 and 11. For corrective actions, see section 11.0.

10.0 QUALITY CONTROL AND METHOD PERFORMANCE

10.1 All analysts must meet the qualifications specified in SOP 030205, *Technical Training and Personnel Qualifications* before approval to perform this method. Analysts must complete an initial demonstration of proficiency before being approved to perform this method. Continuing proficiency must be demonstrated using proficiency testing, laboratory control sample analysis and/or MDL studies. Method performance is assessed per analyst. Updated method performance records are filed and stored in a central location within the department.

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10.2 Batches:

10.2.1 Extraction Batches:

Extraction batches are defined as sets of 1 - 20 samples. Extraction batches must include the following: 1 method blank, 1 Laboratory Control Sample/Laboratory Control Sample Duplicate pair (LCS/LCSD), 1 Matrix Spike/Spike Duplicate (MS/MSD) pair (if sufficient sample is available). Exceptions are made for waste dilution samples where the minimum batch QC must include a blank, an LCS/LCSD pair.
Additional instructions on Batch QC including required frequency, acceptance criteria and corrective actions can be found in Section 11 & Attachment VIII.

10.2.2 Analytical Batches (sequences):

Analytical batches analysis must include the following: 1 Initial Calibration Verification (ICV) and BFB tune at the beginning of run, 1 Continuing Calibration Verification (CCV) and BFB tune every 12 hours.

10.3 Perform BFB tune every 12 hours for 8260B, 8260C, and 6200B. BFB tuning for method 624 is every 24 hours. Tuning acceptance criteria are presented in Section 8.2.1. The computer software automatically evaluates the tune information. The analyst must be aware of the process used. The following options are available for acquiring the spectra for reference to meet the BFB tuning requirements. It is recommended that each initial tune verification utilize the "Autofind" function and be set up to look at three scans (the apex & ±1 scan) and average the three scans then perform background subtraction. Background subtraction is required prior to the start of the peak but no more than 20 scans prior. Background correction cannot include any parts of the target peak. The scans must be averaged and background corrected. Average scans 0.1 minute before to 0.1 minute after the target peak including 2 scans and the peak apex.

10.4 Run a minimum of a 5-point initial calibration curve (3-point can be used if 624/6200B are being run independently of 8260B), using the primary source standards each time major instrument maintenance occurs, or if the CCV does not meet acceptance criteria. Acceptance criteria for initial calibration are presented in Section 8.2. Calibration is verified by analyzing Second Source Calibration Verification (SSCV) standard; acceptance criteria for the SSCV is presented in Section 8.3.1.

10.5 Run a mid-point Initial Calibration Verification (ICV) using the primary source standards every 12 hours before sample analysis. Also run a CCV every 12 hours during an analytical sequence for 8260B, 8260C, 624 and 6200B. See sections 8.3.2 – 8.3.6 for acceptance criteria.

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10.6 Retention Time Windows:

10.6.1 To determine retention time windows for each component in the calibration mix, make three injections of the mid-level standard over the course of 72 hours. Calculate the standard deviation of the three retention times for each analyte in the mix. The retention time window is defined as the mean retention time plus or minus three times the standard deviation of the retention times established during the 72-hour period for each component. The typical estimated retention time windows are set at ±0.05 minutes. However, the experience of the analyst weighs heavily in the interpretation of chromatograms. If the standard deviation for a component is zero, substitute the standard deviation of a closely eluting compound to develop a valid retention time window.

10.6.2 Retention time windows must be re-calculated whenever a new column is installed or whenever a major modification has been made to the instrument system.

10.6.3 Compounds are identified if they fall within the retention time window specified for that compound.

10.6.4 If the retention time for any internal standard changes by more than 30 seconds from the mid-point of the current initial calibration curve, the chromatographic system must be inspected for malfunction and corrections must be made, as required.

10.7 Instrument maintenance must be performed routinely to optimize instrument performance and improve chromatography. Commonly performed maintenance includes baking traps and columns, polishing detector windows, changing injection port liners, changing pump oil, etc. A new calibration curve must be analyzed following any major maintenance performed on the analytical system.

10.8 METHOD BLANK - The analyst must confirm that this blank was analyzed at the required frequency of 1 per batch of 20 samples. The method blank must not exhibit any contamination of any analyte above ½ the report limit for any of the method target analytes. Corrective action must be performed any time method target analytes are detected above the ½ the report limit to reduce and control contamination.

10.9 LABORATORY CONTROL SAMPLES - Assess that LCSs were prepared at the required frequency of 1 per batch of 20. Routine LCS Control limits are in Attachment VIII.

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10.10 MATRIX SPIKE/MATRIX SPIKE DUPLICATE ASSESSMENT: Assess that matrix spike/matrix spike duplicates were analyzed at required frequency of 1 per batch of 20.

- The analyst also verifies that the samples were spiked at the appropriate level.
- The order of preference for spiking levels is as follows;
  1) If the target analyte concentrations are known, spike to increase the background concentration by a factor of approximately two;
  2) if an action level exists, spike at this level
  3) if neither of the first two conditions apply, spike at a level that corresponds between the low and mid-level calibration standards.
  4) All RPD results must be within the indicated control limits on the appropriate precision control charts in Attachment VIII.

Acceptance criteria are that all %Recovery and/or RPD results must be within the indicated control limits on the appropriate MS control charts. See Attachment VIII for LCS/LCSD & MS/MSD limits and QC acceptance.

10.11 SURROGATE EVALUATION: Check the surrogate calculations for correctness for all samples, blanks, ICV/CCV/SSCV, LCS/LCSD, MS, MSD, and MD. Acceptance criteria can be found in Attachment VIII: The surrogate recoveries for all QC samples must be within established control limits.

10.12 INTERNAL STANDARD AREA COUNT: When a calibration is performed at the beginning of an analytical run, the internal standard areas must be evaluated against the mid-point of the curve. Internal standard responses must be -50% to 200% to be acceptable. Samples are analyzed within a 12-hour window; the internal standards of those samples are evaluated against mid-point of the curve. Then a CCV is analyzed, this is compared to the mid-point of the initial calibration curve. Addition samples are analyzed within a 12-hour window; the internal standards of those samples are evaluated against the previous acceptable CCV. When an analytical run is started using a passing ICV (which is compared against the initial calibration mid-point to verify the calibration curve): Samples are analyzed within a 12-hour window, the internal standards of those samples are evaluated against the daily ICV. Then a CCV is analyzed, this is compared to the mid-point of the curve. Additional samples are analyzed within a 12-hour window; the internal standards of those samples are evaluated against the previous acceptable CCV.

CLIENT NOTE: For Marathon, the internal standard area counts for all calibration standards, QC samples, and samples for quantitation must not change by a factor of greater than (-50% to +130%) as per section 8.3.6

10.13 SECOND SOURCE: The second source calibration verification standard must be analyzed following each new initial calibration to verify the validity of the calibration standards. The recovery of the analytes in the SSCV must be within 30% of the expected concentration for CCC and SPCC compounds and within 40% for non-CCC/SPCC compounds. Poor performers listed in section 8.3.4 must recover within in-house calculated LCS recovery acceptance limits.

TITLE: VOLATILE ORGANIC COMPOUNDS BY GC/MS (EPA 8260B, 8260C, 624 AND SM6200B 20TH)

11.0 DATA VALIDATION AND CORRECTIVE ACTION

11.1 SITE-SPECIFIC requirements and STATE SPECIFIC criteria must be reviewed and used, if known, for data review.

STATE NOTE: If the sample is analyzed in conjunction with the Ohio VAP, corrective action for failing QC (i.e. blank, surrogate, spike, ISTD, etc.) must be performed prior to flagging data, if sufficient sample volume was submitted by the client. Corrective action can include re-analysis, if instrument malfunction is suspected, or re-preparation and re-analysis, if the failure is suspected as either extraction or sample related.

11.2 All data must undergo a primary review by the analyst. The analyst must check the performance of the initial calibration, mid-point check standard, and continuing calibrations to ensure that they meet the criteria of the method. The analyst must review any sample that has quantifiable compounds and make sure that they have been confirmed. The analyst must also verify that reported results are derived from quantitation between the MDL and the highest standard of the initial calibration curve. All calculations must be checked (any dilutions, %solids, etc.). Data must be checked for the presence or absence of appropriate flags. Comments must be noted when data is flagged.

11.3 INITIAL AND CONTINUING CALIBRATION VERIFICATION STANDARD: An Initial Calibration Verification (ICV) standard is analyzed before sample analysis can begin and a continuing calibration verification (CCV) standard was analyzed every 12 hours and meets the criteria in Section 8.0. If these criteria are exceeded, corrective action is necessary. If the source of the problem cannot be determined after corrective action has been taken, a new calibration must be generated. This criterion must be met before sample analysis begins and/or re-analysis of all samples up to the last acceptable CCV standard.

11.4 METHOD BLANK - Blank contamination above ½ the reporting limit - Assess the effect on the samples. Samples containing detectable concentrations at or just above the reporting limit may require re-analysis. Samples with concentrations below the reporting limit require no corrective action.

Blank contamination above the report limit – All samples containing detectable amounts above the reporting limit must be re-analyzed or qualified. Samples with no detectable amounts above the reporting limit do not require re-analysis, but the samples must be qualified with blank contamination and it must be mentioned in the case narrative in the data package.

Instrument blanks may be injected at any time in the sequence to verify absence of contamination. The source of contamination must be investigated and reduced or eliminated. Any time contamination is noted in the method blank, the situation and impact on the data should be discussed in the case narrative.

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11.5 LABORATORY CONTROL SAMPLES - If the recovery does not meet criteria, see section 11.6 for marginal failures. If it is still out of control limits, then all field and QC samples in the batch must be re-analyzed.

REQUIRED RE-ANALYSIS - None of the following compounds can recover beyond established criteria: 1,1-dichloroethene, trichloroethene, chlorobenzene, toluene, and benzene. If any of these compounds fail, the LCS/LCSD and all affected batch samples must be re-analyzed.

STATE NOTE: OHIO VAP LCS/LCSD: LCS's are evaluated only for the compounds listed in 7.8.3. The LCS/LCSD contain all method target analytes. For OHIO VAP and project specific requirements, the LCS/LCSD contains, at a minimum: 1,1-dichloroethene, trichloroethene, chlorobenzene, toluene, benzene, and n-hexane (Ohio VAP) and must meet both the acceptance criteria for accuracy and precision found in Attachment VIII.

STATE NOTE: SOUTH CAROLINA LCS: responses must be within 70 – 130%. No failures are acceptable; Qualifiers cannot be used. Failures require a batch re-analysis.

Qualifiers must be applied to any LCS compound that does not meet these criteria and are considered out of control. The percent difference for all method target analytes must be within QC RPD limits. If not, re-analyze the duplicate(s) or prepare a new calibration curve, as necessary.

11.6 LCS/LCSD & MS/MSD CRITERIA

11.6.1 Quality control criteria must be checked for the LCS and LCSD.

LCS or LCSD samples that do not pass the acceptable QC criteria must be re-analyzed. LCS failures can meet marginal excedence criteria below. Normal compound list for 8260/624 contains typical 90 analytes; therefore only five analyte can be considered as marginal excedences. If the failure persists, re-prepare and re-analyze the entire sample batch.

When a large number of analytes exist in the LCS, it is statistically possible for a few analytes to be outside of control limits. Upper and lower marginal excedence (ME) limits are established by +/- four times the standard deviation. The number of marginal excedence is based on the number of analytes in the LCS.

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Number of allowable marginal excedences:

>90 analytes, 5 analytes allowed in the ME limit
71 – 90 analytes, 4 analytes allowed in the ME limit.
51 – 70 analytes, 3 analytes allowed in the ME limit.
31 – 50 analytes, 2 analytes allowed in the ME limit.
11 – 30 analytes, 1 analyte allowed in the ME limit.
< 11 analytes, no analyte allowed in the ME limit.

Marginal excedences must be random events.

STATE NOTE: For South Carolina and Ohio VAP samples, marginal excedences do not apply. All outliers in QC require corrective action when possible and the data must be flagged when necessary.

11.7 MATRIX SPIKE ASSESSMENT: If acceptance criteria is not met, perform the following corrective actions as appropriate.

- If both LCS/LCSD and MS/MSD recoveries are unacceptable, then the entire batch of field and QC samples must be re-analyzed.
- If the MS/MSD is unacceptable, but the LCS is acceptable, then a potential matrix effect has been identified. Re-analyze to verify the matrix effect. If a matrix effect is still suspected, then the project manager must be contacted to discuss further alternatives and the potential impact on the project. Reported data must be flagged. Reasonable attempts must be made to address matrix interference.

Acceptance criteria are that all RPD results must be within the indicated control limits on the appropriate precision control charts in Attachment VIII. If these conditions are not met, perform the following corrective actions as appropriate.

- Re-analyze the sample to verify a matrix effect.
- If the duplicate precision is still unacceptable, and LCS precision is acceptable, then a potential matrix effect has been identified. The project manager must be contacted to discuss further alternatives and the potential impact on the project.

STATE NOTE: South Carolina requires that all target compounds meet the established MS/MSD criteria. No qualifiers can be applied, except in the circumstance where matrix interference is apparent and confirmed.

STATE NOTE: OHIO VAP SPIKES: Matrix spikes are evaluated only for the compounds listed in 7.8.3.

PROJECT SPECIFIC CRITERIA (Non-South Carolina Samples): Acceptance criteria are that all %Recovery and/or RPD results meet project-established goals.

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11.8 SURROGATE EVALUATION: If the surrogate recoveries are outside limits for Blank, ICV/CCV/SSCV, and LCS/LCSD, re-analysis must be performed for verification. If recoveries are still outside control limits, corrective action is necessary. All samples associated with batch or sequence needs to be re-analyzed. The surrogate recoveries for all field samples must be within established control limits. If more than two surrogates recoveries are outside limits, re-analysis must be performed for verification. If recoveries are still outside control limits, corrective action is necessary which includes qualifying data with J1 (outside upper limit) or J2 (outside lower limit). When one or two surrogates fail, data is qualified with J1 (outside upper limit) or J2 (outside lower limit).

STATE NOTE: OHIO VAP: The surrogate recoveries for all field and QC samples must be within established control limits. If the surrogate recoveries are outside limits, re-analysis (undiluted) must be performed for verification. Tfrecoveries are still outside control limits, corrective action is necessary which includes qualifying data with J1 (outside upper limit) or J2 (outside lower limit). The sample(s) needs to be re-analyzed to confirm the failure or following corrective actions.

11.9 INTERNAL STANDARD AREA COUNT: If the area response for any of the internal standards changes by a factor of two (-50% to +100%) as per section 8.3.6, the mass spectrometer must be inspected for malfunctions and corrections must be made, as appropriate. In the event the internal standard area counts fail these criteria, the following corrective actions should be considered.

- Check to be sure there are no errors in the internal standards preparation or addition. Also check instrument performance.
- If any internal standard criteria fails high (> +100%), sample must be re-analyzed with possible dilution. If recoveries are still outside control limits, corrective action is necessary which includes qualifying compounds with associated internal standard with J9 (IS high, data is likely to show low bias).
- If more than two internal standard criteria fails low « -50%), sample must be re-analyzed. If recoveries are still outside control limits, corrective action is necessary which includes qualifying compounds with associated internal standard with J8 (IS low, data is likely to show high bias).
- If one or two internal standard criteria fails low « -50%), corrective action is necessary which includes qualifying compounds with associated internal standard with J8 (IS low, data is likely to show high bias).

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STATE NOTE: **OHIO VAP:** The analyst checks the internal standard area counts for all calibration standards, QC samples, and samples for quantitation. If the area response for any of the internal standards changes by a factor of two (-50% to +100%) as per section 8.3.6, the mass spectrometer must be inspected for malfunctions and corrections must be made, as appropriate. In the event the internal standard area counts fail these criteria, the following corrective actions should be considered:

- Check to be sure there are no errors in the internal standards preparation or addition. Also check instrument performance.
- If any internal standard criteria fails high (> +100%), **the sample must be re-analyzed undiluted, unless obvious matrix interference is noted.** If recoveries are still outside control limits, corrective action is necessary which includes qualifying compounds with associated internal standard with J9 (IS high, data is likely to show low bias).
- If any internal standard criteria fails low « -50%), **the sample must be re-analyzed sample must be re-analyzed undiluted, unless obvious matrix interference is noted.** If recoveries are still outside control limits, corrective action is necessary which includes qualifying compounds with associated internal standard with J8 (IS low, data is likely to show high bias).

11.10 CALIBRATION RANGE: The analyst must verify all reported results are derived from analytical results that are below the highest standard of the initial calibration curve and above the low standard. Values reported below the low standard are to be reported as estimated values (J values). For samples that exceed the calibration curve, dilute and analyze an appropriate sample aliquot.

11.11 SECOND SOURCE: If the SSCV does not meet acceptance criteria, if can be reanalyzed once. If the failure persists, a new initial calibration curve must be prepared and analyzed.

11.12 Data that does not meet acceptable QC criteria may be acceptable for use in certain circumstances.

11.12.1 If a method blank contains an amount of target analyte, but all samples are non-detected or the samples contain analyte at a level that is greater than 10 times the level present in the blank, the data is reported with the appropriate “B” flag.

11.12.1.1 When comparing analyte contamination in the blank to possible analyte contamination in the field sample, utilize the sample concentration without applying the multiplier value unless the same multiplier has been applied to the quantitation of the target analytes in the blank.

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11.12.2 If the sample surrogate is above the acceptable QC range, but the samples are non-detected for all target analytes, flag the sample with a J1 and report. If the surrogate is below the acceptable QC range, re-analyze the sample if the surrogate still fails, re-extract and re-analyze or flag data.

11.12.3 Matrix spike failures must be flagged with "J5" (high) or "J6" (low), when QC limits are exceeded. If there is an RPD failure, the data is flagged with a "J3".

11.13 Quantitation and manual integration of all QC samples and client samples must follow the procedures outlined in SOP 030215, *Manual Integration*. "Before" and "After" quantitation reports must be printed in order to verify that any manual integration is performed properly and consistently.

11.14 Data must be checked to ascertain if it conforms to accepted practices. All sample analytical results used for final data reporting must be between the low standard and the high calibration standard. Values falling above the high standard must be diluted and re-analyzed.

11.14.1 Site specific DQO's may allow the reporting of values above the upper calibration standard with an "E" qualifier indicating that the value is known to be greater than the upper calibration limit. The states of South Carolina, Minnesota, Arizona, and New York, do not accept the practice of applying "E" qualifiers.

11.14.2 Site specific DQO's may require values below the reporting limit but above the method detection limit be reported as "UJ" or estimated value. The reporting limit is the concentration of the lowest standard used in the calibration.

11.14.3 All tentatively identified compounds (TICs) are reported with a "J" qualifier for estimated value and an "N" for presumptive evidence of material.

12.0 POLLUTION PREVENTION AND WASTE MANAGEMENT

12.1 The EPA requires that laboratory waste management practice to be conducted consistent with all applicable federal and state laws and regulations. Excess reagents, samples and method process wastes must be characterized and disposed of in an acceptable manner. See *ESC Waste Management Plan.*

12.2 See SOP #030302, *Pollution Prevention*.

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13.0 METHOD MODIFICATIONS/CLARIFICATIONS

13.1 Adjustments to the concentrations of standards/spiking solutions, standards providers, and quality control are subject to change to better meet client/project/regulatory needs or to improve laboratory method performance.

13.2 Modifications to this method are noted in the body of the text as state notes. Compliance analyses performed in conjunction with specific state requirements must be performed as noted within the specific state(s) note listed.

14.0 REFERENCES

14.1 "Test Methods for Evaluating Solid Waste", EPA SW-846, Method 8000B (Revision 2, September, 1996)

14.2 "Test Methods for Evaluating Solid Waste", EPA SW-846, Method 8260B (Revision 2, December, 1996)

14.2 Code of Federal Regulations, 40, Part 136, Method 624

14.3 Standard Methods for the Examination of Water and Wastewater, APHA, 20th edition, Method 6200B.

14.4 Policy Document, NELAC Standard, Chapter 2: Proficiency Testing Program Standard and the relevant section of NELAC Standard Chapter 5 National Environmental Laboratory Accreditation Conference

14.5 "Test Methods for Evaluating Solid Waste", EPA SW-846, Method 8000C (Revision 3, March, 2003)

14.6 "Test Methods for Evaluating Solid Waste", EPA SW-846, Method 8260C (Revision 3, August, 2006)

TITLE: VOLATILE ORGANIC COMPOUNDS BY GC/MS (EPA 8260B, 8260C, 624 AND SM6200B 20TH)

Attachment I: Revision History

Current Version:

Version	Date	Description of Revisions
17	3/12/12	Technical and Quality Review and update. Added Attachment VIII and sections 2.15 through 2.17, 8.2.7, 8.3.2, 9.10, 10.8 through 10.13, 13.2 14.5, 14.6, state note in section 1.0, and client note in section 11.11; Revised Attachments II through IV, VII and sections 1.3, 3.1, 4.1, 4.2, 4.3, 7.1, 7.5.1, 7.5.2, 7.9.3, 8.2.2, 8.2.3, 8.2.6, 8.3.1, 8.3.3, 8.3.4, 8.7.2, 9.1, 9.2, 9.6 through 9.8, 10.2, 10.3, 10.5, and 12.1; Removed section 9.3.

Superseded Versions:

This document supersedes the following:

Version	Date	Description of Revisions
0	8/23/94	Origination
1	7/25/95	
2	9/12/97	
3	8/4/98	
4	2/11/00	
5	8/21/00	
6	4/1/01	
7	10/16/01	
8	8/19/02	
9	7/23/03	
10	10/30/03	
11	1/26/04	
12	6/28/04	
13	12/11/04	
14	3/23/05	
15	12/19/07	
16	1/30/09	Technical and Quality Review and update. Updated Table 1; Clarified holding times; Updated Note in Section 7; Updated section 7.4.1 & 7.4.2; Updated section 7.6, 7.8.4, 8.1.2, 8.2.3, 8.2.3.2, 8.2.4.1, 8.2.6.2 & 12.0; Added state note (MN) section 8.2.2, added final bullet item, added state note (OH); Added section 8.2.6.4; section 9.11 (state note), section 11.1 (state note), section 11.7.1 (state note), section 11.13, & 13.1; Ohio VAP approved 1/30/09

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TITLE: VOLATILE ORGANIC COMPOUNDS BY GC/MS (EPA 8260B, 8260C, 624 AND SM6200B 20TH)

Attachment II: 8260/624/6200B Reporting Limits and Common Analyte List

	Water		Low Soil		High Soil	
Compound	**RL**	**Units**	**RL***	**Units**	**RL**	**Units**
Acetone	0.050	mg/L	0.050	mg/Kg	2.5	mg/Kg
Acrolein	0.050	mg/L	0.050	mg/Kg	2.5	mg/Kg
Acrylonitrile	0.010	mg/L	0.010	mg/Kg	0.5	mg/Kg
Benzene	0.001	mg/L	0.001	mg/Kg	0.05	mg/Kg
Bromobenzene	0.001	mg/L	0.001	mg/Kg	0.05	mg/Kg
Bromodichloromethane	0.001	mg/L	0.001	mg/Kg	0.05	mg/Kg
Bromoform	0.001	mg/L	0.001	mg/Kg	0.05	mg/Kg
Bromomethane	0.005	mg/L	0.005	mg/Kg	0.25	mg/Kg
1,3-Butadiene	0.0025	mg/L	0.0025	mg/Kg	0.125	mg/Kg
n-Butylbenzene	0.001	mg/L	0.001	mg/Kg	0.05	mg/Kg
sec-Butylbenzene	0.001	mg/L	0.001	mg/Kg	0.05	mg/Kg
tert-Butylbenzene	0.001	mg/L	0.001	mg/Kg	0.05	mg/Kg
Carbon tetrachloride	0.001	mg/L	0.001	mg/Kg	0.05	mg/Kg
Chlorobenzene	0.001	mg/L	0.001	mg/Kg	0.05	mg/Kg
Chlorodibromomethane	0.001	mg/L	0.001	mg/Kg	0.05	mg/Kg
Chloroethane	0.005	mg/L	0.005	mg/Kg	0.25	mg/Kg
2-Chloroethyl vinyl ether	0.050	mg/L	0.050	mg/Kg	2.5	mg/Kg
Chloroform	0.005	mg/L	0.005	mg/Kg	0.25	mg/Kg
Chloromethane	0.0025	mg/L	0.001	mg/Kg	0.05	mg/Kg
2-Chlorotoluene	0.001	mg/L	0.001	mg/Kg	0.05	mg/Kg
4-Chlorotoluene	0.001	mg/L	0.001	mg/Kg	0.05	mg/Kg
1,2-Dibromo-3-Chloropropane	0.005	mg/L	0.005	mg/Kg	0.25	mg/Kg
1,2-Dibromoethane	0.001	mg/L	0.001	mg/Kg	0.05	mg/Kg
Dibromomethane	0.001	mg/L	0.001	mg/Kg	0.05	mg/Kg
1,2-Dichlorobenzene	0.001	mg/L	0.001	mg/Kg	0.05	mg/Kg
1,3-Dichlorobenzene	0.001	mg/L	0.001	mg/Kg	0.05	mg/Kg
1,4-Dichlorobenzene	0.001	mg/L	0.001	mg/Kg	0.05	mg/Kg
Dichlorodifluoromethane	0.005	mg/L	0.005	mg/Kg	0.25	mg/Kg
1,1-Dichloroethane	0.001	mg/L	0.001	mg/Kg	0.05	mg/Kg
1,2-Dichloroethane	0.001	mg/L	0.001	mg/Kg	0.05	mg/Kg
1,1-Dichloroethene	0.001	mg/L	0.001	mg/Kg	0.05	mg/Kg
cis-1,2-Dichloroethene	0.001	mg/L	0.001	mg/Kg	0.05	mg/Kg
trans-1,2-Dichloroethene	0.001	mg/L	0.001	mg/Kg	0.05	mg/Kg
1,2-Dichloropropane	0.001	mg/L	0.001	mg/Kg	0.05	mg/Kg
1,1-Dichloropropene	0.001	mg/L	0.001	mg/Kg	0.05	mg/Kg
1,3-Dichloropropane	0.001	mg/L	0.001	mg/Kg	0.05	mg/Kg

TITLE: VOLATILE ORGANIC COMPOUNDS BY GC/MS (EPA 8260B, 8260C, 624 AND SM6200B 20TH)

Compound	RL	Units	RL*	Units	RL	Units
cis-1,3-Dichloropropene	0.001	mg/L	0.001	mg/Kg	0.05	mg/Kg
trans-1,3-Dichloropropene	0.001	mg/L	0.001	mg/Kg	0.05	mg/Kg
2,2-Dichloropropane	0.001	mg/L	0.001	mg/Kg	0.05	mg/Kg
Dicyclopentadiene	0.001	Mg/L	0.001	mg/Kg	0.05	mg/Kg
Di-isopropyl ether	0.001	mg/L	0.001	mg/Kg	0.05	mg/Kg
4-Ethyltoluene	0.001	mg/L	0.001	mg/Kg	0.05	mg/Kg
Ethylbenzene	0.001	mg/L	0.001	mg/Kg	0.05	mg/Kg
Hexachlorobutadiene	0.001	mg/L	0.001	mg/Kg	0.05	mg/Kg
Hexane	0.010	mg/L	0.010	mg/Kg	0.50	mg/Kg
Isopropylbenzene	0.001	mg/L	0.001	mg/Kg	0.05	mg/Kg
p-Isopropyltoluene	0.001	mg/L	0.001	mg/Kg	0.05	mg/Kg
Proene	0.0025	mg/L	0.0025	mg/Kg	0.125	mg/Kg
2,2,4-Trimethyl Pentane	0.001	mg/L	0.001	mg/Kg	0.05	mg/Kg
2-Butanone (MEK)	0.050	mg/L	0.050	mg/Kg	2.5	mg/Kg
Methylene Chloride	0.005	mg/L	0.005	mg/Kg	0.25	mg/Kg
4-Methyl-2-pentanone (MIBK)	0.010	mg/L	0.010	mg/Kg	0.50	mg/Kg
Methyl tert-butyl ether	0.001	mg/L	0.001	mg/Kg	0.05	mg/Kg
Naphthalene	0.005	mg/L	0.005	mg/Kg	0.25	mg/Kg
n-Propylbenzene	0.001	mg/L	0.001	mg/Kg	0.05	mg/Kg
Styrene	0.001	mg/L	0.001	mg/Kg	0.05	mg/Kg
1,1,1,2-Tetrachloroethane	0.001	mg/L	0.001	mg/Kg	0.05	mg/Kg
1,1,2,2-Tetrachloroethane	0.001	mg/L	0.001	mg/Kg	0.05	mg/Kg
Tetrachloroethene	0.001	mg/L	0.001	mg/Kg	0.05	mg/Kg
Toluene	0.005	mg/L	0.005	mg/Kg	0.25	mg/Kg
1,2,3-Trichlorobenzene	0.001	mg/L	0.001	mg/Kg	0.05	mg/Kg
1,2,4-Trichlorobenzene	0.001	mg/L	0.001	mg/Kg	0.05	mg/Kg
1,1,1-Trichloroethane	0.001	mg/L	0.001	mg/Kg	0.05	mg/Kg
1,1,2-Trichloroethane	0.001	mg/L	0.001	mg/Kg	0.05	mg/Kg
Trichloroethene	0.001	mg/L	0.001	mg/Kg	0.05	mg/Kg
Trichlorofluoromethane	0.005	mg/L	0.005	mg/Kg	0.25	mg/Kg
1,2,3-Trichloropropane	0.001	mg/L	0.001	mg/Kg	0.05	mg/Kg
1,2,4-Trimethylbenzene	0.001	mg/L	0.001	mg/Kg	0.05	mg/Kg
1,3,5-Trimethylbenzene	0.001	mg/L	0.001	mg/Kg	0.05	mg/Kg
Vinyl chloride	0.001	mg/L	0.001	mg/Kg	0.05	mg/Kg
Xylenes, Total	0.003	mg/L	0.003	mg/Kg	0.15	mg/Kg
Additional Compounds						
Acetonitrile	0.050	mg/L	0.050	mg/Kg	2.5	mg/Kg
Allyl Chloride	0.005	mg/L	0.005	mg/Kg	0.25	mg/Kg

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TITLE: VOLATILE ORGANIC COMPOUNDS BY GC/MS (EPA 8260B, 8260C, 624 AND SM6200B 20TH)

Compound	RL	Units	RL*	Units	RL	Units
Chloroprene	0.050	mg/L	0.050	mg/Kg	2.5	mg/Kg
Trans-1,4-Dichloro-2-butene	0.0025	mg/L	0.0025	mg/Kg	0.125	mg/Kg
Isobutanol	0.100	mg/L	0.100	mg/Kg	5.0	mg/Kg
1,4-Dioxane+	0.100	mg/L	0.100	mg/Kg	5.0	mg/Kg
Methacrylonitrile	0.050	mg/L	0.050	mg/Kg	2.5	mg/Kg
Methyl Methacrylate	0.005	mg/L	0.005	mg/Kg	0.25	mg/Kg
Ethyl methacrylate	0.005	mg/L	0.005	mg/Kg	0.25	mg/Kg
Propionitrile	0.050	mg/L	0.050	mg/Kg	2.5	mg/Kg
Pentachloroethane	0.005	mg/L	0.005	mg/Kg	0.25	mg/Kg
Carbon Disulfide	0.001	mg/L	0.001	mg/Kg	0.050	mg/Kg
Cyclohexanone	0.010	mg/L	0.010	mg/Kg	0.50	mg/Kg
2-Hexanone	0.010	mg/L	0.010	mg/Kg	0.50	mg/Kg
Iodomethane	0.010	mg/L	0.010	mg/Kg	0.50	mg/Kg
Isobutanol	0.001	mg/L	0.001	mg/Kg	0.050	mg/Kg
Propionitrile	0.001	mg/L	0.001	mg/Kg	0.050	mg/Kg
Vinyl Acetate	0.050	mg/L	0.050	mg/Kg	2.5	mg/Kg
Tetrahydrofuran	0.005	mg/L	0.005	mg/Kg	0.25	mg/Kg
Bromoethane	0.001	mg/L	0.001	mg/Kg	0.050	mg/Kg
2-Butanol	0.050	mg/L	0.050	mg/Kg	2.5	mg/Kg
Ethanol	0.050	mg/L	0.050	mg/Kg	2.5	mg/Kg
Tert-Butyl Alcohol	0.050	mg/L	0.050	mg/Kg	2.5	mg/Kg
Di-isopropyl ether	0.001	mg/L	0.001	mg/Kg	0.050	mg/Kg
Ethyl tert-butyl ether	0.001	mg/L	0.001	mg/Kg	0.050	mg/Kg
Methyl-tert-butyl ether	0.001	mg/L	0.001	mg/Kg	0.050	mg/Kg
Tert-Butyl alcohol	0.050	mg/L	0.050	mg/Kg	2.5	mg/Kg
Tert-Amyl Methyl Ether	0.001	mg/L	0.001	mg/Kg	0.050	mg/Kg
Tert-Butyl Formate	0.020	mg/L	0.020	mg/Kg	1.0	mg/Kg
Tert Butyl Ethyl Alcohol	0.100	mg/L	0.100	mg/Kg	5.0	mg/Kg
Tert Amyl Alcohol	0.005	mg/L	0.005	mg/Kg	0.25	mg/Kg
Dichlorofluoromethane	0.005	mg/L	0.005	mg/Kg	0.25	mg/Kg

RLs are based on a 5mL purge volume
Low Soil - Using a 5g soil sample to 5mL water – See Method 5035 (SOP# 330751) Section 8.2.4.1
High Soil – Using 100uL extract from 5g. soil sample to 5mL methanol; see Method 5035 (SOP# 330751) Sect. 8.3.1.2
+ 1-4,Dioxane has a RL of .0025 when run using the SIM mode.

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TITLE: VOLATILE ORGANIC COMPOUNDS BY GC/MS (EPA 8260B, 8260C, 624 AND SM6200B 20TH)

Attachment III: Characteristic Masses (m/z) for Purgeable Organic Compounds as printed from SW-846 Method 8260B Table 5

Compound	Primary Characteristic Ion	Secondary Characteristic Ion(s)
Acetone	58	43
Acetonitrile	41	40, 39
Acrolein	56	55, 58
Acrylonitrile	53	52, 51
Allyl alcohol	57	58, 39
Allyl chloride	76	41, 39, 78
Benzene	78	-
Benzyl chloride	91	126, 65, 128
Bromoacetone	136	43, 138, 93, 95
Bromobenzene	156	77, 158
Bromochloromethane	128	49, 130
Bromodichloromethane	83	85, 127
Bromoform	173	175, 254
Bromomethane	94	96
1,3-Butadiene	39	54
iso-Butanol	74	43
n-Butanol	56	41
2-Butanone	72	43
n-Butylbenzene	91	92, 134
sec-Butylbenzene	105	134
tert-Butylbenzene	119	91, 134
Carbon disulfide	76	78
Carbon tetrachloride	117	119
Chloral hydrate	82	44, 84, 86, 111
Chloroacetonitrile	48	75
Chlorobenzene	112	77, 114
1-Chlorobutane	56	49
Chlorodibromomethane	129	208, 206
Chloroethane	64 (49*)	66 (51*)
2-Chloroethanol	49	44, 43, 51, 80
Bis(2-chloroethyl) sulfide	109	111, 158, 160
2-Chloroethyl vinyl ether	63	65, 106
Chloroform	83	85
Chloromethane	50 (49*)	52 (51*)
Chloroprene	53	88, 90, 51
3-Chloropropionitrile	54	49, 89, 91
2-Chlorotoluene	91	126
4-Chlorotoluene	91	126
Dicyclopentadiene	66	132
1,2-Dibromo-3-chloropropane	75	155, 157

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TITLE: VOLATILE ORGANIC COMPOUNDS BY GC/MS (EPA 8260B, 8260C, 624 AND SM6200B 20TH)

Compound	Primary Characteristic Ion	Secondary Characteristic Ion(s)
Dibromochloromethane	129	127
1,2-Dibromoethane	107	109, 188
Dibromomethane	93	95, 174
1,2-Dichlorobenzene	146	111, 148
1,2-Dichlorobenzene-d4	152	115, 150
1,3-Dichlorobenzene	146	111, 148
1,4-Dichlorobenzene	146	111, 148
cis-1,4-Dichloro-2-butene	75	53, 77, 124, 89
trans-1,4-Dichloro-2-butene	53	88, 75
Dichlorodifluoromethane	85	87
1,1-Dichloroethane	63	65, 83
1,2-Dichloroethane	62	98
1,1-Dichloroethene	96	61, 63
cis-1,2-Dichloroethene	96	61, 98
trans-1,2-Dichloroethene	96	61, 98
1,2-Dichloropropane	63	112
1,3-Dichloropropane	76	78
2,2-Dichloropropane	77	97
1,3-Dichloro-2-propanol	79	43, 81, 49
1,1-Dichloropropene	75	110, 77
cis-1,3-Dichloropropene	75	77, 39
trans-1,3-Dichloropropene	75	77, 39
1,2,3,4-Diepoxybutane	55	57, 56
Diethyl ether	74	45, 59
1,4-Dioxane	88	58, 43, 57
Epichlorohydrin	57	49, 62, 51
Ethanol	31	45, 27, 46
Ethyl acetate	88	43, 45, 61
Ethylbenzene	91	106
Ethylene oxide	44	43, 42
Ethyl methacrylate	69	41, 99, 86, 114
4-Ethyltoluene	105	120
Hexachlorobutadiene	225	223, 227
Hexachloroethane	201	166, 199, 203
Hexane	57	86, 56
2-Hexanone	43	58, 57, 100
2-Hydroxypropionitrile	44	43, 42, 53
Iodomethane	142	127, 141
Isobutyl alcohol	43	41, 42, 74
Isopropylbenzene	105	120
p-Isopropyltoluene	119	134, 91
Malononitrile	66	39, 65, 38
Methacrylonitrile	41	67, 39, 52, 66

TITLE: VOLATILE ORGANIC COMPOUNDS BY GC/MS (EPA 8260B, 8260C, 624 AND SM6200B 20TH)

Compound	Primary Characteristic Ion	Secondary Characteristic Ion(s)
Methyl acrylate	55	85
Methyl-t-butyl ether	73	57
Methylene chloride	84	86, 49
Methyl ethyl ketone	72	43
Methyl iodide	142	127, 141
Methyl methacrylate	69	41, 100, 39
4-Methyl-2-pentanone	100	43, 58, 85
Naphthalene	128	-
Nitrobenzene	123	51, 77
2-Nitropropane	46	-
2-Picoline	93	66, 92, 78
Pentachloroethane	167	130, 132, 165, 169
Propargyl alcohol	55	39, 38, 53
Propene	41	39, 42
Propiolactone	42	43, 44
Propionitrile (ethyl cyanide)	54	52, 55, 40
n-Propylamine	59	41, 39
n-Propylbenzene	91	120
Pyridine	79	52
Styrene	104	78
1,2,3-Trichlorobenzene	180	182, 145
1,2,4-Trichlorobenzene	180	182, 145
1,1,1,2-Tetrachloroethane	131	133, 119
1,1,2,2-Tetrachloroethane	83	131, 85
Tetrachloroethene	164	129, 131, 166
Toluene	92	91
1,1,1-Trichloroethane	97	99, 61
1,1,2-Trichloroethane	83	97, 85
Trichloroethene	95	97, 130, 132
Trichlorofluoromethane	151	101, 153
1,2,3-Trichloropropane	75	77
1,2,4-Trimethylbenzene	105	120
1,3,5-Trimethylbenzene	105	120
Vinyl acetate	43	86
Vinyl chloride	62	64
o-Xylene	106	91
m-Xylene	106	91
p-Xylene	106	91
Internal Standards/Surrogates:		
1,4-Difluorobenzene	114	63
1,4-Dichlorobenzene-d4	152	115, 150
1,1,2-Trichloroethane-d3	100	97, 132
4-Bromofluorobenzene	95	174, 176

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TITLE: VOLATILE ORGANIC COMPOUNDS BY GC/MS (EPA 8260B, 8260C, 624 AND SM6200B 20TH)

Compound	Primary Characteristic Ion	Secondary Characteristic Ion(s)
Chloroform-d1	84	
Dibromofluoromethane	113	
4-Bromofluorobenzene	95	174, 176
Chloroform-d1	84	
Dibromofluoromethane	113	
Dichloroethane-d4	102	
Toluene-d8	98	
Pentafluorobenzene	168	
Fluorobenzene	96	77

* Characteristic ion for an ion trap mass spectrometer (to be used when ion-molecule reactions are observed).

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TITLE: VOLATILE ORGANIC COMPOUNDS BY GC/MS (EPA 8260B, 8260C, 624 AND SM6200B 20TH)

Attachment IV: Potential Compounds to be Analyzed by this Procedure

Acetone
Acetonitrile
Acrolein
Acrylonitrile
Allyl alcohol
Allyl chloride
Benzene
Benzyl chloride
Bromoacetone
Bromochloromethane (I.S.)
Bromodichloromethane
4-Bromofluorobenzene (Surr.)
Bromoform
Bromomethane
2-Butanone
Carbon disulfide
Carbon tetrachloride
Chloral hydrate
Chlorobenzene
Chlorobenzene d-5 (I.S.)
Chlorodibromomethane
Chloroethane
2-Chloroethanol
bis-(2-Chloroethyl) sulfide
2-Chloroethyl vinyl ether
Chlorofrom
Chloromethane
Chloroprene
3-Chloropropionitrile
1,2- Dibromo-3-chloropropane
1,2-Dibromoethane
Dibromomethane
1,4-Dichloro-2-butene
dichlorodifluoromethane
1,1-Dichloroethane
1,2-Dichloroethane
1,2-Dichloroethane d-4 (surr.)
1,1-Dichloroethene
Trans-1,2-dichloroethene
Cis-1,2-dichloroethene
1,2-dichloropropane
Dicyclopentadiene
1,4-Dioxane
Epichlorohydrin
Ethanol
Ethylbenzene
Ethylene oxide
Ethyl methacrylate
n-Hexane
2-Hexanone
2-Hydroxypropionitrile
Iodomethane
Isobutylalcohol
Malononitrile
Methacrylonitrile
Methylene chloride
Methyl iodide
Methyl methacrylate
4-methyl-2-pentanone
Pentachloroethane
2-Picoline
Propargyl alcohol
B-propiolactone
Propionitrile
n-Propylamine
Pyridine
Styrene
1,1,1,2-Tetrachloroethane
1,1,2,2-Tetrachloroethane
Tetrachloroethene
Toluene
1,3-Butadiene
1,1,1-Trichloroethane
1,1,2-Trichloroethane
Trichloroethene
Trichlorofluoromethane
1,2,3-Trichloropropane
Vinyl acetate
Vinyl chloride
Xylene (total)
1,2,3,4-Diepoxybutane
4-Ethyltoluene

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Attachment V: The SIM Mode:

An alternate way of running compounds to achieve lower detection limits is by way of the Single Ion Monitoring (SIM) method. The SIM method allows the Mass spec to dwell on certain ions rather than scanning the full range of masses from 35 to 300. This process allows for much lower detection limit of desired compounds. This method is only for the detection of known compounds while a TIC cannot be performed while running the SIM method. Currently 1,4-Dioxane is the only compound that is analyzed using the SIM method in the volatiles laboratory.

STATE NOTE: This procedure is not required for Ohio VAP samples as action levels for this analyte within the Ohio VAP standards are easily achievable using the normal SCAN mode.

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TITLE: VOLATILE ORGANIC COMPOUNDS BY GC/MS (EPA 8260B, 8260C, 624 AND SM6200B 20TH)

Attachment VI: EPA 624 CCC Criteria:

TABLE 5—CALIBRATION AND QC ACCEPTANCE CRITERIA—METHOD 624[a]

Parameter	Range for Q (µ/g/L)	Limit for s (µ/g/L)	Range for X (µ/g/L)	Range for P, P_s (%)
Benzene	12.8 – 27.2	6.9	15.2 – 26.0	37 – 151
Bromodichloromethane	13.1 – 26.9	6.4	10.1 – 28.0	35 – 155
Bromoform	14.2 – 25.8	5.4	11.4 – 31.1	45 – 169
Bromomethane	2.8 – 37.2	17.9	D – 41.2	D – 242
Carbon tetrachloride	14.6 – 25.4	5.2	17.2 – 23.5	70 – 140
Chlorobenzene	13.2 – 26.8	6.3	16.4 – 27.4	37 – 160
Chloroethane	7.6 – 32.4	11.4	8.4 – 40.4	14 – 230
2-Chloroethylvinyl ether	D – 44.8	25.9	D – 50.4	D – 305
Chloroform	13.5 – 26.5	6.1	13.7 – 24.2	51 – 138
Chloromethane	D – 40.8	19.8	D – 45.9	D – 273
Dibromochloromethane	13.5 – 26.5	6.1	13.8 – 26.6	53 – 149
1,2-Dichlorobenzene	12.6 – 27.4	7.1	11.8 – 34.7	18 – 190
1,3-Dichlorobenzene	14.6 – 25.4	5.5	17.0 – 28.8	59 – 156
1,4-Dichlorobenzene	12.6 – 27.4	7.1	11.8 – 34.7	18 – 190
1,1-Dichloroethane	14.5 – 25.5	5.1	14.2 – 28.5	59 – 155
1,2-Dichloroethane	13.6 – 26.4	6.0	14.3 – 27.4	49 – 155
1,1-Dichlorothene	10.1 – 29.9	9.1	3.7 – 42.3	D – 234
trans-1,2-Dichloroethene	13.9 – 26.1	5.7	13.6 – 28.5	54 – 156
1,2-Dichloropropane	6.8 – 33.2	13.8	3.8 – 36.2	D – 210
cis-1,3-Dichloropropene	4.8 – 35.2	15.8	1.0 – 39.0	D – 227
trans-1,3-Dichloropropene	10.0 – 30.0	10.4	7.6 – 32.4	17 – 183
Ethyl benzene	11.8 – 28.2	7.5	17.4 – 26.7	37 – 162
Methylene chloride	12.1 – 27.9	7.4	D – 41.0	D – 221
1,1,2,2-Tetrachloroethane	12.1 – 27.9	7.4	13.5 – 27.2	46 – 157
Tetrachloroethene	14.7 – 25.3	5.0	17.0 – 26.6	64 – 148
Toluene	14.9 – 25.1	4.8	16.6 – 26.7	47 – 150
1,1,1-Trichloroethane	15.0 – 25.0	4.6	13.7 – 30.1	52 – 162
1,1,2-Trichloroethane	14.2 – 25.8	5.5	14.3 – 27.1	52 – 150
Trichloroethene	13.3 – 26.7	6.6	18.6 – 27.6	71 – 157
Trichlorofluoromethane	9.6 – 30.4	10.0	8.9 – 31.5	17 – 181
Vinyl chloride	0.8 – 39.2	20.0	D – 43.5	D – 251

Q= Concentration measured in QC check sample, in µg/L (Section 7.5.3).
s= Standard deviation of four recovery measurements, in µg/L (Section 8.2.4).
X= Average recovery of four recovery measurements, in µg/L (Section 8.2.4).
P, P_s= Percent recovery measured, (Section 8.3.2, Section 8.4.2).
D= Detected; result must be greater than zero.
[a]Criteria were calculated assuming a QC check sample concentration of 20 µg/L.

TITLE: VOLATILE ORGANIC COMPOUNDS BY GC/MS (EPA 8260B, 8260C, 624 AND SM6200B 20TH)

Attachment VII: EPA 8260C Minimum Relative Response Factor Criteria for Initial and Continuing Calibration Verification:

Volatile Compound	Minimum Response Factor (RF)		Volatile Compound	Minimum Response Factor (RF)
Dichlorodifluoromethane	0.100		1,2-Dichloropropane	0.100
Chlorom ethane	0.100		Bromodichloromethane	0.200
Vinyl chloride	0.100		cis-1,3-Dichloropropene	0.200
Bromomethane	0.100		trans-1,3-Dichloropropene	0.100
Chloroethane	0.100		4-Methyl-2-pentanone	0.100
Trichlorofluoromethane	0.100		Toluene	0.400
1,1-Dichloroethene	0.100		1,1,2-Trichloroethane	0.100
1,1,2-Trichloro-1,2,2-trifluoroethane	0.100		Tetrachloroethene	0.200
Acetone	0.100		2-Hexanone	0.100
Carbon disulfide	0.100		Dibromochloromethane	0.100
Methyl Acetate	0.100		1,2-Dibromoethane	0.100
Methylene chloride	0.100		Chlorobenzene	0.500
trans-1,2-Dichloroethene	0.100		Ethylbenzene	0.100
cis-1,2-Dichloroethene	0.100		meta-/para-Xylene	0.100
Methyl tert-Butyl Ether	0.100		ortho-Xylene	0.300
1,1-Dichloroethane	0.200		Styrene	0.300
2-Butanone	0.100		Bromoform	0.100
Chloroform	0.200		Isopropylbenzene	0.100
1,1,1-Trichloroethane	0.100		1,1,2,2-Tetrachloroethane	0.300
Cyclohexane	0.100		1,3-Dichlorobenzene	0.600
Carbon tetrachloride	0.100		1,4-Dichlorobenzene	0.500
Benzene	0.500		1,2-Dichlorobenzene	0.400
1,2-Dichloroethane	0.100		1,2-Dibromo-3-chloropropane	0.050
Trichloroethene	0.200		1,2,4-Triichlorobenzene	0.200
Methylcyclohexane	0.100			

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TITLE: VOLATILE ORGANIC COMPOUNDS BY GC/MS (EPA 8260B, 8260C, 624 AND SM6200B 20TH)

Attachment VIII: Laboratory Control Standard and Matrix Spike QC Limits

ESC Water Acceptance Criteria for LCS and MS/MSD
(*Limits are subject to change – State Specific Limits may apply*)

ANALYTE	LCS/LCSD LIMITS *		RPD LIMITS	LCS/LCSD MARGINAL EXCEDENCE LIMITS		MS/MSD LIMITS*		RPD LIMITS
	Lower	Upper		Lower	Upper	Lower	Upper	
1,1,1,2-TETRACHLOROETHANE	77	128	20	69	136	71	130	20
1,1,1-TRICHLOROETHANE	71	126	20	62	135	58	137	20
1,1,2,2-TETRACHLOROETHANE	78	130	20	70	138	64	149	20
1,1,2-TRICHLOROETHANE	81	121	20	74	128	73	128	20
1,1,2-TRICHLOROTRIFLUOROETHANE	53	143	20	38	158	36	159	21
1,1-DICHLOROETHANE	73	123	20	64	131	58	133	20
1,1-DICHLOROETHENE	54	134	20	41	147	32	152	20
1,1-DICHLOROPROPENE	67	127	20	57	137	50	140	20
1,2,3-TRICHLOROBENZENE	77	130	20	68	138	68	135	20
1,2,3-TRICHLOROPROPANE	78	131	20	69	140	74	137	20
1,2,3-TRIMETHYLBENZENE	77	126	20	69	134	67	133	20
1,2,4-TRICHLOROBENZENE	76	127	20	68	135	67	133	20
1,2,4-TRIMETHYLBENZENE	77	129	20	68	137	62	141	20
1,2-DIBROMO-3-CHLOROPROPANE	55	142	20	40	157	55	148	22
1,2-DIBROMOETHANE	78	124	20	70	132	71	129	20
1,2-DICHLOROBENZENE	82	121	20	76	127	75	125	20
1,2-DICHLOROETHANE	69	128	20	59	138	59	135	20
1,2-DICHLOROPROPANE	77	121	20	70	128	68	126	20
1,3,5-TRIMETHYLBENZENE	78	127	20	70	135	67	136	20
1,3-DICHLOROBENZENE	77	127	20	69	135	69	131	20
1,3-DICHLOROPROPANE	78	117	20	72	123	70	122	20
1,4-DICHLOROBENZENE	79	117	20	73	124	70	123	20
1-METHYLNAPHTHALENE	58	146	20	43	161	49	151	22
2,2,4-TRIMETHYLPENTANE	47	141	20	31	156	30	153	21
2,2-DICHLOROPROPANE	63	130	20	52	141	51	141	20
2-BUTANONE (MEK)	58	144	20	44	159	51	149	22
2-CHLOROETHYL VINYL ETHER	26	172	22	5	197	10	161	40
2-CHLOROTOLUENE	78	123	20	70	131	65	133	20
2-HEXANONE	62	144	20	48	158	58	148	24

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TITLE: VOLATILE ORGANIC COMPOUNDS BY GC/MS (EPA 8260B, 8260C, 624 AND SM6200B 20TH)

ANALYTE	LCS/LCSD LIMITS *		RPD LIMITS	LCS/LCSD MARGINAL EXCEDENCE LIMITS		MS/MSD LIMITS*		RPD LIMITS
	Lower	Upper		Lower	Upper	Lower	Upper	
2-METHYLNAPHTHALENE	60	146	20	46	161	52	151	24
4-CHLOROTOLUENE	78	122	20	70	130	67	129	20
4-METHYL-2-PENTANONE (MIBK)	58	147	20	43	161	53	154	21
ACETONE	49	153	21	32	171	34	146	22
ACROLEIN	10	181	30	5	200	10	189	30
ACRYLONITRILE	53	153	20	37	170	49	162	20
BENZENE	72	119	20	64	127	51	134	20
BROMOBENZENE	76	121	20	68	128	64	130	20
BROMOCHLOROMETHANE	79	124	20	72	131	67	131	20
BROMODICHLOROMETHANE	75	127	20	66	136	67	132	20
BROMOFORM	61	136	20	48	148	59	137	20
BROMOMETHANE	42	172	20	21	194	23	177	21
1,3-BUTADIENE	70	130	20	60	140	70	130	20
CARBON DISULFIDE	19	150	20	10	172	10	165	22
CARBON TETRACHLORIDE	63	129	20	52	140	49	140	20
CHLOROBENZENE	78	123	20	70	130	69	126	20
CHLORODIBROMOMETHANE	73	128	20	64	137	68	130	20
CHLOROETHANE	52	164	20	33	182	32	177	21
CHLOROFORM	76	122	20	68	129	64	130	20
CHLOROMETHANE	50	141	20	35	157	27	155	20
CIS-1,2-DICHLOROETHENE	75	121	20	68	128	54	137	20
CIS-1,3-DICHLOROPROPENE	74	124	20	66	132	63	127	20
DIBROMOMETHANE	77	124	20	69	132	68	131	20
DICHLORODIFLUOROMETHANE	33	173	20	10	196	16	188	22
DICHLOROFLUOROMETHANE	71	141	20	59	153	46	159	20
DICYCLOPENTADIENE	70	130	20	60	140	70	130	20
DI-ISOPROPYL ETHER	66	129	20	56	140	58	133	20
ETHYL ETHER	56	144	20	42	159	47	147	20
ETHYLBENZENE	77	124	20	69	132	64	135	20
4-ETHYLTOLUENE	70	130	20	60	140	70	130	20
HEXACHLORO-1,3-BUTADIENE	71	134	20	60	145	64	140	20
IODOMETHANE	54	139	20	40	153	38	149	20

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TITLE: VOLATILE ORGANIC COMPOUNDS BY GC/MS (EPA 8260B, 8260C, 624 AND SM6200B 20TH)

ANALYTE	LCS/LCSD LIMITS *		RPD LIMITS	LCS/LCSD MARGINAL EXCEDENCE LIMITS		MS/MSD LIMITS*		RPD LIMITS
	Lower	Upper		Lower	Upper	Lower	Upper	
ISOPROPYLBENZENE	74	126	20	65	135	62	134	20
M&P-XYLENE	76	123	20	69	131	62	135	20
METHYL TERT-BUTYL ETHER	67	127	20	57	137	55	136	20
METHYLENE CHLORIDE	67	122	20	58	131	52	130	20
NAPHTHALENE	70	134	20	59	145	65	140	20
N-BUTYLBENZENE	74	130	20	65	140	62	142	20
N-HEXANE	41	143	20	24	161	16	164	20
N-PROPYLBENZENE	77	125	20	69	133	62	137	20
PROPENE	70	130	20	60	140	70	130	20
O-XYLENE	77	125	20	69	133	63	135	20
P-ISOPROPYLTOLUENE	77	132	20	68	141	64	142	20
SEC-BUTYLBENZENE	77	130	20	68	139	67	139	20
STYRENE	69	145	20	56	157	58	152	20
TERT-BUTYLBENZENE	76	131	20	67	140	66	139	20
TETRACHLOROETHENE	69	131	20	58	142	56	139	20
TETRAHYDROFURAN	41	147	22	24	165	32	163	23
TOLUENE	75	114	20	69	121	61	126	20
TPH (GC/MS) LOW FRACTION	56	133	20	43	146	35	149	20
TRANS-1,2-DICHLOROETHENE	63	127	20	52	137	45	137	20
TRANS-1,3-DICHLOROPROPENE	69	124	20	60	133	59	130	20
TRANS-1,4-DICHLORO-2-BUTENE	58	137	20	45	150	51	145	24
TRICHLOROETHENE	75	121	20	67	129	40	155	20
TRICHLOROFLUOROMETHANE	53	161	20	35	179	35	177	23
VINYL ACETATE	47	161	20	28	180	36	186	20
VINYL CHLORIDE	55	142	20	41	156	32	159	21
XYLENES, TOTAL	77	123	20	69	131	64	133	20
SURROGATE LIMITS								
4-BROMOFLUOROBENZENE	82	120						
A,A,A-TRIFLUOROTOLUENE	90	116						
DIBROMOFLUOROMETHANE	82	126						
TOLUENE-D8	92	112						

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TITLE: VOLATILE ORGANIC COMPOUNDS BY GC/MS (EPA 8260B, 8260C, 624 AND SM6200B 20TH)

ESC Soil Acceptance Criteria for LCS and MS/MSD
(Limits are subject to change – State Specific Limits may apply*)

ANALYTE	LCS/LCSD LIMITS *		RPD LIMITS	LCS/LCSD MARGINAL EXCEDENCE LIMITS		MS/MSD LIMITS*		RPD LIMITS
	Lower	Upper		Lower	Upper	Lower	Upper	
1,1,1,2-TETRACHLOROETHANE	77	129	20	68	138	34	150	23
1,1,1-TRICHLOROETHANE	70	127	20	60	137	26	159	24
1,1,2,2-TETRACHLOROETHANE	76	133	20	66	143	25	165	25
1,1,2-TRICHLOROETHANE	79	123	20	72	131	37	148	21
1,1,2-TRICHLOROTRIFLUOROETHANE	52	145	20	36	160	10	178	29
1,1-DICHLOROETHANE	74	121	20	66	129	37	144	21
1,1-DICHLOROETHENE	53	135	20	39	149	10	165	28
1,1-DICHLOROPROPENE	67	127	20	57	137	24	152	24
1,2,3-TRICHLOROBENZENE	74	131	20	65	140	10	163	33
1,2,3-TRICHLOROPROPANE	75	135	20	65	145	23	167	28
1,2,3-TRIMETHYLBENZENE	76	128	20	67	136	14	165	27
1,2,4-TRICHLOROBENZENE	72	130	20	62	140	10	162	32
1,2,4-TRIMETHYLBENZENE	75	131	20	66	141	10	162	30
1,2-DIBROMO-3-CHLOROPROPANE	55	142	20	40	156	10	171	31
1,2-DIBROMOETHANE	77	126	20	68	134	33	147	22
1,2-DICHLOROBENZENE	80	123	20	73	130	21	152	25
1,2-DICHLOROETHANE	70	128	20	60	137	36	145	20
1,2-DICHLOROPROPANE	74	125	20	65	133	36	146	21
1,3,5-TRIMETHYLBENZENE	77	129	20	68	138	10	164	30
1,3-DICHLOROBENZENE	76	128	20	67	137	10	159	28
1,3-DICHLOROPROPANE	77	118	20	70	125	38	138	22
1,4-DICHLOROBENZENE	77	119	20	70	126	18	149	26
1-METHYLNAPHTHALENE	56	146	20	41	161	5	178	36
2,2,4-TRIMETHYLPENTANE	43	144	20	26	161	10	168	38
2,2-DICHLOROPROPANE	60	132	20	48	144	16	167	26
2-BUTANONE (MEK)	56	146	20	41	161	22	168	27
2-CHLOROETHYL VINYL ETHER	17	179	22	10	200	5	200	33
2-CHLOROTOLUENE	76	125	20	68	134	17	153	28
2-HEXANONE	61	144	20	47	158	22	165	29
2-METHYLNAPHTHALENE	58	146	20	44	161	5	172	36

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TITLE: VOLATILE ORGANIC COMPOUNDS BY GC/MS (EPA 8260B, 8260C, 624 AND SM6200B 20TH)

ANALYTE	LCS/LCSD LIMITS *		RPD LIMITS	LCS/LCSD MARGINAL EXCEDENCE LIMITS		MS/MSD LIMITS*		RPD LIMITS
	Lower	Upper		Lower	Upper	Lower	Upper	
4-CHLOROTOLUENE	76	125	20	68	133	13	154	27
4-METHYL-2-PENTANONE (MIBK)	55	148	20	40	163	18	172	27
ACETONE	47	155	22	28	173	5	200	28
ACROLEIN	10	182	29	10	213	5	200	40
ACRYLONITRILE	50	155	20	33	173	12	179	26
BENZENE	72	120	20	64	128	30	145	21
BROMOBENZENE	74	122	20	66	130	20	148	26
BROMOCHLOROMETHANE	75	129	20	66	137	37	148	21
BROMODICHLOROMETHANE	74	128	20	65	137	34	148	20
BROMOFORM	62	137	20	49	149	16	158	24
BROMOMETHANE	38	180	20	14	200	5	199	25
1,3-BUTADIENE	70	130	20	60	140	70	130	20
CARBON DISULFIDE	18	152	20	10	174	10	183	29
CARBON TETRACHLORIDE	62	130	20	50	141	18	158	26
CHLOROBENZENE	77	124	20	70	131	27	149	24
CHLORODIBROMOMETHANE	74	128	20	65	137	30	149	23
CHLOROETHANE	46	173	20	25	194	5	200	25
CHLOROFORM	76	122	20	69	129	39	143	21
CHLOROMETHANE	49	143	20	33	159	10	167	23
CIS-1,2-DICHLOROETHENE	73	123	20	65	131	39	141	21
CIS-1,3-DICHLOROPROPENE	73	126	20	64	134	32	145	21
DIBROMOMETHANE	75	127	20	66	136	37	146	21
DICHLORODIFLUOROMETHANE	30	177	20	10	200	10	200	27
DICHLOROFLUOROMETHANE	70	148	20	57	161	14	183	26
DICYCLOPENTADIENE	70	130	20	60	140	70	130	20
DI-ISOPROPYL ETHER	64	131	20	53	142	31	149	20
ETHYL ETHER	56	144	20	41	159	22	161	22
ETHYLBENZENE	76	126	20	67	134	21	155	27
4-ETHYLTOLUENE	70	130	20	60	140	70	130	20
HEXACHLORO-1,3-BUTADIENE	71	134	20	60	145	10	170	37
IODOMETHANE	50	144	20	35	160	10	167	25
ISOPROPYLBENZENE	70	128	20	61	138	17	155	29

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TITLE: VOLATILE ORGANIC COMPOUNDS BY GC/MS (EPA 8260B, 8260C, 624 AND SM6200B 20TH)

ANALYTE	LCS/LCSD LIMITS *		RPD LIMITS	LCS/LCSD MARGINAL EXCEDENCE LIMITS		MS/MSD LIMITS*		RPD LIMITS
	Lower	Upper		Lower	Upper	Lower	Upper	
M&P-XYLENE	75	125	20	67	133	22	152	27
METHYL TERT-BUTYL ETHER	66	127	20	57	137	30	148	22
METHYLENE CHLORIDE	67	124	20	57	134	25	148	28
NAPHTHALENE	68	136	20	57	147	5	167	30
N-BUTYLBENZENE	71	133	20	60	143	10	171	32
N-HEXANE	42	143	20	25	160	10	167	29
N-PROPYLBENZENE	76	126	20	67	134	10	161	29
PROPENE	70	130	20	60	140	70	130	20
O-XYLENE	75	128	20	66	137	21	155	26
P-ISOPROPYLTOLUENE	75	134	20	65	144	10	172	31
SEC-BUTYLBENZENE	75	132	20	66	141	10	168	31
STYRENE	68	148	20	55	161	10	177	26
TERT-BUTYLBENZENE	75	132	20	66	141	13	165	30
TETRACHLOROETHENE	70	131	20	60	141	18	157	27
TETRAHYDROFURAN	36	148	20	17	167	10	171	28
TOLUENE	74	155	20	68	122	29	141	21
TPH (GC/MS) LOW FRACTION	64	136	20	52	148	10	158	34
TRANS-1,2-DICHLOROETHENE	63	126	20	53	137	26	147	23
TRANS-1,3-DICHLOROPROPENE	68	126	20	58	136	28	144	23
TRANS-1,4-DICHLORO-2-BUTENE	56	139	20	42	153	5	170	31
TRICHLOROETHENE	75	121	20	68	129	27	151	23
TRICHLOROFLUOROMETHANE	48	170	20	28	191	10	200	30
VINYL ACETATE	43	163	20	23	183	5	200	37
VINYL CHLORIDE	54	144	20	39	159	10	178	24
XYLENES, TOTAL	76	126	20	67	134	22	154	26
SURROGATE LIMITS								
4-BROMOFLUOROBENZENE	67	133						
A,A,A-TRIFLUOROTOLUENE	89	115						
DIBROMOFLUOROMETHANE	72	135						
TOLUENE-D8	90	113						

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TITLE: VOLATILE ORGANIC COMPOUNDS BY GC/MS (EPA 8260B, 8260C, 624 AND SM6200B 20TH)

STATE NOTE: SOUTH CAROLINA requires that ALL LCS compounds meet the 70 – 130% acceptance criteria for all target analytes and that all MS/MSD compounds meet the established criteria for all target analytes. No qualifiers are accepted.

Control limits for laboratory control samples, matrix spikes and surrogates must be updated at least annually. Even though control charts must be maintained for the LCS, MS and surrogates, the acceptance criteria derived from these charts may not be appropriate if the charted ranges are too wide. Corrective action should be performed to improve analytical procedures and/or instrument conditions in any case where the charted ranges are too wide and new limits generated.

The RPD of batch LCS/LCSD's should fall within the control limits determined from the precision control charts. However, if the RPD is outside these control limits, the batch will not be rejected, as long as the LCS recovery is acceptable. This precision information should be evaluated to see how systematic problems could be identified. If problems are suspected, the method should be fully evaluated.

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SOP/Document REVISION FORM

02/06/07 R.1.0

SOP/DOC# 330363 Current revision date & number: 3/12/12 R17

Procedure/Method : Volatile Organic Compounds by GC/MS (EPA 8260B, 8260C, 624 and SM6200B 20th)

Date	Analyst	Section	Revision	Reason*	Approvals	
					Supervisor	QA
10/10/12	SLP	8.3.1.4	When analyzing 8260B and 624 concurrently, calibration verifications are evaluated using both the 8260B criteria (Secs. 8.3.1.2 and 8.3.1.3) and the 624 criteria (Attachment VI). For analytes not on the 624 list, all target analytes (except for the poor performers) must meet a maximum of 40% drift from the calibration curve. The analyst evaluates all analytes carefully and the experience of the analyst weighs heavily when determining the usability of the data. Poor performers are allowed a maxium of 50% drift from the calibration curve. See section 8.3.3 for a listing of poor performers. STATE NOTE: For South Carolina 624 samples are only analyze for the compound list found in the 624 method.		JD6	

*Comments: Clarification of CCV criteria

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SOP/Document REVISION FORM

02/06/07 R.1.0

SOP/DOC# 330363 Current revision date & number: Rev 17, 3/12/12

Procedure/Method : Volatile Organic Compounds by GC/MS (EPA 8260B, 8260C, 624 and SM6200B 20th)

Date	Analyst	Section	Revision	Reason*	Approvals	
					Supervisor	QA
12/11/12	SLP	8.2.7.3	Add: **Linear Regression Weighting**: As an alternative to calculating mean response factors and applying the RSD test, use the GC/MS data system software or other available software to generate a linear or second order regression calibration curve, by plotting A/A(is) vs. Q(x) using the equations found in section 9.2. Either equal weighting factors or 1/x regressions may be used.	**	JD6	
		10.1	Add: **STATE NOTE**: IDOCs for South Carolina need to meet 70-130% for all compounds except the poor purgers. Poor purgers can be 60-140%.	**	JD6	

12/14/12

*Comments: ** South Carolina audit

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SOP Revision Summary

SOP:			
Author - S. Wani/C. Johnson	**Number -** 330345	**Department -** SVOC	
Title - SEMI-VOLATILE ORGANICS BY GAS CHROMATOGRAPHY/MASS SPECTROMETRY USING CAPILLARY COLUMN (EPA METHOD 8270C, EPA 8270D, EPA METHOD 625, SM			
Revision - R15	**Rev. Date -** 06/12/2012		

This Standard Operating Procedure has been amended to include changes required during normal business operations. These changes as defined by SOP 010103 (Document Control and Distribution) are routine modifications that will be incorporated into the SOP upon the next scheduled review.

Rev.	Date	Section	Brief Description
1	12/14/2012	8.3.1, 11.4, & 11.11.1	Addition of South Carolina Requirements

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(see Revision History at the end of this document for more information)

TITLE: SEMI-VOLATILE ORGANICS BY GAS CHROMATOGRAPHY/MASS SPECTROMETRY USING CAPILLARY COLUMN (EPA METHOD 8270C, EPA 8270D, EPA METHOD 625, SM 6410B, 20TH ED.), INCLUDING PROVISIONS FOR ANALYSIS IN SIM MODE.

SOP NUMBER: 330345

Prepared by: Shakir Wani/Chris Johnson

Reviewed by: Chris Johnson/Blake Judge/Dixie Marlin

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Department Manager

_____
QA Department

1.0 SCOPE AND APPLICATION

1.1 This method is used to determine the concentration of semi-volatile organic compounds in extracts prepared from many types of solid waste matrices, soils, and water samples. The lists of compounds that are routinely determined by this method are listed in Attachment II. This table represents a default list to be used in the absence of a project-specific list, which would take precedence. See section 13.4.

1.2 This method is used to quantitate most neutral, acidic and/or basic organic compounds that are soluble in methylene chloride and capable of being eluted, without derivatization, from a gas chromatographic fused-silica column coated with a slightly polar methyl silicone phase. Such compounds include polynuclear aromatic hydrocarbons, chlorinated hydrocarbons and pesticides, phthalate esters, organophosphate esters, nitrosamines, haloethers, aldehydes, ethers, ketones, anilines, pyridines, quinolines, aromatic nitro compounds, and phenols, including nitrophenols.

1.3 In general, this method is not appropriate for the quantitation of multi-component analytes (i.e. Toxaphene, Chlordane, Aroclors, etc.) because of the limited sensitivity for those analytes; however when those analytes are identified using another analytical technique, this procedure is appropriate for confirmation pending sufficient analyte concentration is present in the extract.

1.4 Detection limits, sensitivity and optimum ranges of organic compounds vary with sample matrices, extraction technique, detector parameters, and model of GC/MS.

1.5 Qualifier ions are method specified and can be found in Attachment V.

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TITLE: SEMI-VOLATILE ORGANICS BY GAS CHROMATOGRAPHY/MASS SPECTROMETRY USING CAPILLARY COLUMN (EPA METHOD 8270C, EPA 8270D, EPA METHOD 625, SM 6410B, 20TH ED.), INCLUDING PROVISIONS FOR ANALYSIS IN SIM MODE.

1.6 Use of this method is restricted to analysts who are knowledgeable in the interpretation of Mass Spectrometry and use of GC/MS systems.

1.7 The use of selected ion monitoring (SIM) is acceptable for applications requiring limits below the normal range of electron impact mass spectrometry. However, SIM may provide a lesser degree of confidence in the compound identification unless multiple ions are monitored for each compound.

1.8 An MDL study must be completed at least annually or more frequently if major instrumentation changes occur. Method Detection Limits (MDLs) are performed based on ESC SOP #030206. Updated MDL records are filed and stored in a central location within the department.

1.8.1 Limit of Detection (LOD) and Limit of Quantitation (LOQ) studies are completed at the frequency required by the TNI standard per the procedure identified in the ESC SOP #030206, *Method Detection Limits (MDL) and Limits of Detection (LOD).* Should the procedure be utilized for DOD support; then the frequency of these studies must meet the requirements of the current DOD QSM.

2.0 METHOD SUMMARY AND DEFINITIONS

2.1 Field samples are prepared for analysis by gas chromatography/mass spectrometry (GC/MS) using the appropriate sample extraction technique. See ESC SOPs 330702/330702A/330702B/330705/330707/330708/330709/330754 for extraction and extract concentration methods. A measured volume or weight of sample is extracted using the appropriate extraction technique. Liquid samples are extracted at neutral pH with methylene chloride using a separatory funnel (SOP #330702) per EPA method 3510C. Reduced volume (RV) extraction using EPA method 3510C that requires a smaller volume (usually 100mL) of field sample is also available for use where applicable. Large volume injection (LVI) extraction using EPA method 3511 that requires a smaller volume (usually 40mL) of field sample is also available for use where applicable. See section 13.5 of this procedure and ESC SOP #330702B. Solid samples are extracted with methylene chloride-acetone (1:1) using sonication (SOP #330705) or microwave (SOP #330707), where permitted.

2.2 The semi-volatile compounds are introduced into the GC/MS by directly injecting a volume of the sample extract into a gas chromatograph oven (GC) equipped with a narrow-bore fused-silica capillary column. The oven, containing the capillary column, is temperature and pressure programmed to separate the analytes by molecular composition. The capillary column transfers the eluting analytes to the detector (MS) connected to a computer that then collects and stores the information for each injection.

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TITLE: SEMI-VOLATILE ORGANICS BY GAS CHROMATOGRAPHY/MASS SPECTROMETRY USING CAPILLARY COLUMN (EPA METHOD 8270C, EPA 8270D, EPA METHOD 625, SM 6410B, 20TH ED.), INCLUDING PROVISIONS FOR ANALYSIS IN SIM MODE.

2.3 Identification of target analytes is accomplished by comparing the mass spectra of each peak with the reference spectra of authentic standards.

2.4 Quantitation of the analytes of interest is accomplished by comparing the response of a major (quantitation) ion, present in the target analyte, relative to an internal standard in each extract, in conjunction with the response factor generated from a calibration curve.

2.5 Proper quantitation ions for each compound must be selected so that no interferences are present from adjoining (or co-eluting) analytes with common ions. Proper GC conditions must be used to resolve compounds with similar mass spectra. Background subtraction of mass spectra may be necessary when matrix interference is present.

2.6 Qualitative - The identification of compounds based on retention time and comparison of the sample mass spectra, after background correction, with characteristic ions in the reference mass spectra. The reference mass spectra must be generated by the laboratory using the same analytical conditions used for the analysis of field samples. The characteristic ions from the reference mass spectra are defined as the three ions of greatest relative intensity or any ions over 30% relative intensity if less than three such ions occur in the reference spectra.

2.7 Quantitative – Following qualitative identification, the quantitation of the identified compound is based on the integrated abundance of the primary characteristic ion from the Extracted Ion Current Profile (EICP).

2.8 Tentatively Identified Compound (TIC) – Non-calibrated analytes that are, minimally, 10% of the response of the nearest internal standard. These peaks are tentatively identified using the comparison of the spectra of the peak in question to spectra in the mass spectral library for possible matches.

2.9 Initial Demonstration of Capability (IDOC) - A demonstration of capability (DOC) must be made prior to using any analytical method and any time there is a change in instrument type, personnel or testing method. Such performance must be documented and the four preparation batches following the change in personnel must not result in the failure of any batch acceptance criteria, e.g., method blank, laboratory control sample, etc. or the demonstration of capability must be repeated. See also CDOC.

2.10 Continuing Demonstration of Capability (CDOC) – At least annual verification of analyst continued ability to perform method acceptably.

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2.11 Laboratory Control Sample (LCS) / Laboratory Control Sample Duplicate (LCSD) – Duplicate aliquots of a control sample of known composition. This sample is prepared from a source that is different from the stock used to prepare the initial and continuing calibration standards. LCS/LCSD are analyzed exactly like a sample and the purpose is to determine whether the methodology is in control and whether the laboratory is capable of making accurate and precise measurements. Method precision can be determined using the results of the LCS/LCSD analysis.

2.12 Matrix Spike (MS) / Matrix Spike Duplicate (MSD) - Two aliquots of a field sample (water or soil) spiked with known quantities of specific compounds and subjected to the entire analytical procedure in order to indicate the appropriateness of the method for the matrix by measuring recovery. Method precision can be determined using the results of the MS/MSD analysis, but are subject to matrix variability issues not present in the LCS/LCSD pair.

2.13 Method Blank - An analytical control consisting of all reagents used in the analytical procedure. The method blank is used to define the level of laboratory background and reagent contamination.

2.14 Method Detection Limit (MDL) - The minimum concentration of a substance that can be analyzed with 99% confidence that the analyte concentration is greater than zero.

2.15 Reporting Limit (RL) – Also see Practical Quantitation Limit (PQL). Routinely the reporting limit is the lowest standard of the calibration curve. Technically, the reporting limit is the lowest level that can be reliably achieved within the established limits of precision and accuracy during routine laboratory operating conditions.

2.16 Practical Quantitation Limit (PQL) – The default reporting limit when other limits are not specified by the client or project. The PQL is usually a factor of 3-10 times the MDL.

2.17 Second Source Calibration Verification (SSCV) – A mid-point or low standard made from a secondary standard that is not used to construct the calibration curve. The SSCV is used to represent the calibration accuracy of the instrument and must perform within method stated criteria.

2.18 Internal Standard (ISTD) – Analytes not expected to occur naturally in field samples that are spiked to provide a consistent basis for use in internal calibration models.

2.19 Initial Calibration Verification (ICV) – Standards prepared from the primary source that are analyzed at the beginning of each workgroup to confirm that the instrument maintains calibration stability within acceptable limits.

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2.20 Internal Calibration – Internal standard calibration involves the comparison of instrument responses from the target compounds in the sample to the responses of specific spikes added to the sample or sample extract prior to injection.

2.21 Response Factor (RF) - The ratio of the peak area (or height) of the target compound in the sample or sample extract to the peak area (or height) of the relevant internal standard in the sample or sample extract.

2.22 Relative Response Factor (RRF) – The Response Factor (RF) calculated relative to the response factor of the internal standard.

2.23 Sample Extraction - A sample of a known volume or weight is prepared for analysis by removing soluble substances using solvent.

2.24 Surrogate - A compound, similar to the target analytes in chemical composition and behavior, but not expected to occur naturally in field samples. Analytes are spiked by preparation/analytical personnel to assess sample extraction and analytical efficiency in each individual field sample.

2.25 Elution - The order of emergence of chemicals from the column of a chromatograph. The chemicals then typically flow into a detector of some type. Predicting and controlling the order of elution is a key aspect of column chromatographic methods and can be modified using instrument operating conditions, column selections, etc.

2.25.1 Co-elution – Peaks that are not distinctly separated or resolved by a chromatograph. Co-elution is problematic when peaks share primary and secondary mass ions making accurate quantitation questionable.

2.26 Retention Time – The expected time that it takes for a particular analyte to pass through the system (from the column inlet to the detector) under set conditions.

2.27 External Calibration - External standard calibration involves comparison of instrument responses from the sample to the responses from the target compounds in the calibration standards. Sample peak areas (or peak heights) are compared to peak areas (or heights) of the standards. The ratio of the detector response to the amount (mass) of analyte in the calibration standard is defined as the calibration factor (CF). See DRO modification for Kansas sample analysis.

2.28 Reporting Limit Verification (RLV) – A standard analyzed following initial calibration/calibration verification at or below the analyte concentration of the routine reporting level. It is analyzed per regulatory/method requirements for drinking water analyses and various other state/national regulatory programs to verify the accuracy of field sample results at the reporting level.

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2.29 Calibration Standards – Solutions of known concentrations used to create graphic representation of the relationship between the known values, such as concentrations and instrument responses.

2.30 Linear Regression - Mathematical technique for finding the straight line that best-fits the values of a linear function, plotted on a scatter graph as data points. If a 'best fit' line is found, it can be used as the basis for estimating the future values of the function by extending it while maintaining its slope.

2.31 Quadratic Regression - Mathematical technique for finding the parabolic line that best fits the values plotted on a scatter graph as data points. If a 'best fit' line is found, it can be used as the basis for estimating the future values of the function.

2.32 Limit of Detection (LOD) - A laboratory's estimate of the minimum amount of an analyte in a given matrix that an analytical process can reliably detect in their facility. The validity of the LOD shall be verified by detection of the analyte(s) in a spiked clean matrix sample in each quality system matrix. This sample shall contain the analyte at no more than 3X the MDL for single analyte tests and 4X the MDL for multiple analyte tests. This verification shall be performed on every instrument that is to be used for analysis of samples and reporting of data. The samples used for this verification must be prepared and analyzed through all steps in the analytical process used for client samples.

2.33 Limit(s) of Quantitation (LOQ) - The minimum levels, concentrations, or quantities of a target variable (e.g., target analyte) that can be reported with a specified degree of confidence. The validity of the LOQ shall be verified by successful analysis of a spiked clean matrix sample containing the analytes of concern in each quality system matrix at 1 to 2 times the claimed LOQ. A successful analysis is one where the recovery of each analyte is within the laboratory established method acceptance criteria or client data quality objectives for accuracy. The samples used for this verification must be prepared and analyzed through all steps in the analytical process used for client samples.

2.34 Relative Retention Time (RRT) – The process of normalizing the response (peak area) of the target compound to the response of the internal standard.

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3.0 HEALTH AND SAFETY

3.1 The toxicity or carcinogenicity of each reagent used in the laboratory has not been fully established. Each chemical must be regarded as a potential health hazard and exposure to these compounds must be as low as reasonably achievable. Each laboratory is responsible for maintaining a current awareness file of OSHA regulations regarding the safe handling of the chemicals specified in this method. A reference file of material data handling sheets must also be made available to all personnel involved in the chemical analysis. Specifically, concentrated nitric and hydrochloric acids present various hazards and are moderately toxic and extremely irritating to skin and mucus membranes. Use these reagents in a fume hood whenever possible and if eye or skin contact occurs, flush with large volumes of water. Always wear safety glasses or a shield for eye protection, protective clothing and observe proper mixing when working with these reagents.

3.2 **CAUTION:** Be careful when diluting and mixing acids. ALWAYS pour acid into water when mixing. Gently heat acid mixtures (NEVER HEAT RAPIDLY), to prevent splatter from extremely exothermic reactions typical of acid-water mixtures, etc.

3.3 Prior to performing this procedure, the analyst should be familiar with the proper use of corrosive liquid spill kits and contaminant procedures.

3.4 Much of the instrumentation used in this procedure has heated zones that can cause severe burns. Always unplug all instruments before doing any maintenance that involves electrical parts.

4.0 SAMPLE PRESERVATION, CONTAINERS, HANDLING, AND STORAGE

4.1 All samples must have been collected using a sampling plan that addresses the considerations of this method.

4.2 Requirements for sample extraction are detailed in SOP numbers 330702, 330702A, 330702B, 330705, 330707, 330708, 330709, and 330754.

4.3 The sample holding time for solid samples is 14 days to extraction and, for aqueous samples, the holding time is 7 days. Holding time begins when (date and time) the samples are collected and ends either 14 or 7 days following sampling, at the time sampled.

4.4 The holding time for each extract is 40 days from sample preparation to analysis.

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4.5 The container for aqueous samples and liquid sludge being extracted using the traditional 1L EPA 3510 method are 1L amber glass bottles. For the reduced volume extraction process using the EPA 3510 method, 100mL amber glass bottles are utilized. The containers for aqueous samples being extracted using EPA Method 3511 are 40mL amber glass bottles. Add 0.008% $Na_2S_2O_3$ per liter, if residual chlorine is expected or present.

4.6 Collect solid sample materials in 4 oz. jars or larger, depending on the weight and density of the sampled materials.

4.7 All samples and extracts must be shipped and stored at <6°C.

4.8 Samples submitted for analysis that do not meet the requirements contained within this section must be addressed before performing the logging process within the laboratory. In some cases, exceeding the appropriate preservation and storage criteria can cause significant bias in the resulting data. Clients may need to resubmit samples where the conditions during shipment cause uncertainty regarding sample integrity. If samples do not meet the requirements for preservation, sampling, shipment and storage and the client approves the completion of the analytical process, sample results can be qualified and possible bias is narrated per the ESC SOP# 030201, *Data Handling*.

5.0 INTERFERENCES

5.1 Raw GC/MS data from all method blanks, samples, and spikes is evaluated for interferences. Determine if the source of interference is in the preparation and/or cleanup of samples and take corrective action to eliminate the problem.

5.2 Contamination by carryover can occur whenever high-concentration and low-concentration samples are sequentially analyzed. To reduce carryover, the sample syringe is rinsed between sample injections. Whenever an unusually concentrated sample is encountered, it should be followed by analysis of solvent to check for cross-contamination. Clean/replace injector liner or clip column, check with solvent blanks, and repeat samples if necessary.

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5.3 Choice of quantitative ions and qualifier ions: Some compounds may co-elute, so the selection of quantitation ions and qualifier ions must be made carefully so these ions are specific to each of the compounds that co-elute. Qualifier ions that are most commonly used are listed in Attachment V and are recommended from the published 8270 methods. ESC uses these ions as recommended by the method with exception of the following: 2,4-Dimethylphenol, 2-Methylphenol, 2-Nitroaniline, 4-Nitrophenol, Acenaphthene, Benzoic Acid, Benzyl Alcohol, bis(2-Chloroisopropyl)ether. There is no method stated ions for the following: Pyridine, 1-Methylnaphthalene, Biphenyl, Carbazole. Aniline and Bis (2-Chloroethyl)ether quantitation ions may vary due to chromatographic conditions causing co-elution of the shared primary ion. Both targets have strongly-responding, analyte-specific secondary ions suitable for quantitative use. Refer to Attachment V for ESC ions.

5.4 Problematic Compounds:

5.4.1 Benzidine may be subject to oxidative losses during solvent concentration and exhibits poor chromatographic behavior.

5.4.2 Hexachlorocyclopentadiene is subject to thermal decomposition in the GC inlet, as well as photochemical decomposition.

5.4.3 N-nitrosodimethylamine may be difficult to separate from the solvent using the chromatographic conditions listed in this method.

5.4.4 N-nitrosodiphenylamine decomposes in the GC inlet and can't be separated from diphenylamine.

5.4.5 Pentachlorophenol, 2,4-Dinitrophenol, 4-Nitrophenol, Benzoic Acid, 4,6-Dinitro-2-methylphenol, 4-Chloro-3-methylphenol, 2-Nitroaniline, 3-Nitroaniline, 4-Chloroaniline, and Benzyl Alcohol are subject to erratic chromatographic behavior, especially when there is high boiling material contamination of the GC system.

5.4.6 Pyridine may perform poorly at the GC injection port temperatures listed in this method. The amount of degradation may be reduced by lowering the injection port temperature. Modification of the injection port temperature may adversely affect the performance of other target analytes.

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6.0 EQUIPMENT AND SUPPLIES

6.1 Gas chromatograph/mass spectrometer system.

6.1.1 Gas chromatograph (HP 6890/7890 or equivalent)- An analytical system complete with a temperature- programmable gas chromatograph suitable for split-less injection and all required accessories, including, auto sampler, syringes, analytical columns, and gases. The capillary column is directly coupled with the source.

6.1.2 Column 1 - 30m x 0.25mm ID with a 0.25µm film thickness silicon-coated fused silica capillary column (Phenomonex ZB-5MS or equivalent).

6.1.3 Column 2 – J&W 30m x 0.25mm x 0.5um film DB5MS or an equivalent is used. Ultrapure (99.999%) Helium gas is used for a mobile phase.

6.1.4 Column 3 (LVI/RV) - 20m x 0.18mm ID with a 0.36µm film Rxi-5Sil MS or equivalent.

6.1.5 Syringes: Agilent (or equivalent) syringes sizes 10µL, 25µL, 50µL, 100µL and 1.0mL.

6.2 Mass spectrometer (HP-5973/5975 or equivalent) capable of scanning from 35 to 550 amu every 1 second, using 70 volts (nominal) electron energy in the electron impact ionization mode. The mass spectrum for decafluorotriphenylphosphine (DFTPP) must meet the applicable criteria in method 8270C, 8270D or 525 when 50ng of DFTPP GC/MS tuning standard is injected.

6.3 GC/MS interface - The interface is capillary-direct into the mass spectrometer source.

6.4 Data system (HP Chemstation with Enviroquant) - A computer system is interfaced to the mass spectrometer. The system allows the continuous acquisition and storage of machine-readable media of all mass spectra obtained throughout the duration of the chromatographic program. The computer has software that can search any GC/MS data file for ions of a specific mass and that can plot such ion abundances versus time or scan number. This type of plot is defined as Extracted Ion Current Profile (EICP). The most recent version of the EPA/NIST Mass Spectral Library is also available

6.5 Volumetric flasks, Class A - Appropriate sizes with ground-glass stoppers.

6.6 Balance - Analytical, capable of weighing 0.0001g

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7.0 REAGENTS AND STANDARDS

7.1 All reagents and standards must be recorded in the appropriate preparation log and assigned a unique number. See SOP 030203, *Reagent Logs and Records*, and SOP 030230, *Standard Logger*. Additional information regarding reagent preparation can be found in the Standards Logger (Tree) digital archive system. All spiking solutions and surrogate standard solutions should be replaced at least every 6 months, or sooner if a problem is detected unless otherwise noted.

7.2 Reagent grade inorganic chemicals are used in all tests. Unless otherwise indicated, it is intended that all reagents shall conform to the specifications of the committee on Analytical Reagents of the American Chemical Society, where such specifications are available. Other grades may be used, provided it is ascertained that the reagent is of sufficiently high purity to permit its use without lessening the accuracy of the determination.

7.3 Organic-free reagent water - all references to water in this method refer to organic-free reagent water (ASTM II or equivalent).

7.4 Burdick & Jackson Omni Solv Dichloromethane Dx0831-1 (or equivalent)

7.5 Stock standard solutions - Standard solutions are purchased as certified solutions. Commercially–prepared stock standards are used at concentrations that are certified by the manufacturer or by an independent source.

7.5.1 Restek, Custom 8270 Mix – 56321, or equivalent, at 200ppm

7.5.2 NSI, 8270 TCL Project Mix – Q4296, or equivalent, at 1000ppm

7.5.3 AccuStandard, Composite Mix #3 – Z-014E-R3, or equivalent, at 2000ppm

7.5.4 Restek, Benzoic Acid Mix – 31879, or equivalent, at 2000ppm

7.5.5 Restek, Benzidine Mix #2 – 31852, or equivalent, at 1000ppm

7.5.6 AccuStandard, 2-Nitrodiphenylamine – S-4829A, or equivalent, at 200ppm

7.5.7 B/N Surrogate Mix – C-376M-39, or equivalent, at 2000ppm

7.5.8 Organic Acid Surrogate Mix – C-131M-24, or equivalent, 4000ppm

7.5.9 Second Source: Restek, 8270 MegaMix – 31850, or equivalent, at 1000ppm

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7.5.10 Transfer the stock standard solutions into bottles with PTFE-lined screw caps. Store, protected from light, at –10°C or less or as recommended by the standard manufacturer. Stock standards should be checked frequently for signs of degradation or evaporation, especially just prior to preparing calibration standards from them. Stock standards are assigned a 6 month expiration date from the day that a sealed ampoule is opened. Standards are discarded if signs of degradation are apparent when compared to a second source standard.

7.6 For PAHs by SIM, use a custom mix purchased from Ultra Scientific (Cat#: CUS-9356) with all required PAH targets. Each target compound is at concentration of 200ppm. Other concentrations may be acceptable with dilutions as appropriate for yielding the appropriate concentrations in the secondary source. The secondary source is also a custom mix from Ultra Scientific (Cat#: CUS-9345) at 200ppm.

7.7 For the DROMO by GC/MS, use a custom mix purchased from Ultra Scientific (Cat#: CUS-8255), or equivalent, which is a neat solution of diesel and an Ultra Scientific custom mix (Cat#: CUS-8254) for the gasoline components at neat. Alternatively, calibration standards can be prepared using the TX TPH Calibration Mix from Restek (Cat#: 31483) at 10,000ppm each. The secondary source is an NSI, Diesel Range Organic Spike (Cat#: Q4394) at 2500ppm each.

7.8 Internal standards solutions- the internal standards are naphthalene-d8, acenaphthene-d10, phenanthrene-d10, chrysene-d12, perylene-d12 and 1-4 dichlorobenzene-d4. Purchase from NSI (Cat # Q-6343-O) as certified stock solution at 800µg/mL. Alternative internal standard concentrations may be used for RV and LVI work. Internal standard intermediates at 16µg/mL and 4µg/mL are prepared for spiking, LVI 8270 and RV/LVI 8270SIM analyses, respectively.

7.8.1 For all soil, 1000mL water, and 8270 full run water reduced volume extracts, use the 800µg/mL internal standard solution. Each sample extract undergoing analysis is spiked with 10µL of internal standard intermediate solution, resulting in a concentration of 8µg/mL of each internal standard.

7.8.2 For other reduced volume and 3511 analyses, including DROMO by LVI, use the 16µg/mL ISTD intermediate. For reduced volume and EPA 3511 extracts being analyzed by the SIM process, use the 4µg/mL ISTD intermediate. Each sample extract undergoing analysis is spiked with 10µL of the appropriate internal standard intermediate solution, resulting in a concentration of 160µg/L and 40µg/L, respectively, for each internal standard.

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7.9 Preparation of Intermediate Standard

Stock Mix	Section ID:	Amount Added (mL)	Concentration of Stock in ppm	Concentration in Intermediate (ppm)
Restek - Custom Mix with Surrogates	7.5.1	--	200	--
NSI - 8270 TCL Project Mix	7.5.2	2.0	1000	200
AccuStandard - Composite Mix #3	7.5.3	1.0	2000	200
Restek - Benzoic Acid Mix	7.5.4	1.0	2000	200
Restek - Benzidine Mix #2	7.5.5	1.0	2000	200
AccuStandard - 2-Nitrodiphenylamine	7.5.6	2.0	1000	200

Using a volumetric syringe, measure each of the solutions listed in Section 7.9 and place into a 10mL volumetric flask. The final concentration will be 200µg/mL of each component. Use this solution or the certified custom mix purchased from Restek in section 7.5.1 to prepare the working standards in the tables in section 7.10.

7.9.1 For 1L extractions using SIM, prepare a 5µg/mL intermediate by diluting the 10µg/mL PAH mix described in section 7.6.

7.9.2 For EPA Method 3511 extracts for 8270 analyses, PAH reduced volume, and EPA 3511 analyses, a 2µg/mL intermediate is prepared directly by diluting 100µL of the 200µg/mL stock to a final volume of 10mL using volumetric glassware.

7.9.3 For SIM analyses using reduced volume or EPA 3511 extracts, a 200ug/L intermediate is prepared directly by diluting 10µL from the 200µg/mL stock to a final volume of 10mL using volumetric glassware.

7.9.4 For DROMO analyses using extraction method 3511, prepare a 200ug/mL intermediate in 10mL of methylene chloride by adding 40uL of each Gasoline and Diesel at 50,000ug/mL.

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7.10 Preparation of Working Standards

Standards must be stored at 4 ± 2°C. The expiration date of any working standard will be 6 months unless the manufacturer's stock expires prior to that date or if the standard starts showing signs of degradation. See section 7.10.1 through 7.10.6 for preparation instructions. Concentrations of standards used are subject to change depending on instrument condition, client needs and sample preparation method of the variety of analysis being performed. A minimum of five calibration levels is required for Method 8270C and 8270D, while a minimum of 3 calibration levels is required for Method 625.

7.10.1 8270C/D Calibration standards for all soil and 1L water extractions: A minimum of five calibration standards is prepared at different concentrations. At least one of the calibration standards must correspond to a sample concentration at or below the laboratory's reporting limit (RL). The remaining standards correspond to the working range of the GC/MS system. Each standard contains each analyte for detection. Working standards are made directly from the intermediate stock standard described in section 7.9o give solutions at concentrations of 0.2µg/mL up to 50µg/mL. Surrogates are included at the same concentrations. Internal standards are spiked at a constant of 8µg/mL for quantitation purposes.

TABLE 7.10.1
BNA Standard Initial Calibration (5-point)

SVOC mix (200ppm) µL	ISTD mix uL	Final volume	Final conc. ppm	Level
1	10	1.0mL	0.2	1
5	10	1.0mL	1	2
10	10	1.0mL	2	3
25	10	1.0mL	5	4
50	10	1.0mL	10	5
75	10	1.0mL	15	6
100	10	1.0mL	20	7
150	10	1.0mL	30	8
200	10	1.0mL	40	9
250	10	1.0mL	50	10

A minimum of 5 points are used to construct the calibration curve.

7.10.2 Calibration standards for 8270C/D reduced volume and EPA 3511 extracted samples: A minimum of five calibration standards is prepared at different concentrations. At least one of the calibration standards must correspond to a sample concentration at or below the laboratory-reporting limit (RL). The remaining standards correspond to the working range of the GC/MS system. Each standard contains each analyte for detection. Working standards are made directly from the intermediates described in section 7.9 to give solutions at concentrations of 0.01µg/mL up to 1µg/mL. Surrogates are included at the same concentrations. Internal standards are spiked at a constant of 160µg/L for quantitation purposes.

SVOC mix (2ppm) µL	ISTD mix uL	Final volume	Final conc. ppb	Level
5	10	1.0mL	10	1
25	10	1.0mL	50	2
50	10	1.0mL	100	3
100	10	1.0mL	200	4
200	10	1.0mL	400	5
300	10	1.0mL	600	6
400	10	1.0mL	800	7
500	10	1.0mL	1000	8

7.10.3 For SIM analyses for all soil and 1L water extractions**,** calibration standards are diluted from the intermediate standard solution (section 7.9.1) to give a calibration at the following concentrations: 20, 50, 100, 500, 1000, 2000, 4000, 10,000µg/L. A minimum of five calibration standards is prepared at different concentrations. At least one of the calibration standards must correspond to a sample concentration at or below the laboratory-reporting limit (RL). The calibration levels may change based on the working range of the GC/MS system. Surrogates are included at the same concentrations. The internal standards are at a constant 8µg/mL.

SIM Standard Concentration (ug/L)	Amount Added (uL) 5µg/mL Int.	Final Volume (mL)
20	2.0	1.0
50	5.0	1.0
100	10.0	1.0
500	50.0	1.0
1000	100.0	1.0
2000	200.0	1.0
4000	400.0	1.0
10000	1000.0	1.0

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7.10.4 For SIM analyses using reduced volume or EPA 3511 extracts**,** calibration standards are diluted from the intermediate standard solution (section 7.9.3) to give a calibration at the following concentrations: 1, 5, 10, 20, 40, 80, 200µg/L. A minimum of five calibration standards is prepared at different concentrations. At least one of the calibration standards must correspond to a sample concentration at or below the laboratory-reporting limit (RL). The calibration levels may change based on the working range of the GC/MS system. Surrogates are included at the same concentrations. The internal standards are at a constant 8µg/mL.

SIM RV/LVI Standard Concentration (ug/L)	Amount Added (uL) 200µg/L Int.	Final Volume (mL)
1	5	1.0
5	25	1.0
10	50	1.0
20	100	1.0
40	200	1.0
80	400	1.0
200	1000	1.0

7.10.5 For SIM, prepare a 1.0µg/mL surrogate standard. Add a 1.0mL to samples QC (method blanks, MS/MSD and LCS/LCSD) prior to extraction. Concentration will be 1µg/mL in the extract. Matrix spike and laboratory control sample standards are from a different source or a different lot number than the calibration. Prepare a PAH matrix spiking solution at 2.0µg/mL. Add 1.0mL to spikes (MS/MSD/LCS/ LCSD) prior to extraction. Concentration will be 2ppm in the extract.

7.10.6 For Missouri DRO analysis by 3511, prepare the working calibration curve as reflected in the following table.

DROMO mix (200ppb) µL	Final Volume	Final conc. (ppb)
25	1.0 mL	5
50	1.0 mL	10
100	1.0 mL	20
200	1.0 mL	40
400	1.0 mL	80
600	1.0 mL	120
800	1.0 mL	160

7.11 DFTPP Standard Prep for 50ppm Solution – 50µL of 1000 ppm DFTPP (AccuStandard M-625-TS-20X) + 950µL of Methylene Chloride (final volume of 1mL).

7.12 DFTPP Standard Prep for 25ppm Solution – 25µL of 1000 ppm DFTPP (AccuStandard M-625-TS-20X) + 950µL of Methylene Chloride (final volume of 1mL).

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7.12 Surrogates and Spike Solutions - Preparation techniques are detailed in SOP numbers 330702, 330702A, 330702B, 330705, 330707, 330708, 330709, and 330754.

7.13 See section 13.4 for additional information regarding standards and spiking solutions.

8.0 PROCEDURE

STATE NOTE: For samples analyzed in conjunction with the Ohio VAP program, the criteria found and itemized in this procedure for EPA method 8270C must be utilized.

8.1 GC Conditions: The GC conditions are listed in each instrument maintenance log and are updated as necessary.

8.2 Mass Spectrometer Tuning Criteria: The GC/MS is hardware-tuned using a 50ng injection of DFTPP. Analyses must not begin until the tuning criteria in Table 8.2a/b are met. The following options are available for acquiring the spectra for reference to meet the DFTPP tuning requirements. It is recommended that each initial tune verification utilize the "Autofind" function and be set up to look at three scans (the apex & ±1 scan) and average the three scans then perform background subtraction. Background subtraction is required prior to the start of the peak but no more than 20 scans prior. Background correction cannot include any parts of the target peak. The scans must be averaged and background corrected. Average scans 0.1 minute before to 0.1 minute after the target peak including 2 scans and the peak apex. The mass spectrometer must be tuned every 12 hours if samples, standards, etc. are to be analyzed for Method 8270C or 8270D or every 24 hours for Method 625.

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TABLE 8.2a
Method 8270C/625/SM6410B:
DFTPP Key Ions And Ion Abundance Criteria[a, b]

Mass	Ion Abundance Criteria
51	30-60% of mass 198
68	<2% of mass 69
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 < mass 442
442	>40% of mass 198
443	17-23% of mass 442

(a) Data taken from Reference 3 in SW-846 Method 8270C.
(b) Alternate tuning criteria may be used (e.g., CLP, Method 525, or manufacturers' instructions), providing that method performance is not adversely affected.

TABLE 8.2b
Method 8270D:
DFTPP Key Ions And Ion Abundance Criteria[a, b]

Mass	Ion Abundance Criteria
51	10-80% of mass 198
68	<2% of mass 69
70	<2% of mass 69
127	10-80% of mass 198
197	<2% of mass 198
198	Base peak, or >50% of mass 442
199	5-9% of mass 198
275	10-60% of mass 198
365	>1% of mass 198
441	Present, but <24% of mass 442
442	Base peak, or >50% of mass 198
443	15-24% of mass 442

(a) Data taken from Table 3 in SW-846 Method 8270D.
(b) Alternate tuning criteria may be used (e.g., CLP, Method 525, or manufacturers' instructions), providing that method performance is not adversely affected.

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TABLE 8.2c
Method 525:
DFTPP Key Ions and Ion Abundance Criteria

Mass	Ion Abundance Criteria
51	10-80% of base peak
68	<2% of mass 69
70	<2% of mass 69
127	10-80% of base peak
197	<2% of mass 198
198	Base peak, or >50% of mass 442
199	5-9% of mass 198
275	10-60% of base peak
365	>1% of base peak
441	Present, but < mass 443
442	Base peak, or >50% of mass 198
443	15-24% of mass 442

STATE NOTE: All South Carolina samples require a tune every 12 hours, regardless of which method is being utilized.

The GC/MS tuning standard solution must also be used to assess GC column performance and injection port inertness. Degradation of DDT to DDE and DDD is used to assess breakdown occurring in the injection port. The calculation for the determination of the breakdown occurring is found in section 9.1 and must include both DDD and DDE. Breakdown must not exceed 20%. Benzidine and pentachlorophenol are used to assess tailing occurring within the analytical system and both analytes should be present at their normal responses with no obvious peak tailing. To determine the tailing factor for benzidine and pentachlorophenol, use the calculation found in section 9.2. For EPA Methods 625 and 8270C, benzidine must have a tailing ratio of <3 and pentachlorophenol must have a tailing ratio of <5. For EPA Method 8270D, benzidine and pentachlorophenol must have a tailing ratio of <2. The Missouri diesel method does not require tailing or degradation checks prior to or during analysis.

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8.3 Calibration

8.3.1 **Initial Calibration**

EPA Method 8270C: The working standards prepared in section 7.10 are injected and average response factors are calculated. The calibration curve is typically constructed of six to nine standards, however, this may change depending on instrument conditions and/or client needs (see Section 13.4). See section 8.3.2 for information regarding use and deletion of calibration points. The calibration check compounds (CCCs) listed in Section 8.3.1a must have an average percent relative standard deviation (%RSD) of less than or equal to 30%. Any target analyte that has a %RSD >15% for the RF must be calculated by linear or quadratic regression instead of RF. If the RSD of any target analyte is ≤15%, the average response factor may be used for quantitation. When any compound does not meet the calibration criteria for RF, the analyst MUST use linear regression or quadratic curve fit. The calibration curve cannot be forced through zero and does not include a method blank. It must also meet a correlation coefficient of 0.990 or better. Analyses being generated for USACE projects must meet a correlation coefficient of 0.995 or better. If a quadratic curve fit is used, a minimum of 6 calibration standards must be utilized to obtain a working calibration curve.

The system performance check compounds (SPCCs) in Table 8.3.1b must have an average RF of ≥0.05. When these criteria are met, samples can be analyzed.

Table 8.3.1a: Calibration Check Compounds (CCC)

Base/Neutral Fraction	Acid Fraction
Acenaphthene	4-Chloro-3-methylphenol
1,4-Dichlorobenzene	2,4-Dichlorophenol
Hexachlorobutadiene	2-Nitrophenol
n-Nitrosodiphenylamine	Phenol
Di-n-octyl phthalate	Pentachlorophenol
Fluoranthene	2,4,6-Trichlorophenol
Benzo(a)pyrene	

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Table 8.3.1b: System Performance Check Compounds (SPCC)

Compound	*Minimum Average Response Factor*
n-Nitroso-di-n-propylamine	>0.05
Hexachlorocyclopentadiene	>0.05
2,4-Dinitrophenol	>0.05
4-Nitrophenol	>0.05

EPA Method 8270D: The working standards prepared in section 7.10 are injected and average response factors are calculated. The calibration curve is typically constructed of six to nine standards, however, this may change depending on instrument conditions and/or client needs (see section 13.4). At least five standards are required for Response Factor and linear regression calibration. If a quadratic curve fit is used, a minimum of 6 calibration standards must be utilized to obtain a working calibration curve. See section 8.3.2 for information regarding use and deletion of calibration points.

Target analytes must have an average RSD of ≤20%. Any target analyte that has a %RSD >20% for the RF must be calculated by linear or quadratic regression instead of RF. If the RSD of any target analyte is ≤20%, the average response factor may be used for quantitation. When any compound does not meet the calibration criteria for RF, the analyst MUST use linear regression or, if permitted, quadratic curve fit. The calibration curve cannot be forced through zero. It must also meet a correlation coefficient of 0.990 or better. Analyses being generated for USACE projects must meet a correlation coefficient of 0.995 or better.

In addition to the minimum %RSD criteria, it is recommended that a minimum response factor for the most common target analytes be demonstrated for each individual calibration level to ensure that these compounds are performing as expected. See Table 8.3.1c. Meeting the minimum response factor criteria for the lowest calibration standard is critical in establishing and demonstrating the desired sensitivity.

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Table 8.3.1c: Recommended Minimum Response Factors for Each Calibration Level (Initial and Continuing Calibration)

Compound	*Minimum Response Factor*	*Compound*	*Minimum Response Factor*
Benaldehyde	0.010	4-Nitrophenol	0.010
Phenol	0.800	Dibenzofuran	0.800
Bis(2-chloroethyl)ether	0.700	2,4-Dinitrotoluene	0.200
2-Chlorophenol	0.800	Diethyl phthalate	0.010
2-Methylphenol	0.700	1,2,4,5-Tetrachlorobenzene	0.010
2,2-Oxybis-(1-chloropropane)	0.010	4-Chlorophenyl-phenyl ether	0.400
Acetophenone	0.010	Fluorene	0.900
4-Methylphenol	0.600	4-Nitroaniline	0.010
n-Nitroso-di-n-propylamine	0.500	4,6-Dinitro-2-methylphenol	0.010
Hexachloroethane	0.300	4-Bromophenyl-phenyl ether	0.100
Nitrobenzene	0.200	n-Nitrosodiphenylamine	0.010
Isophorone	0.400	Hexachlorobenzene	0.100
2-Nitrophenol	0.100	Atrazine	0.010
2,4-Dimethylphenol	0.200	Pentachlorophenol	0.050
Bis(2-chloroethoxy)methane	0.300	Phenanthrene	0.700
2,4-Dichlorophenol	0.200	Anthracene	0.700
Naphthalene	0.700	Carbazole	0.010
4-Chloroaniline	0.010	Di-n-butyl phthalate	0.010
Hexachlorobutadiene	0.010	Fluoranthene	0.600
Caprolactam	0.010	Pyrene	0.600
4-Chloro-3-methylphenol	0.200	Butyl Benzyl phthalate	0.010
2-Methylnaphthalene	0.400	3,3-Dichlorobenzidine	0.010
Hexachlorocyclopentadiene	0.050	Benzo(a)anthracene	0.800
2,4,6-Trichlorophenol	0.200	Chrysene	0.700
2,4,5-Trichlorophenol	0.200	Bis (2-ethylhexyl)phthalate	0.010
1,1-Biphenyl	0.010	Di-n-octyl phthalate	0.010
2-Chloronaphthalene	0.800	Benzo(b)fluoranthene	0.700
2-Nitroaniline	0.010	Benzo(k)fluoranthene	0.700
Dimethyl phthalate	0.010	Benzo(a)pyrene	0.700
2,6-Dinitrotoluene	0.200	Indeno(1,23-c,d)pyrene	0.500
Acenaphthylene	0.900	Dibenz(a,h)anthracene	0.400
3-Nitroaniline	0.010	Benzo(g,h,i)perylene	0.500
Acenaphthene	0.900	2,3,4,6-Tetrachlorophenol	0.010
2,4-Dinitrophenol	0.010		

GC/MS SIM: All target compounds must be treated as CCCs. All analytes must have an average RSD of ≤30%.

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EPA Method 625: The working standards prepared in section 7.10 are injected and average response factors are calculated. The calibration curve is typically constructed of three standards, however, this may change depending on instrument conditions and/or client needs. A minimum of 3 points calibration is required for method 625. The %RSD is calculated for the standards analyzed and must be ≤35% for all compounds in order to assume linearity.

All Published Methods: Reference spectra must be updated upon analysis of each new calibration curve.

STATE NOTE: For all Minnesota sample analyses, the RL level standard is re-injected and quantitated against the newly updated calibration curve or the applicable standards are reprocessed (re-quantitated) using the completed calibration curve and is evaluated for the ±40% deviation criterion with the exception of the listed poor performers in this procedure.

STATE NOTE: For all Wisconsin sample analyses, analysts must evaluate the %RSD of calibrations to ensure that they do not have unacceptable curvature. The %RSD limit criteria, as found in the specific methods listed above, applies to calibrations using average RF calibrations. For linear and quadratic curve fits, a limit of 40% RSD is used for normal target analytes and 50% RSD is utilized for known poor performing compounds.

STATE NOTE: When analyzing samples in conjunction with the Ohio VAP and South Carolina programs, the calibration model must be RSD or linear. Quadratic curve modeling is not permitted unless historical performance of analytes exhibited a nonlinear response. Quadratic models cannot be used to extend the calibration range or bypass instrument maintenance.

8.3.2 CALIBRATION POINTS – Usage and Deletion

When the appropriate number of calibration standards is used, all points must be considered in the average response factor calculation or linear regression calculation. The deletion of the highest point is acceptable when necessary, with the analyst noting that the high end of the calibration has been lowered. The deletion of the lowest calibration point is acceptable, when necessary, provided that the analyst notes the deletion on the injection log and raises the reporting limit, if necessary, for that compound.

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8.3.3 **EPA Method 8270D:** LINEAR REGRESSION USE – The method of linear regression calibration has the potential for a significant bias to the lower portion of the calibration model. This bias is not normally seen in relative percent difference methods. When utilizing linear regression fits, a minimum quantitation check on the viability of the lowest calibration point should be performed by re-fitting the response from the lowest concentration standard back into the completed calibration curve. It is not necessary to re-analyze a low concentration standard, but using the analytical system software, the low standard can be re-quantitated as if it were a field sample. The recalculated concentrations of the analytes utilizing the linear regression curve fit must be within ±30% of the true standard concentration.

STATE NOTE: For the analysis of South Carolina samples, Hexachlorophene is required to utilize linear regression. Quadratic curve fit is not allowed. To achieve this, the calibration curve may be modified by the removal of the lowest two levels and will utilize calibration levels of 60, 80, 100, 120, and 140 for quantitation of this analyte in South Carolina samples. The reporting limit (RL) for South Carolina will routinely be 100ppb for water samples.

8.3.4 Second Source Calibration Verification – the initial calibration for each target analyte must be checked with a standard from a source that is different from those used for initial calibration.

8.3.5 **Daily Tuning and Continuing Calibration**

As with the initial calibration, the system must be tuned with 50ng of DFTPP to meet the acceptance criteria found in section 8.1. Following successful tuning, the midpoint level standard (CCV) is analyzed. Calibration verification for each method, as listed below, must be met prior to the analysis of field samples.

EPA Method 8270C: The percent difference of the CCCs (see Table 8.3.1a & b) in the mid-level standard must be ≤20% and the SPCCs must have an RF ≥0.05. The retention time of the internal standards must be within ±30 seconds from the mid-point standard level of the last initial calibration curve and the area response must be within –50% to +100%. Once these criteria are met, samples can be analyzed.

EPA Method 625: The calculated recovery for any parameter in the method from the mid-level standard must not vary by more than ±20% drift from the initial calibration curve.

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EPA Methods 8270C and 625 (analyzed concurrently): The CCV must be evaluated for CCC and SPCCs as per EPA Method 8270C requirements. All non-CCC and other target analytes must meet the criteria established in Method 625 for all analytes (±20%). For analytes not contained in the Method 625 analyte list, the analyst evaluates the CCV and the experience of the analyst weighs heavily in determining the usability of the data.

STATE NOTE: For all Wisconsin sample analyses, non-CCC compounds for 8270C requires a ±50% criteria for the CCV.

GC/MS SIM: When using the SIM method, all compounds in the CCV must be treated as CCCs and must meet the minimum requirements of ≤20% difference.

EPA Method 8270D: Each of the most common target analytes in the CCV must meet the minimum response factors in Table 8.3.1c. When using the average RF, the percent difference for each target compound in the CCV must be ≤20%. When using regression fit calibration, the percent drift of the CCV must be ≤20%. The retention time of the internal standards must be within ±30 seconds from the mid-point standard level of the last initial calibration curve and the area response must be within –50% to +100%.

8.3.6 For corrective action regarding tuning and calibration, see sections 11.1 and 11.2.

8.4 Method Blank Analysis – A method blank should be analyzed prior to any field sample analysis to verify that the analytical system is free from contaminants. If the method blank indicates that contamination may be present in the analytical system, it may be necessary to analyze a solvent blank to demonstrate the source of the contamination is not carryover from standards or lingering field sample artifacts.

8.5 GC/MS analysis of field samples and preparation QC.

8.5.1 It is highly recommended that the extracts be screened on a GC/FID or GC/PID using the same type of capillary column used in the GC/MS system. This will minimize contamination of the GC/MS system from unexpectedly high concentrations of organic compounds.

8.5.2 Allow the extracts to warm to room temperature. Just prior to analysis, add 10µL of the internal standard solution to the 1mL concentrated extract or 5µL of the internal standard solution to the 0.5mL extract obtained from sample preparation.

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8.5.3 The extracts are injected into the GC/MS system via autosampler, using the same operating conditions that were used for the calibration. The volume to be injected should contain 10µg/mL of base/neutral and 20µg/mL of acid surrogates (assuming 100% recovery). If SIM analysis is performed, the volume injected should result in 1ug/mL of the base/neutral analytes (SIM is not performed for acid compounds) and for 1L or soil PAHMO/DROMO analysis, the resulting concentration injected should be 50ug/mL. For DROMO using EPA 3511 (LVI) the resulting concentration injected should be 10ug/mL.

8.5.4 If the response for any quantitation ion exceeds highest level of the initial calibration range, the extract must be diluted and re-analyzed. Additional internal standard must be added to the diluted extract to maintain the same concentration as in the calibration standards (0.04, 0.16 or 8ng/uL, unless a more sensitive GC/MS system is being used). For example, if performing a 1:10 dilution on a concentrated extract, take 100uL of the extract and dilute to a volume of 1mL with the appropriate solvent. Add 9uL of the appropriate internal standard solution to the diluted extract and inject on the analytical system. It can be assumed that 1uL of internal standard was contained in the 100uL extract used for the initial dilution.

8.5.5 Internal standard area counts and retention times must be monitored in all samples, spikes and method blanks to monitor system performance, check for drifting, ensure effective autosampler performance, etc. If the area of the Extracted Ion Current Profile (EICP) changes by a factor of 2 (-50% to +100%) from the areas in the daily CCV, corrective action is required. The RRT of the internal standard in the extract must be within ± 0.06RRT units of the RRT of the daily CCV.

STATE NOTE: With each new calibration curve, a reporting limit verification (RLV) standard must be analyzed for samples analyzed from Minnesota. This standard consists of either re-injecting the low calibration standard(s) or re-processing the low standard(s) utilized in the construction of the calibration curve. The RLV must recover within ±40% of the expected concentration. See section 11.10 for additional information.

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8.6 Qualitative Identification

8.6.1 The qualitative identification of compounds determined by this method is based on retention time and on comparison of the sample mass spectrum, after background correction, with characteristic ions in a reference mass spectrum. The reference mass spectrum must be generated by the laboratory using the conditions of this method. The characteristic ions from the reference mass spectrum are defined as 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. Retention time windows for internal standards and target compounds integrations are updated with each calibration curve and after any instrument maintenance occurs that causes a shift that may affect ChemStation integrations. Retention time windows are set using either method defined limits or by analytical judgment in order to provide the best chance for the software to routinely perform a proper automated integration for the compound. Compounds are identified when the following criteria are met.

8.6.1.1 The intensities of the characteristic ions of a compound must maximize in the same scan or within one scan of each other. Selection of a peak by a data system target compound search routine where the search is based on the presence of a target chromatographic peak containing ions specific for the target compound at a compound-specific retention time will be accepted as meeting this criterion.

8.6.1.2 The RRT of the sample component is within $\pm$ 0.06RRT units of the RRT of the standard component.

8.6.1.3 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%.) Analyst experience is vital in this determination when interferences are present.

8.6.1.4 Structural isomers that produce very similar mass spectra should be identified as individual isomers if they have sufficiently different GC retention times. Sufficient GC resolution is achieved if the height of the valley between the two isomers is <50% of the average of the two peak heights (for Method 8270D) and <25% of the sum of the two peak heights (for Methods 8270C & 625). Otherwise, structural isomers are identified as isomeric pairs.

8.6.1.5 Identification is hampered when sample components are not resolved chromatographically and produce mass spectra containing ions contributed by more than one analyte. When gas chromatographic peaks obviously represent more than one sample component (i.e., a broadened peak with shoulder(s) or a valley between two or more maxima), appropriate selection of analyte spectra and background spectra is important.

8.6.1.6 Examination of extracted ion current profiles of appropriate ions can aid in the selection of spectra, and in qualitative identification of compounds. When analytes co-elute (i.e., only one chromatographic peak is apparent), the identification criteria can be met, but each analyte spectrum will contain extraneous ions contributed by the co-eluting compound.

8.7 TICs – Tentatively Identified Compounds

Periodically, clients may request the tentative identification of compounds present in the field sample that are not normal target compounds and are not normally calibrated. This identification is limited to the compounds in the current NBS (National Bureau of Standards) mass spectral library employed by ESC.

Library Search Identification – For samples containing components not associated with the calibration standards, a library search may be made for the purpose of a tentative identification. Data system library searches must not use normalization routines that would misrepresent the library or unknown spectra when making comparisons. For example, the RCRA permit or waste delisting requirements may require the reporting of non-target analytes. The analyst may only assign tentative identifications after visual comparison of sample spectra with the nearest library searches.

Guidelines for tentative identification are:

- Relative intensities of major ions in the reference spectrum (ions >10% of the most abundant ion) should be present in the sample spectrum.
- 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%.)
- Molecular ions present in the reference spectrum should be present in the sample spectrum.

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- 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.

- 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.

Routinely, ESC employs a minimum Q value of 60 for tentative identifications and a minimum concentration of 10ppb. Peaks below those criteria could result from baseline noise or other interferences, not necessarily attributable to the field sample or reliably quantifiable using GCMS technology.

8.8 Quantitative analysis

8.8.1 Once a compound has been identified, the quantitation of that compound will be based on the integrated abundance of the primary characteristic ion from the EICP.

8.8.1.1 It is recommended to use the integrations produced by the software if the integration is correct because the software will produce more consistent integrations of peaks in chromatograms. Manual integrations may be necessary in some cases and must be performed in conjunction with ESC SOP #030215, *Manual Integration*.

8.8.2 If the RSD of a compound's response factor meets method requirements, then the concentration in the extract may be determined using the average response factor (average RF) from initial calibration data.

8.8.3 Where applicable, the concentration of any tentatively identified compounds in the sample should be estimated. The same formula as is used to calculate target analyte concentrations is used with the following modifications: The areas A_x and A_{is} must be from the total ion chromatograms and the RF for the compound is assumed at 1. See section 9.7 for calculation.

8.8.4 The resulting concentration must be reported indicating that the value is an estimate. Use the nearest internal standard free of interferences for estimated concentration calculations.

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8.8.5 Quantitation of multi-component compounds (e.g., Toxaphene, Aroclors, etc.) is beyond the scope of Method 8270. Normally, quantitation is performed using a GC/ECD, by Methods 8081or 8082. However, Method 8270 may be used to confirm the identification of these compounds, when the concentrations are at least 10ng/µL in the concentrated sample extract.

8.8.6 **Peak Resolution:** Structural isomers that produce very similar spectra must be quantitated as individual isomers if they have sufficiently different GC retention times. Sufficient GC resolution is achieved if the height of the valley between the two isomers is <50% of the average of the two peak heights (for Method 8270D) and <25% of the sum of the two peak heights (for Methods 8270C & 625). Otherwise, structural isomers should be identified as isomeric pairs.

STATE NOTE: Minnesota MPCA requires that peak resolution of all co-eluters, analyzed using Method 8270C, must be resolved as close to 75% as possible. Resolution must be adequate at lower levels and not worsen as concentration increases.

9.0 DATA ANALYSIS AND CALCULATIONS

9.1 GC/MS Tune: DDT Breakdown Determination during Tuning:

$$\%\ \text{breakdown of DDT} = \frac{\text{sum of degradation peak areas (DDD + DDE)}}{\text{sum of all peak areas (DDT + DDE + DDD)}} \times 100$$

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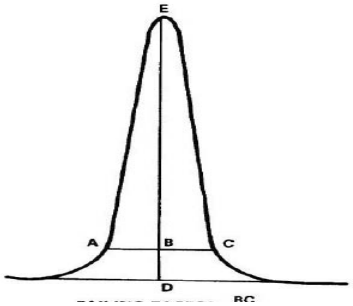
9.2 GC/MS Tune: Benzidine and Pentachlorophenol Tailing Factor

$$\text{TailingFactor} = \frac{BC}{AB}$$



where: BC is the width of the back ½ of the peak at 10% of the peak height
AB is the width of the front ½ of the peak at 10% of the peak height.

9.3 Internal Calibration Equations (Response Factors):

$$RF = \frac{[A_s][C_{is}]}{[A_{is}][C_s]}$$

where:
A_s = Peak area (or height) of the analyte or surrogate.
A_{is} = Peak area (or height) of the internal standard.
C_s = Concentration of the analyte or surrogate, in µg/L.
C_{is} = Concentration of the internal standard, in µg/L.

- Percent Relative Standard Deviation (%RSD)

$$\overline{RF} = \frac{\sum_{i=1}^{n} RF_i}{n} \qquad SD = \sqrt{\frac{\sum_{i=1}^{n} (RF_i - \overline{RF})^2}{n-1}} \qquad RSD = \frac{SD}{\overline{RF}} x100\%$$

where:
RSD = Relative standard deviation.
$\overline{RF}$ = Mean of 5 initial RFs for a compound.
SD = Standard deviation of average RFs for a compound.

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- Concentration of an analyte in an extract using RF:

$$Conc_{.analyte} = \frac{(Conc_{.IStd})(Area_{Analyte})}{(Average\ RF_{analyte})(Area_{IStd})}$$

9.4 Linear calibration model:

$$y = mx + b$$

where: y = Response A_X for External Standard
x = Concentration C_X for External Standard
m = Slope
b = Intercept

- Slope (m):

$$m = \frac{[(Swx_i y_i * Sw) - (Swx_i * Swy_i)]}{[(Sw * Swx_i^2) - (Swx_i * Swx_i)]}$$

- Intercept (b):

$$b = y_{AVE} - (m * (x_{AVE}))$$

- Correlation Coefficient (r):

$$r = \frac{[(Sw * Swx_i y_i) - (Swx_i * Swy_i)]}{\sqrt{\{[(Sw * Swx_i^2) - (Swx * Swx_i)] * [(Sw * Swy_i^2) - (Swy_i * Swy_i)]\}}}$$

- Coefficient of Determination (r^2):

$$r^2 = r * r$$

Where: n = number of x, y pairs
x_i = individual values for the independent variable
y_i = individual values for the dependent variable
w = weighting factor, for equal or no weighting w = 1
x_{AVE} = average of the x values
y_{AVE} = average of the y values
S = the sum of all the individual values

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9.5 The quadratic calibration fit is performed using the following equation:

$$y = ax^2 + bx + c$$

where: y = Response A_x for External Standard or A_x/A_{is} for Internal Standard
x = Concentration C_x for External Standard or C_x/C_{is} for Internal Standard

- Coefficients (a,b,c)

$$a = \frac{\{[S(x^2y) * S(xx)] - [S(xy) * S(xx^2)]\}}{\{[(S(xx) * S(x^2x^2)] - [S(xx^2)]^2\}}$$

$$b = \frac{\{[S(xy) * S(x^2x^2)] - [S(x^2y) * S(xx^2)]\}}{\{[S(xx) * S(x^2x^2)] - [S(xx^2)]^2\}}$$

$$c = [(Sy_i)/n] - \{b * [(Sx_i)/n)]\} - \{a * [S(x_i^2)/n]\}$$

where: $S(xx) = (Sx_i^2) - [(Sx_i)^2/n]$
$S(xy) = (Sx_iy_i) - [(Sx_i)*(Sy_i)/n]$
$S(xx^2) = (Sx_i^3) - [(Sx_i)*(Sx_i^2)/n]$
$S(x^2y) = (Sx_i^2y_i) - [(Sx_i^2)*(Sy_i)/n]$
$S(x^2x^2) = (Sx_i^4) - [(Sx_i^2)^2/n]$

- Coefficient of Determination (r^2)

$$r^2 = \frac{[S(y_i-y_{AVE})^2] - \{[(n-1)/(n-p)] * [S(y_i-Y_i)^2]\}}{S(y_i-y_{AVE})^2}$$

where: y_i = individual values for each dependent variable
x_i = individual values for each independent variable
y_{AVE} = average of the y values
n = number of pairs of data
p = number of parameters in the polynomial equation (i.e., 3 for third order, 2 for second order)
$Y_i = \{[(2a*(C_x/C_{is})^2] - b^2 + b + (4ac)\}/(4a)$
S = the sum of all the individual values

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- Quadratic Regression Equations for Analyte Concentration:

$$C_x = \frac{-b \pm \sqrt{(b^2 - 4a(c - A_x))}}{2a}$$

9.6 Continuing Calibration:

- Percent Difference:

$$\% \text{ Difference} = \frac{RF_v - \overline{RF}}{\overline{RF}} \times 100$$

- Percent Drift:

$$\% \text{ Drift} = \frac{\text{Calculated concentration} - \text{Theoretical concentration}}{\text{Theoretical concentration}} \times 100$$

9.7 Relative Retention Time (RRT):

$$RRT = \frac{\text{Retention Time of the Analyte}}{\text{Retention Time of the ISTD}}$$

9.8 To calculate the concentration of the analyte in the original samples:

- Water:

$$(\text{Concentration of analyte extract}) X \frac{(\text{volume of extract})}{(\text{volume of sample})}$$

- Soil:

$$(\text{Concentration of analyte extract}) X \frac{(\text{volume of extract})}{(\text{weight of sample})}$$

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9.9 **(Soil Samples only)** Dry weight target analyte corrections (performed automatically by the laboratory LIMS when required by project/client/regulatory program). See ESC SOP# 060111, *Total Solids*:

- Percent Dry Weight

$$\%DryWeight = \frac{g\ of\ dry\ sample}{g\ of\ sample} X100\%$$

- Target Analyte Concentration Correction:

$$Concentration\ of\ Target\ Analyte = \frac{Concentration\ of\ Analyte\ from\ Instrument}{\%\ Dry\ Weight} X\ 100\%$$

9.10 LCS/LCSD/SSCV Percent Recovery (%R):

$$\%R = \frac{Measured\ concentration}{Actual\ concentration} x\ 100$$

9.11 Matrix Spike Recoveries ($\%R_{MS/MSD}$):

$$\%R_{MS/MSD} = \frac{O_i - O_s}{T_i} X100$$

where: O_i = observed sample concentration with the spike added
O_s = the observed value for the sample without the spike
T_i = True value of the spike added

9.12 Relative Percent Difference (%RPD):

$$RPD = \frac{Value\ 1 - Value\ 2}{\left(\frac{Value\ 1 + Value\ 2}{2}\right)} \times 100$$

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9.13 External Standard Calibration (see section 13.1): The calibration factor for each standard can be calculated:

$$CF = \frac{A_s}{C_s}$$

where: A_s - Average Peak Area over the number of peaks used for quantitation
C_s – Concentration of the analyte in the standard.

- The average (or mean) calibration factor ($\overline{CF}$) is calculated:

$$\overline{CF} = \frac{\sum_{i=1}^{n} CF_i}{n}$$

where: CF_i – Calibration Factor for each level of the calibration curve
n – number of standards analyzed in the calibration curve

- The standard deviation (SD) of the calibration is determined:

$$SD = \sqrt{\frac{\sum_{i=1}^{n} (CF_i - \overline{CF})^2}{n-1}}$$

where: $\overline{CF}$ – Average Calibration Factor for the calibration curve
CF_i – Calibration Factor for each level of the calibration curve
n – number of standards analyzed in the calibration curve

- The Percent Relative Standard Deviation for each analyte in the curve is determined:

$$RSD = \frac{SD}{\overline{CF}} \times 100$$

where: SD – Standard Deviation for each analyte
$\overline{CF}$ – Average calibration factor for the specific analyte

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- Percent Difference for daily calibration curve verification:

$$\% \text{ Difference} = \frac{\overline{CF} - CF_v}{\overline{CF}} \times 10($$

where: $\underline{CF_v}$ – Calibration Factor from the calibration verification standard
$\overline{CF}$ – Average (or mean) calibration factor from the initial calibration curve

9.14 Relative Retention Time (RRT):

$$RRT = \frac{RT\ of\ Target\ Analyte}{RT\ of\ Internal\ Standard}$$

10.0 QUALITY CONTROL AND METHOD PERFORMANCE

10.1 All analysts must meet the qualifications specified in SOP 030205, *Technical Training and Personnel Qualifications* before approval to perform this method. Analysts must complete an initial demonstration of proficiency before being approved to perform this method. Continuing proficiency must be demonstrated using proficiency testing, laboratory control sample analysis and/or MDL studies. Method performance is assessed per analyst. Updated method performance records are filed and stored in a central location within the department.

10.2 Use the designated Run log to record batch order and standards/reagents used during analysis. See SOP 030201, *Data Handling and Reporting*.

10.3 Batches:

Batches are defined as sets of 1 - 20 samples. Batch analysis must include the following: 1 Instrument Tune, 1 method blank, 1 Laboratory Control Sample (LCS), 1 Laboratory Control Sample Duplicate (LCSD), 1 Second Source Verification (SSCV), 1 Matrix Spike/Spike Duplicate (MS/MSD). All batch information must be maintained in the preparation documentation assigned to the department.

10.4 For acceptance criteria for calibration standards, QC samples and field samples and corrective actions, see section 11.0.

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11.0 DATA VALIDATION AND CORRECTIVE ACTION

11.1 A successful DFTPP tune must be achieved prior to initial calibration or daily calibration verification. If a tune does not meet the acceptance criteria in section 8.2, then re-inject the tuning solution. If the failure persists, instrument maintenance or detector adjustment is required. The instrument is equipped with detector adjustments in routines called "Autotunes" that can make minor adjustments to m/z ratios and detector setting and can align the analytical system to return the system to peak performance. If after performing the Autotune routine, the injected tuning standard still fails, the system may require injector and/or detector cleaning, column cutting or replacement, injection liner cleaning or replacement, or other maintenance as specified by the manufacturer.

Following successful tuning of the DFTPP solution, the DDT degradation and Benzidine/Pentachlorophenol tailing must be assessed. If either fail to meet the required acceptance criteria, instrument maintenance is required. The DDT degradation is most likely an inlet or column condition and corrective action entails clipping 6-12" from the injector end of the column, changing the injection port liner, possibly changing the gold inlet seal and re-injecting the tuning solution. The tailing issue is most likely caused by the same type of inlet issues and the same corrective action steps should occur when the tailing criteria is not met. Tailing may also be caused by incorrect column positioning in the inlet and the correct position of the column should be verified prior to performing more involved corrective action processes.

A successful instrument tune, including degradation and tailing acceptability, must be achieved prior to the analysis of calibration standards and sample extracts.

11.2 Initial or Continuing Calibration:

Method 8270C, SM 6410B & Method 625: If the calibration curve or daily calibration verification fails to meet the applicable method verification criteria for RSD, the analyst MUST use linear regression or quadratic curve fit. Quadratic curve modeling is not permitted unless historical performance of analytes exhibited a nonlinear response. Quadratic models cannot be used to extend the calibration range or bypass instrument maintenance. If the method criteria is still not met when using the alternate curve fits, samples may not be quantitated using the calibration curve and a new calibration curve must be analyzed. Instrument maintenance and/or new standard preparation may also be required prior to the analysis of the new calibration curve. Following maintenance, the new calibration curve can be generated. The system may require injector and/or detector cleaning, column cutting or replacement, injection liner cleaning or replacement, or other maintenance as specified by the manufacturer. Additional actions that can be taken to address failures in calibration are included in section 8.3.

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Method 8270D: Due to the large number of compounds that may be analyzed by this method, some compounds in the initial and/or daily calibration verification will fail to meet the initial and continuing calibration acceptance criteria. For these instances, failing compounds may not be critical to specific project needs and therefore may be utilized as qualified data or estimated values for screening purposes. If more than 10% of the compounds in the initial or continuing calibration exceed the 20% RSD limit and/or do not meet the minimum correlation coefficient (0.990) for alternate curve fits, then the chromatographic system is considered too reactive for analysis. Instrument maintenance must be performed and the calibration process must be repeated. The system may require injector and/or detector cleaning, column cutting or replacement, injection liner cleaning or replacement, or other maintenance as specified by the manufacturer. Additional actions that can be taken to address failures in calibration are included in section 8.3.

11.3 The method blank must be extracted and analyzed with each set of samples and must be free of the analytes of interest at ½ the level being reported (RL). If the method blank contains target analytes at a concentration >½ the RL, it may be necessary to analyze a solvent blank to demonstrate the source of the contamination is not carryover from standards or lingering field sample artifacts. Following verification that the analytical system is free from interferences, the method blank can be re-analyzed once. If the method blank continues to demonstrate target analytes above the ½ criteria, then corrective action is required. Corrective action can take the form of checking the original calculations to ensure accuracy or instrument maintenance (i.e. column clipping or changing, inlet liner cleaning/replacement, etc.) or re-calibration. The surrogate recoveries in the method blank must meet the established control criteria, listed in Attachments IVa & IVb. If not, the recovery demonstrates an analytical system that is in an out-of-control mode and the batch must be re-extracted/re-analysis unless directed otherwise by the client.

11.4 **Method 8270D:** The value determined from the second source calibration verification (SSCV) should be within ±30% of the expected concentration. Alternative recovery limits may be appropriate based on analyte performance and project specific requirements. Quantitative analysis cannot proceed for analytes that fail the SSCV, except for screening purposes only.

Method 8270C/625/SM6410B: The value determined from the second source calibration verification (SSCV) must be ≤50% for non-CCC compounds; ≤20% for CCC compounds and meet the minimum response factor criteria for SPCC compounds as in the initial calibration construction. Historical performance weighs heavily in the acceptability of those analytes that are known to perform poorly. Corrective action can take the form of checking the original calculations to ensure accuracy, re-analysis of the SSCV to verify initial results, instrument maintenance (i.e. column clipping or changing, inlet liner cleaning/replacement, etc.) or re-calibration.

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11.5 Surrogates: If the surrogate recoveries in the samples do not fall within the established limits (listed in Attachments IVa & IVb), ensure that there were no errors in calculations, internal standard, or instrument performance. If no errors were found, re-extract the sample, unless directed otherwise by the client. If there is no more sample available or it has exceeded holding times, the data must be flagged with a "J1" (surrogate high) or a "J2" (surrogate low). See SOP #030201, *Data Handling and Reporting*, for more information on qualifying out of control data.

STATE NOTE: If the sample is from North Carolina, two of the three acid and two of the three base/neutral surrogates must pass. If two of the three acid or base/neutral surrogates fail, the sample must be re-extracted. For all other samples, one of the three surrogates must pass from both the acid and base/neutral sides. If any surrogates have less than a 10% recovery, the sample must be re-extracted.

STATE NOTE: If field samples are analyzed in conjunction with the Ohio VAP program, surrogate outliers in batch QC samples, including the method blank, LCS/LCSD, MS/MSD require re-extraction of the entire batch, if sufficient volume has been submitted by the client and an obvious matrix interferent is not present.

STATE NOTE: If the sample is analyzed in conjunction with the Ohio VAP, corrective action for failing QC (i.e. method blank, surrogate, MS/MSD, LCS/LCSD, ISTD, etc.) must be performed prior to flagging data, if sufficient sample volume was submitted by the client. Corrective action can include re-analysis, if instrument malfunction is suspected, or re-preparation and re-analysis, if the failure is suspected as either extraction or sample related.

11.6 Internal Standard: The internal standard area counts must be monitored for all ICVs. ISTDs must recover within –50% to +100% of the area counts from the internal standard area counts of the midpoint standard of the most recent initial calibration sequence. If any internal standard response is beyond the acceptable recovery, corrective action is required. Corrective action can take the form of checking the original calculations to ensure accuracy, re-analysis of the ICV to verify initial results, instrument maintenance (i.e. column clipping or changing, inlet liner cleaning/replacement, etc.) or re-calibration.

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The internal standard responses and retention times in the check calibration standard must be evaluated immediately after or during data acquisition. If the retention time for any internal standard changes by more than 30 seconds from the last calibration verification, the chromatographic system must be inspected for malfunctions and corrections must be made, as required. When corrections are made, re-analysis of the CCV or a complete re-calibration is necessary, depending on the impact of the correction on the analytical system.

Internal standards must be monitored for each sample. ISTDs in samples must meet the –50% to +100% criteria when compared to the ISTDs in the daily CCV or mid-level of the calibration curve, on 12h shifts when full calibration is performed. Possible corrective actions include: re-analysis, if instrument malfunction is suspected, or re-preparation and re-analysis, if the failure is suspected as either extraction or sample related. If the sample has an obvious matrix interferent and the internal standard recovery is greater than +100%, the sample can be diluted (if acceptable reporting limits can be achieved) to minimize the interference or the sample must be re-extracted and re-analyzed to confirm the original results.

11.7 LCS/LCSD and MS/MSD: The laboratory control sample, laboratory control sample duplicate, matrix spike and matrix spike duplicate recoveries must be evaluated against the limits listed in Attachments IVa & IVb. The LCS/LCSD and MS/MSD are spiked with the same list of compounds for which the instrument is calibrated. Due to the large number of compounds analyzed using these methods, it is statistically likely that accuracy and precision failures will occur.

LCS or LCSD samples that do not pass the acceptable QC criteria must be re-analyzed. LCS/LCSD failures must meet the marginal excedence criteria below. The normal compound list for 8270/625 typically contains 90 analytes; therefore only 5 analytes can be considered as marginally exceeding the acceptance criteria. If more than 5 failures occur or if the failures demonstrate a pattern that is causing the outliers, the entire sample batch with associated QC must be re-extracted and re-analyzed. Marginal excedences must be random events.

Upper and lower marginal excedence (ME) limits are established by +/- four times the standard deviation of historical accuracy data and the number of marginal excedences allowed is based on the number of analytes spiked in the LCS.

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Number of allowable marginal excedences:

90 analytes, 5 analytes allowed in the ME limit
71 – 90 analytes, 4 analytes allowed in the ME limit.
51 – 70 analytes, 3 analytes allowed in the ME limit.
31 – 50 analytes, 2 analytes allowed in the ME limit.
11 – 30 analytes, 1 analyte allowed in the ME limit.
< 11 analytes, no analyte allowed in the ME limit.

If the MS/MSD fails to meet recovery limits listed in Attachment II, the data on the unspiked field sample for that compound must be flagged with a "J5" (high recovery) or a "J6" (low recovery). If the MS/MSD fail to pass precision limits (%RSD), the data on the unspiked field sample for that compound must be flagged with a "J3" qualifier.

STATE NOTE: For South Carolina and Ohio VAP samples, marginal excedences do not apply. All outliers in QC require corrective action when possible and the data must be flagged when necessary.

STATE NOTE: For all samples from South Carolina, the LCS/LCSD recovery must be within 70-130% for both SC matrices with an RPD of 20%.

11.8 Calibration Range: For any compound found in a sample at a level above the highest standard, the extract must be diluted and re-analyzed to allow quantitation within the range of instrument calibration. Whenever an extract dilution is made, the appropriate amount of internal standard must be added to bring the ISTD concentrations back to the original amount added prior to the dilution.

11.9 All data must undergo a primary review by the analyst. The analyst must check the performance of the initial calibration, check standard, and continuing calibrations to ensure that they meet the criteria of the method. The analyst must review any sample that has quantifiable compounds and make sure that they have been confirmed, if necessary. The analyst must also verify that reported results are derived from quantitation between the RL and the highest standard of the initial calibration curve. All calculations must be checked (any dilutions, %solids, etc.). Data must be checked for the presence or absence of appropriate flags. Comments must be noted when data is flagged.

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11.10 All data must undergo a second analyst review. The analyst checking the data must check the performance of the initial calibration, mid-point check standard, and continuing calibrations to ensure that they meet the criteria of the method.

11.10.1 The analyst should must review any sample that has quantifiable compounds and make sure that they have been confirmed.

11.10.2 All calculations must be checked.

11.10.3 All surrogate recoveries must be checked to ensure that they are within QC acceptance criteria or that corrective action has occurred.

11.10.4 Blanks must be free of all interfering peaks.

11.10.5 Quality control criteria must be checked for the LCS, LCSD, MS, and MSD.

11.10.6 Data must be checked to determine the need for appropriate flags. Comments are noted when results are flagged.

11.10.7 The reviewer must verify all reported results are derived from analytical results that are either above the reporting limit/MDL, as applicable, and below the highest standard of the initial calibration curve.

11.10.8 All manual integrations must be verified through checking the before/after shot of the sample, method blank, and/or QC (LCS/LCSD/MS/MSD).

11.10.9 All multipliers/dilutions must be verified on the quant report and must agree with the information provided on the injection log.

11.10.10 Retention times of the samples must be compared to that of the calibration standard. Random spot checking of 10% of the data should be sufficient.

11.10.11 Verify linear regression by reviewing the calibration curve printout.

11.10.12 See SOP #030201, *Data Handling and Reporting* and SOP #030227*, Data Review*.

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STATE NOTE: For all samples analyzed from Minnesota, the reporting limit must be verified at least monthly, with each new initial calibration, or when there has been significant change to the instrument (column replacement, cleaning source, etc.) whichever is more frequent. The reporting limit verification can be performed by either re-injecting the low standard or by re-processing the low standard that was analyzed in the calibration curve. The reporting limit verification (RLV) must recovery within ±40% of the expected concentration. If this criteria is not met, the RLV may be re-analyzed once, instrument maintenance can be performed, a higher concentration standard can be injected, or a new calibration curve must be generated. If a higher concentration standard is utilized, the reporting limit must be raised to the higher level verified.

11.11 Data that does not meet acceptable QC criteria may be acceptable for use in certain circumstances.

11.11.1 If a method blank contains an amount of target analyte, but all samples are non-detected, the data may be reported with a “B3” flag. If a method blank contains an amount of target analyte, but the samples contain analyte at a level that is 10 times the level present in the method blanks, the data may be reported with a “B” flag.

STATE NOTE: The Ohio VAP program does not accept data released using the 10X criteria for method blank contamination as noted in section 11.11.1.

11.11.2 If the MS/MSD fails (recovery less than 30% or greater than 150% and/or RPD greater than 30%) in an initial analysis and again upon re-analysis, the data is released with an appropriate qualifier as the failure is accepted as matrix related.

11.11.3 If a calibration verification standard is above the acceptable QC criteria and all samples being bracketed are below the reporting limit, the data is acceptable based on a high calibration bias with undetectable levels in the field samples. Any positive samples require re-analysis. . If MDL reporting is required by the client, reported samples must calculate <MDL to be considered not impacted by the high bias.

11.11.4 If the surrogate exhibits high recovery in the field samples and the target analytes in the field samples are below the reporting limit, the data may be released with a J1 qualifier indicating the high bias. If the QC samples (LCS, LCSD, MS, MSD) exhibit a high bias in the surrogate and the field samples are below the reporting limit for the target analyte, the data may be released with a J1 qualifier.

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11.11.5 If the target analyte spiked in the quality control samples (LCS, LCSD, MS, MSD) exhibits high recovery and the target analytes in the field samples are below the reporting limit, the data may be released with a J4 qualifier indicating the high bias.

11.11.6 If the target analyte spiked into the QC pair (LCS/LCSD, MS/MSD) exhibit acceptable recoveries, but high calculated RPD values for precision, target analytes in the field sample are flagged with a J3 for the precision beyond acceptable quality control limits.

11.11.7 Sample results can be qualified and possible bias is narrated per the ESC SOP# 030201, *Data Handling*.

12.0 POLLUTION PREVENTION AND WASTE MANAGEMENT

12.1 The EPA requires that laboratory waste management practice to be conducted consistent with all applicable federal and state laws and regulations. Excess reagents, samples and method process wastes must be characterized and disposed of in an acceptable manner. See *ESC Waste Management Plan.*

12.2 See SOP #030302, *Pollution Prevention*.

13.0 METHOD MODIFICATIONS/CLARIFICATIONS

13.1 The **Missouri Department of Natural Resources requires** that **DRO** be analyzed by GC/MS. Tuning and frequency requirements are the same as 8270C, omitting DDT, pentachlorophenol, and benzidine assessments. Extract samples the same as 8270PAH using the appropriate extraction method. Only base/neutral surrogates are needed. GC/MS mass range should be 35-550amu. Prepare a five-point calibration curve with 1:1 unleaded gasoline and #2 diesel fuel at 10,000 µg/mL each in methylene chloride. Calibration standards range from 200 to 10,000ug/mL for soil or 1L water extractions and calibration levels for EPA 3511 extracted water samples range from 5-200ppm from a 200ppm intermediate. Retention time windows are set using C_{10}, C_{21}, and C_{35}. For DRO, set RT 0.1 minutes after C_{10} to 0.1 minutes after C_{21}. For ORO, set RT 0.1 minutes after C_{21} to 0.1 minutes after C_{35}. Verify RT windows daily (24 hours) by running component standard. Quantitate using baseline-to baseline, not valley–to-valley. The total ion chromatogram must be used to quantitate. DRO is quantitated using external standard method. The response factor determined for DRO (C_{10}-C_{21}) **must** be used for C_{21}-C_{35}. Subtract area for any internal standard and surrogates. %RSD <20. Run a CCV every 12 hours near mid-point of calibration, %D <20. Run a method blank, LCS and MS/MSD every extraction batch. May re-process files to quantitate PAH analytes, if needed. Quantitation of DRO must be performed using the external standard process.

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13.2 EPA method 625 employs the use of 2 separate packed GC columns for base/neutral and acidic analyte separations. Modern capillary column technology employs a single column that provides sufficient separatory abilities for use in this analytical process as is demonstrated in EPA method 8270C.

13.3 Modifications to this method are noted in the body of the text as state notes. Compliance analyses performed in conjunction with specific state requirements must be performed as noted within the specific state(s) note listed.

13.4 Adjustments to the concentrations of standards/spiking solutions, standards providers, and quality control samples are subject to change to better meet client/project/regulatory needs or to improve laboratory method performance.

13.5 The reduction of the size of the field sample used in this procedure is performed in accordance with section 7.1 of the published EPA 3510C method. The reduction in volume extracted along with analysis of the resulting extract using large volume injection (up to 250uL can be injected with the LVI injection port) on each GCMS allows for low detection limits in line with those obtained using a 1L extraction and the 1-2uL injection. Complete method validation is performed for each method prior to utilizing the reduced volume extraction. This validation is maintained by the Regulatory Affairs Department and is regularly verified using LCS/LCSD, MDL studies and DOCs.

STATE NOTE: The reduced volume extraction method using EPA 3510C is not approved for use with samples analyzed in conjunction with South Carolina DHEC.

14.0 REFERENCES

14.1 SW846, Method 8270C, *SVOC by GC/MS*, Rev. 3, December 1996.

14.2 SW846, Method 8270D, *SVOC by GC/MS*, Rev. 4, February 2007.

14.3 SW846, Method 8000B, *Determinative Chromatographic Separations*, Rev. 2, December 1996.

14.4 SW846, Method 8000C, *Determinative Chromatographic Separations*, Rev. 3, March 2003.

14.5 40 CFR Part 136, EPA Method 625, October 1991.

14.6 Standard Methods for the Examination of Water and Wastewater, APHA, 20th edition, *Method 6410B*.

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Attachment I: Revision History

Current Version:

Version	Date	Description of Revisions
15	6/12/12	Technical and Quality Review and update. Revised sections 2.1, 7.9, 7.10, 8.3.2, 9.9, 9.14, 11.2, 11.3, 11.4, and 11.11; Added sections 2.19, 2.34, 4.8, 11.9 through 11.10, and 11.11.4 through 11.11.7.

Superseded Versions:

This document supersedes the following:

Version	Date	Description of Revisions
0	4/27/95	Origination
1	7/13/95	
2	8/22/96	
3	8/20/99	
4	4/18/00	
5	8/21/00	
6	12/20/00	
7	9/3/01	
8	7/30/02	
9	7/9/03	
10	3/25/04	
11	8/7/06	Technical and Quality Review and update.
12	2/11/09	Addition of 8270D requirements; Addition of State Notes; Update of standards information; Technical and Quality Review and update. Ohio VAP approval 2/11/09.
13	11/23/10	Technical and Quality Review and update. Revised sections 2.1, 2.10, 4.2 through 4.6, 7.1, 7.6, 7.8, 7.10.2, 7.12, 7.13, 8.3, 8.6, 9.3, 9.4, 9.5, 9.10 through 9.13, 11.3, 11.6, 12.1; Added sections 2.27 through 2.30, 4.7, 7.14, state note following section 11.9, 11.10, and 13.4; Removed section 1.2.
14	2/24/12	Technical and Quality Review and update. Revised sections 2.1, 4.2, 4.5, 5.3, 6.1.4, 6.2, 7.8, 7.9, 7.10, 7.13, 8.2, 8.3, 8.5, 8.6, 8.8, 11.2, 11.10, 13.1 and Attachment IV; Added state note to section 1.0; Added sections 1.8.1, 2.31, 2.32, and 13.5.

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Attachment II: 8270/625 Common Calibration List & Reporting Limits *(may be updated without notice)*

Analyte	Water mg/L	Soil mg/Kg
Acenaphthene	0.001	0.033
Acenaphthylene	0.001	0.033
Acetophenone	0.01	0.33
Anthracene	0.001	0.033
Atrazine	0.01	0.33
Benzaldehyde	0.01	0.33
Benzidine	0.05	0.33
Benzo(a)anthracene	0.001	0.033
Benzo(b)fluoranthene	0.001	0.033
Benzo(k)fluoranthene	0.001	0.033
Benzo(g,h,i)perylene	0.001	0.033
Benzo(a)pyrene	0.001	0.033
Bis(2-chlorethoxy)methane	0.01	0.33
Bis(2-chloroethyl)ether	0.01	0.33
Bis(2-chloroisopropyl)ether	0.01	0.33
4-Bromophenyl-phenylether	0.01	0.33
Caprolactam	0.01	0.33
2-Chloronaphthalene	0.01	0.33
4-Chlorophenyl-phenylether	0.01	0.33
Chrysene	0.001	0.033
Dibenz(a,h)anthracene	0.001	0.033
3,3-Dichlorobenzidine	0.01	0.33
2,4-Dinitrotoluene	0.01	0.33
2,6-Dinitrotoluene	0.01	0.33
Fluoranthene	0.001	0.033
Fluorene	0.001	0.033
Hexachlorobenzene	0.01	0.33
Hexachloro-1,3-butadiene	0.01	0.33
Hexachlorocyclopentadiene	0.01	0.33
Hexachloroethane	0.01	0.33
Indeno(1,2,3-cd)pyrene	0.001	0.033

Analyte	Water mg/L	Soil mg/Kg
Isophorone	0.01	0.33
Naphthalene	0.001	0.033
Nitrobenzene	0.01	0.33
n-Nitrosodimethylamine	0.01	0.33
n-Nitrosodiphenylamine	0.01	0.33
n-Nitrosodi-n-propylamine	0.01	0.33
Phenanthrene	0.001	0.033
Benzylbutyl phthalate	0.001	0.033
Bis(2-ethylhexyl)phthalate	0.001	0.033
Di-n-butyl phthalate	0.001	0.033
Diethyl phthalate	0.001	0.033
Dimethyl phthalate	0.001	0.033
Di-n-octyl phthalate	0.001	0.033
Pyrene	0.001	0.033
1,2,4-Trichlorobenzene	0.01	0.33
4-Chloro-3-methylphenol	0.01	0.33
2-Chlorophenol	0.01	0.33
2,4-Dichlorophenol	0.01	0.33
2,4-Dimethylphenol	0.01	0.33
4,6-Dinitro-2-methylphenol	0.01	0.33
2,4-Dinitrophenol	0.01	0.33
2-Methylphenol	0.01	0.33
4-Methylphenol	0.01	0.33
2-Nitrophenol	0.01	0.33
4-Nitrophenol	0.01	0.33
Pentachlorophenol	0.01	0.33
Phenol	0.01	0.33
2,4,6-Trichlorophenol	0.01	0.33
1-Methylnapthalene	0.001	0.033
2-Methylnapthalene	0.001	0.033
4-Chloroaniline	0.01	0.33

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Analyte	Water mg/L	Soil mg/Kg
2-Nitroaniline	0.01	0.33
3-Nitroaniline	0.01	0.33
4-Nitroaniline	0.01	0.33
1,2,3,4-Tetrachlorobenzene	0.05	1.65
1,2,3,5-Tetrachlorobenzene	0.05	1.65
1,2,4,5-Tetrachlorobenzene	0.05	1.65
1,2,4,5-Tetrachlorobenzene	0.05	1.65
1,2-diphenylhydrazine	0.01	0.33
1,3-Dinitrobenzene	0.05	1.65
1,4-Naphthoquinone	0.05	1.65
1-Chloronaphthalene	0.05	1.65
1-Naphthylamine	0.05	1.65
2,3,4,6-Tetrachlorophenol	0.05	1.65
2,3-Dichloroaniline	0.01	0.33
2,6-Dichlorophenol	0.05	1.65
2-Acetylaminofluorene	0.05	1.65
2-Naphthylamine	0.05	1.65
2-Picoline	0.05	1.65
3,3'-Dimethylbenzidine	0.05	1.65
3-Methylcholanthrene	0.05	1.65
4-Aminobiphenyl	0.05	1.65
4-Nitroquinoline-1-oxide	0.05	1.65
5-Nitro-o-toluidine	0.05	1.65
7,12-Dimethylbenz(a)anthracene	0.05	1.65
7H-Dibenzo (c,g) carbazole	0.05	1.65
a,a-Dimethylphenethylamine	0.05	1.65
Acetophenone	0.01	0.33
Alpha-terpineol	0.01	0.33
Aniline	0.01	0.33
Aramite	0.05	1.65
Benzal Chloride	0.05	1.65
Benzo (j) fluoranthene	0.05	1.65

Analyte	Water mg/L	Soil mg/Kg
Benzotrichloride	0.05	1.65
Benzyl Chloride	0.05	1.65
Chlorobenzilate	0.05	1.65
Diallate (cis)	0.05	1.65
Diallate (trans)	0.05	1.65
Dibenz (a,e) pyrene	0.05	1.65
Dibenz (a,h) acridine	0.05	1.65
Dibenz (a,h) pyrene	0.05	1.65
Dibenz (a,i) pyrene	0.05	1.65
Dimethoate	0.05	1.65
Dinoseb	0.05	1.65
Diphenylamine	0.05	1.65
Disulfoton	0.05	1.65
Ethyl methanesulfonate	0.05	1.65
Famphur	0.05	1.65
Hexachlorophene	0.05	1.65
Hexachloropropene	0.05	1.65
Isodrin	0.05	1.65
Isosafrole (cis)	0.05	1.65
Isosafrole (trans)	0.05	1.65
Kepone	0.05	1.65
Methapyriline	0.05	1.65
Methyl methanesulfonate	0.05	1.65
Methyl parathion	0.05	1.65
N-Nitrosodiethylamine	0.05	1.65
n-nitrosodi-n-butylamine	0.01	0.33
N-Nitrosodi-n-butylamine	0.05	1.65
N-Nitrosomethylethylamine	0.05	1.65
N-Nitrosomorpholine	0.05	1.65
N-Nitrosopiperidine	0.05	1.65
N-Nitrosopyrrolidine	0.05	1.65
o,o,o-Triethylphoshorothioate	0.05	1.65

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Analyte	Water mg/L	Soil mg/Kg
o-cresol	0.01	0.33
o-Toluidine	0.05	1.65
Parathion	0.05	1.65
p-cresol	0.01	0.33
p-Dimethylaminoazobenzene	0.05	1.65
Pentachlorobenzene	0.05	1.65
Pentachloroethane	0.05	1.65
Pentachloronitrobenzene	0.05	1.65
Phenacetin	0.05	1.65
Phorate	0.05	1.65
p-Phenyleneamine	0.05	1.65
Pronamide	0.05	1.65
Safrole	0.05	1.65
Sulfotepp	0.05	1.65
sym-Trinitrobenzene	0.05	1.65
Thionazin	0.05	1.65
2-nitrodiphenylamine	0.01	0.33
n-decane	0.01	0.33
n-octadecane	0.01	0.33
Pentachlorphenol (SIM)	0.001	-

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Attachment III – Appropriate Extraction Methods by Analyte *(printed from SW-846 Method 8270C)*

ANALYTE:	3510*	3520	3540/3541	3550*	3580*	CAS #(a)
Acenaphthene	X	X	X	X	X	83-32-9
Acenaphthylene	X	X	X	X	X	208-96-8
Acetophenone	X	ND	ND	ND	X	98-86-2
2-Acetylaminofluorene	X	ND	ND	ND	X	53-96-3
1-Acetyl-2-thiourea	LR	ND	ND	ND	LR	591-08-2
Aldrin	X	X	X	X	X	309-00-2
2-Aminoanthraquinone	X	ND	ND	ND	X	117-79-3
Aminoazobenzene	X	ND	ND	ND	X	60-09-3
4-Aminobiphenyl	X	ND	ND	ND	X	92-67-1
3-Amino-9-ethylcarbazole	X	X	ND	ND	ND	132-32-1
Anilazine	X	ND	ND	ND	X	101-05-3
Aniline	X	X	ND	X	X	62-53-3
Ortho-anisidine	X	ND	ND	ND	X	90-04-0
Anthracene	X	X	X	X	X	120-12-7
Aramite HS	(43)	ND	ND	ND	X	140-57-8
Aroclor 1016	X	X	X	X	X	12674-11-2
Aroclor 1221	X	X	X	X	X	11104-28-2
Aroclor 1232	X	X	X	X	X	11141-16-5
Aroclor 1242	X	X	X	X	X	53469-21-9
Aroclor 1248	X	X	X	X	X	12672-29-6
Aroclor 1254	X	X	X	X	X	11097-69-1
Aroclor 1260	X	X	X	X	X	11096-82-5
Azinphos-methyl HS	(62)	ND	ND	ND	X	86-50-0
Barban	LR	ND	ND	ND	LR	101-27-9
Benzidine	CP	CP	CP	CP	CP	92-87-5
Benzoic Acid	X	X	ND	X	X	65-85-0
Benz(a)anthracene	X	X	X	X	X	56-55-3
Benzo(b)fluoranthene	X	X	X	X	X	205-99-2
Benzo(k)fluoranthene	X	X	X	X	X	207-08-9
Benzo(g,h,i)perylene	X	X	X	X	X	191-24-2
Benzo(a)pyrene	X	X	X	X	X	50-32-8
Para-benzoquinone	OE	ND	ND	ND	X	106-51-4
Benzyl Alcohol	X	X	ND	X	X	100-51-6
Alpha-BHC	X	X	X	X	X	319-84-6
Beta-BHC	X	X	X	X	X	319-85-7
Delta-BHC	X	X	X	X	X	319-86-8
Gamma-BHC	X	X	X	X		58-89-9

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ANALYTE:	3510*	3520	3540/3541	3550*	3580*	CAS #(a)
Lindane	X	X	X	X	X	58-89-9
Bis(2-chloroethoxy)methane	X	X	X	X	X	111-91-1
Bis(2-chloroethyl) Ether	X	X	X	X	X	111-44-4
Bis(2-chloroisopropyl) Ether	X	X	X	X	X	108-60-1
Bis(2-ethylhexyl) Phthalate	X	X	X	X	X	117-81-7
4-Bromophenyl Phenyl Ether	X	X	X	X	X	101-55-3
Bromoxynil	X	ND	ND	ND	X	1689-84-5
Butyl Benzyl Phthalate	X	X	X	X	X	85-68-7
Captafol HS	(55)	ND	ND	ND	X	6/1/2425
Captan HS	(40)	ND	ND	ND	X	133-06-2
Carbaryl	X	ND	ND	ND	X	63-25-2
Carbofuran	X	ND	ND	ND	X	1563-66-2
Carbophenothion	X	ND	ND	ND	X	786-19-6
Chlordane	X	X	X	X	X	57-74-9
Chlorfenvinphos	X	ND	ND	ND	X	470-90-6
4-Chloroaniline	X	ND	ND	ND	X	106-47-8
Chlorobenzilate	X	ND	ND	ND	X	510-15-6
5-Chloro-2-methylaniline	X	ND	ND	ND	X	95-79-4
4-Chloro-3-methylphenol	X	X	X	X	X	59-50-7
hydrochloride	X	ND	ND	ND	X	6959-48-4
1-Chloronaphthalene	X	X	X	X	X	90-13-1
2-Chloronaphthalene	X	X	X	X	X	91-58-7
2-Chlorophenol	X	X	X	X	X	95-57-8
4-Chloro-1,2-phenylenendiamine	X	X	ND	ND	ND	95-83-0
4-Chloro-1,3-phenylenendiamine	X	X	ND	ND	ND	5131-60-2
4-Chlorophenyl Phenyl Ether	X	X	X	X	X	7005-72-3
Chrysene	X	X	X	X	X	218-01-9
Coumaphos	X	ND	ND	ND	X	56-72-4
Para-cresidine	X	ND	ND	ND	X	120-71-8
Crotoxyphos	X	ND	ND	ND	X	7700-17-6
2-Cyclohexyl-4,6-dinitrophenol	X	ND	ND	ND	LR	131-89-5
4,''-DDD	X	X	X	X	X	72-54-8
4,''-DDE	X	X	X	X	X	72-55-9
4,''-DDT	X	X	X	X	X	50-29-3
Demeton-O HS	(68)	ND	ND	ND	X	298-03-3
Demeton-S	X	ND	ND	ND	X	126-75-0
Diallate (cis or trans)	X	ND	ND	ND	X	2303-16-4
2,4-Diaminotoluene DC,	OE(42) ND	ND	ND	ND	X	95-80-7

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ANALYTE:	3510*	3520	3540/3541	3550*	3580*	CAS #(a)
Dibenz(a,j)acridine	X	ND	ND	ND	X	224-42-0
Dibenz(a,h)anthracene	X	X	X	X	X	53-70-3
Dibenzofuran	X	X	ND	X	X	132-64-9
Dibenzo(a,e)pyrene	ND	ND	ND	ND	X	192-65-4
1,2-Dibromo-3-chloropropane	X	X	ND	ND	ND	96-12-8
Di-n-butyl Phthalate	X	X	X	X	X	84-74-2
Dichlone	OE	ND	ND	ND	X	117-80-6
1,2-Dichlorobenzene	X	X	X	X	X	95-50-1
1,3-Dichlorobenzene	X	X	X	X	X	541-73-1
1,4-Dichlorobenzene	X	X	X	X	X	106-46-7
3,3''-Dichlorobenzidine	X	X	X	X	X	91-94-1
2,4-Dichlorophenol	X	X	X	X	X	120-83-2
2,6-Dichlorophenol	X	ND	ND	ND	X	87-65-0
Dichlorovos	X	ND	ND	ND	X	62-73-7
Dicrotophos	X	ND	ND	ND	X	141-66-2
Dieldrin	X	X	X	X	X	60-57-1
Diethyl Phthalate	X	X	X	X	X	84-66-2
Diethylstilbestrol	AW,OS(67)	ND	ND	ND	X	56-53-1
Diethyl Sulfate	LR	ND	ND	ND	LR	64-67-5
Dihydrosaffrole	ND	ND	ND	ND	ND	56312-13-1
Dimethoate	HE,HS	ND	ND	ND	X	60-51-5
3,''-Dimethoxybenzidine	X	ND	ND	ND	LR	119-90-4
Dimethylaminoazobenzene	X	ND	ND	ND	X	60-11-7
7,12-Dimethylbenz(a)-anthracene	CP(45)	ND	ND	ND	CP	57-97-6
3,''-Dimethylbenzidine	X	ND	ND	ND	X	119-93-7
Alpha,alpha-Dimethylphenethylamine	ND	ND	ND	ND	X	122-09-8
2,4-Dimethylphenol	X	X	X	X	X	105-67-9
Dimethyl Phthalate	X	X	X	X	X	131-11-3
1,2-Dinitrobenzene	X	ND	ND	ND	X	528-29-0
1,3-Dinitrobenzene	X	ND	ND	ND	X	99-65-0
1,4-Dinitrobenzene	HE(14)	ND	ND	ND	X	100-25-4
4,6-Dinitro-2-methylphenol	X	X	X	X	X	534-52-1
2,4-Dinitrophenol	X	X	X	X	X	51-28-5
2,4-Dinitrotoluene	X	X	X	X	X	121-14-2
2,6-Dinitrotoluene	X	X	X	X	X	606-20-2
Dinocap	CP,HS(28)	ND	ND	ND	CP	39300-45-3
Dinoseb	X	ND	ND	ND	X	88-85-7

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ANALYTE:	3510*	3520	3540/3541	3550*	3580*	CAS #(a)
Dioxathion	ND	ND	ND	ND	ND	78-34-2
Diphenylamine	X	X	X	X	X	122-39-4
5,5-Diphenylhydantoin	X	ND	ND	ND	X	57-41-0
1,2-Diphenylhydrazine	X	X	X	X	X	122-66-7
Di-n-octyl Phthalate	X	X	X	X	X	117-84-0
Disulfoton	X	ND	ND	ND	X	298-04-4
Endosulfan I	X	X	X	X	X	959-98-8
Endosulfan II	X	X	X	X	X	33212-65-9
Endosulfan Sulfate	X	X	X	X	X	1031-07-8
Endrin	X	X	X	X	X	72-20-8
Endrin Aldehyde	X	X	X	X	X	7421-93-4
Endrin Ketone	X	X	ND	X	X	53494-70-5
EPN	X	ND	ND	ND	X	2104-64-5
Ethion	X	ND	ND	ND	X	563-12-2
Ethyl Carbamate	DC(28)	ND	ND	ND	X	51-79-6
Ethyl Methanesulfonate	X	ND	ND	ND	X	62-50-0
Famphur	X	ND	ND	ND	X	52-85-7
Fensulfothion	X	ND	ND	ND	X	115-90-2
Fenthion	X	ND	ND	ND	X	55-38-9
Fluchloralin	X	ND	ND	ND	X	33245-39-5
Fluoranthene	X	X	X	X	X	206-44-0
Fluorene	X	X	X	X	X	86-73-7
2-Fluorobiphenyl (Surr)	X	X	X	X	X	321-60-8
2-Fluorophenol (Surr)	X	X	X	X	X	367-12-4
Heptachlor	X	X	X	X	X	76-44-8
Heptachlor Epoxide	X	X	X	X	X	1024-57-3
Hexachlorobenzene	X	X	X	X	X	118-74-1
Hexachlorobutadiene	X	X	X	X	X	87-68-3
Hexachlorocyclopentadiene	X	X	X	X	X	77-47-4
Hexachloroethane	X	X	X	X	X	67-72-1
Hexachlorophene	AW,CP(62)	ND	ND	ND	CP	70-30-4
Hexachloropropene	X	ND	ND	ND	X	1888-71-7
Hexamethylphosphoramide	X	ND	ND	ND	X	680-31-9
Hydroquinone	ND	ND	ND	ND	X	123-31-9
Indeno(1,2,3-cd)pyrene	X	X	X	X	X	193-39-5
Isodrin	X	ND	ND	ND	X	465-73-6
Isophorone	X	X	X	X	X	78-59-1
Isosafrole	DC(46) ND	ND	ND	ND	X	120-58-1

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ANALYTE:	3510*	3520	3540/3541	3550*	3580*	CAS #(a)
Kepone	X	ND	ND	ND	X	143-50-0
Leptophos	X	ND	ND	ND	X	21609-90-5
Malathion	HS(5)	ND	ND	ND	X	121-75-5
Maleic Anhydride	HE	ND	ND	ND	X	108-31-6
Mestranol	X	ND	ND	ND	X	72-33-3
Methapyrilene	X	ND	ND	ND	X	91-80-5
Methoxychlor	X	ND	ND	ND	X	72-43-5
3-Methylcholanthrene	X	ND	ND	ND	X	56-49-5
4,''-Methylenebis (2-chloroaniline)	OE,OS(0)	ND	ND	ND	LR	101-14-4
4,''-Methylenebis-(N-n-dimethylaniline)	X	X	ND	ND	ND	101-61-1
Methyl methanesulfonate	X	ND	ND	ND	X	66-27-3
2-Methylnaphthalene	X	X	ND	X	X	91-57-6
Methyl Parathion	X	ND	ND	ND	X	298-00-0
2-Methylphenol	X	ND	ND	ND	X	95-48-7
3-Methylphenol	X	ND	ND	ND	X	108-39-4
4-Methylphenol	X	ND	ND	ND	X	106-44-5
2-Methylpyridine	X	X	ND	ND	ND	109-06-8
Mevinphos	X	ND	ND	ND	X	7786-34-7
Mexacarbate	HE,HS(68)	ND	ND	ND	X	315-18-4
Mirex	X	ND	ND	ND	X	2385-85-5
Monocrotophos	HE	ND	ND	ND	X	6923-22-4
Naled	X	ND	ND	ND	X	300-76-5
Naphthalene	X	X	X	X	X	91-20-3
1,4-Naphthoquinone	X	ND	ND	ND	X	130-15-4
1-Naphthylamine	OS(44)	ND	ND	ND	X	134-32-7
2-Naphthylamine	X	ND	ND	ND	X	91-59-8
Nicotine	DE(67)	ND	ND	ND	X	54-11-5
5-Nitroacenaphthene	X	ND	ND	ND	X	602-87-9
2-Nitroaniline	X	X	ND	X	X	88-74-4
3-Nitroaniline	X	X	ND	X	X	99-09-2
4-Nitroaniline	X	X	ND	X	X	100-01-6
5-Nitro-o-anisidine	X	ND	ND	ND	X	99-59-2
Nitrobenzene	X	X	X	X	X	98-95-3
4-Nitrobiphenyl	X	ND	ND	ND	X	92-93-3
Nitrofen	X	ND	ND	ND	X	1836-75-5
2-Nitrophenol	X	X	X	X	X	88-75-5
4-Nitrophenol	X	X	X	X	X	100-02-7

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ANALYTE:	3510*	3520	3540/3541	3550*	3580*	CAS #(a)
5-Nitro-o-toluidine	X	ND	ND	ND	X	99-55-8
Nitroquinoline-1-oxide	X	ND	ND	ND	X	56-57-5
N-nitrosodi-n-butylamine	X	ND	ND	ND	X	924-16-3
N-nitrosodiethylamine	X	ND	ND	ND	X	55-18-5
N-nitrosodimethylamine	X	X	X	X	X	62-75-9
N-nitrosomethylethylamine	X	ND	ND	ND	X	10595-95-6
N-nitrosodiphenylamine	X	X	X	X	X	86-30-6
N-nitrosodi-n-propylamine	X	X	X	X	X	621-64-7
N-nitrosomorpholine	ND	ND	ND	ND	X	59-89-2
N-nitrosopiperidine	X	ND	ND	ND	X	100-75-4
N-nitrosopyrrolidine	X	ND	ND	ND	X	930-55-2
Octamethyl Pyrophosphoramide	LR	ND	ND	ND	LR	152-16-9
Parathion	X	ND	ND	ND	X	56-38-2
Pentachlorobenzene	X	ND	ND	ND	X	608-93-5
Pentachloronitrobenzene	X	ND	ND	ND	X	82-68-8
Pentachlorophenol	X	X	X	X	X	87-86-5
Phenacetin	X	ND	ND	ND	X	62-44-2
Phenanthrene	X	X	X	X	X	85-01-8
Phenobarbital	X	ND	ND	ND	X	50-06-6
Phenol DC(28)	X	X	X	X		108-95-2
1,4-Phenylenediamine	X	ND	ND	ND	X	106-50-3
Phorate	X	ND	ND	ND	X	298-02-2
Phosalone	HS(65)	ND	ND	ND	X	2310-17-0
Phosmet	HS(15)	ND	ND	ND	X	732-11-6
Phosphamidon	HE(63)	ND	ND	ND	X	13171-21-6
Phthalic Anhydride	CP,ME(1)	ND	ND	ND	CP	85-44-9
2-Picoline	X	X	ND	ND	ND	109-06-8
Piperonyl Sulfoxide	X	ND	ND	ND	X	120-62-7
Pronamide	X	ND	ND	ND	X	23950-58-5
Pyrene	X	X	X	X	X	129-00-0
Pyridine	ND	ND	ND	ND	ND	110-86-1
Resorcinol	X	ND	ND	ND	X	94-59-7
Safrole	AW,OS(55)	ND	ND	ND	X	60-41-3
Sulfallate	X	ND	ND	ND	X	95-06-7
Terbufos	X	ND	ND	ND	X	13071-79-9
Terphenyl d(l4)(surr)	X	X	ND	X	X	1718-51-0
1,2,4,5-Tetrachlorobenzene	X	ND	ND	ND	X	95-94-3
2,3,4,6-Tetrachlorophenol	X	ND	ND	ND	X	58-90-2

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ANALYTE:	3510*	3520	3540/3541	3550*	3580*	CAS #(a)
Tetrachlorvinphos	X	ND	ND	ND	X	961-11-5
Tetraethyl Dithiopyrophosphate	X	X	ND	ND	ND	3689-24-5
Tetraethyl Pyrophosphate	X	ND	ND	ND	X	107-49-3
Thionazine	X	ND	ND	ND	X	297-97-2
Thiophenol	X	ND	ND	ND	X	108-98-5
Benzenethiol	X	ND	ND	ND	X	108-98-5
Toluene Diisocyanate	HE(6)	ND	ND	ND	X	584-84-9
Ortho-toluidine	X	ND	ND	ND	X	95-53-4
Toxaphene	X	X	X	X	X	8001-35-2
1,2,4-Trichlorobenzene	X	X	X	X	X	120-82-1
2,4,5-Trichlorophenol	X	X	ND	X	X	95-95-4
2,4,6-Trichlorophenol	X	X	X	X	X	88-06-2
Trifluralin	X	ND	ND	ND	X	1582-09-8
2,4,5-Trimethylaniline	X	ND	ND	ND	X	137-17-7
Trimethyl Phosphate	HE(60)	ND	ND	ND	X	512-56-1
1,3,5-Trinitrobenzene	X	ND	ND	ND	X	99-35-4
Tris(2,3-dibromopropyl) phosphate	X	ND	ND	ND	LR	126-72-7
O,O,O-Triethyl Phosphorothioate	X	ND	ND	ND	X	126-68-1

KEY TO ANALYTE LIST ESC extraction technique Chemical Abstract Service Registry Number
(b) See Sec. 1.2 for other acceptable preparation methods.
(IS) This compound may be used as an internal standard.
(surr) This compound may be used as a surrogate.
(AW) Adsorption to walls of glassware during extraction and storage.
(CP) Nonreproducible chromatographic performance.
(DC) Unfavorable distribution coefficient (number in parenthesis is percent recovery).
(HE) Hydrolysis during extraction accelerated by acidic or basic conditions (number in parenthesis is percent recovery).
(HS) Hydrolysis during storage (number in parenthesis is percent stability).
(LR) Low response.
(ND) Not determined.
(OE) Oxidation during extraction accelerated by basic conditions (number in parenthesis is percent recovery).
(OS) Oxidation during storage (number in parenthesis is percent stability).
(X) Greater than 70 percent recovery by this technique.

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TITLE: SEMI-VOLATILE ORGANICS BY GAS CHROMATOGRAPHY/MASS SPECTROMETRY USING CAPILLARY COLUMN (EPA METHOD 8270C, EPA 8270D, EPA METHOD 625, SM 6410B, 20TH ED.), INCLUDING PROVISIONS FOR ANALYSIS IN SIM MODE.

Attachment IVa 8270/625– Common Calibration List & ESC-calculated water limits for 3510C 1L extraction as of 7/28/11- *(may be updated without notice)*

Compound Name	LCS / LCSD Limits					MS/MSD Limits		
	Control Limits		RPD Limit	Marginal Excedence		Control Limits		RPD Limit
	Lower	Upper		Lower	Upper	Lower	Upper	
PYRIDINE	11	52	36	*10*	59	*e*	64	*40*
PYRENE	65	116	*20*	57	124	47	135	21
PHENOL	10	53	*20*	*10*	60	*10*	69	34
PHENANTHRENE	61	110	*20*	53	118	49	130	*20*
PENTACHLOROPHENOL	*10*	101	*40*	*5*	117	*10*	152	40
N-OCTADECANE	27	136	*20*	*10*	154	*10*	*200*	*40*
N-NITROSODIPHENYLAMINE	55	98	*20*	48	105	43	124	*20*
N-NITRODIPHENYLAMINE	10	186	*20*	10	*200*	*10*	195	*20*
N-NITROSODI-N-PROPYLAMINE	50	115	*20*	39	125	36	139	24
N-NITROSODIMETHYLAMINE	12	68	31	*10*	77	*10*	83	*40*
NITROBENZENE	39	102	*20*	28	113	36	110	24
N-DECANE	10	96	27	*10*	110	*10*	*200*	*40*
NAPHTHALENE	42	103	*20*	32	113	32	116	23
ISOPHORONE	55	108	*20*	46	117	47	116	20
INDENO(1,2,3-CD)PYRENE	56	129	*20*	43	141	17	147	21
HEXACHLOROETHANE	24	93	25	13	104	*10*	121	31
HEXACHLOROCYCLOPENTADIENE	20	121	27	*10*	137	*10*	125	35
HEXACHLOROBENZENE	55	117	*20*	44	127	48	126	*20*
HEXACHLORO-1,3-BUTADIENE	34	115	*22*	21	128	32	120	24
FLUORENE	58	110	*20*	49	119	51	120	*20*
FLUORANTHENE	66	120	*20*	57	129	58	130	*20*
DI-N-OCTYL PHTHALATE	59	143	*20*	46	157	23	173	25
DI-N-BUTYL PHTHALATE	56	133	*20*	43	146	43	154	*20*
DIMETHYL PHTHALATE	*10*	152	22	*10*	177	*10*	159	30
DIETHYL PHTHALATE	33	136	*20*	15	153	28	150	21
DIBENZOFURAN	53	109	*20*	43	118	51	117	*20*
DIBENZ(A,H)ANTHRACENE	54	130	*20*	42	143	16	149	22
CHRYSENE	65	114	*20*	57	122	55	124	*20*
CARBAZOLE	62	114	*20*	53	123	50	130	*20*
CAPROLACTAM	*10*	30	24	*5*	34	*10*	108	*40*
BIS(2-ETHYLHEXYL)PHTHALATE	61	147	*20*	47	162	23	173	24
BIS(2-CHLOROISOPROPYL)ETHER	43	108	*20*	32	119	35	121	25
BIS(2-CHLOROETHYL)ETHER	39	109	23	28	121	31	122	29
BIS(2-CHLORETHOXY)METHANE	56	116	*20*	46	126	39	139	22
BIPHENYL	48	105	*20*	39	114	45	115	*20*
BENZYLBUTYL PHTHALATE	12	166	*20*	*10*	192	14	180	25

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Compound Name	LCS / LCSD Limits					MS/MSD Limits		
	Control Limits		RPD Limit	Marginal Excedence		Control Limits		RPD Limit
	Lower	Upper		Lower	Upper	Lower	Upper	
BENZYL ALCOHOL	32	91	*20*	22	101	15	113	24
BENZOIC ACID	*10*	62	37	*5*	73	*10*	132	*40*
BENZO(K)FLUORANTHENE	62	116	*20*	53	125	40	136	21
BENZO(G,H,I)PERYLENE	52	132	*20*	38	145	*10*	152	22
BENZO(B)FLUORANTHENE	67	114	*20*	59	122	42	138	20
BENZO(A)PYRENE	68	115	*20*	60	123	47	132	*20*
BENZO(A)ANTHRACENE	68	113	*20*	60	121	55	127	*20*
BENZIDINE	*10*	31	*40*	*5*	36	*10*	47	*40*
BENZALDEHYDE	*10*	56	26	*5*	64	*10*	64	*40*
AZOBENZENE	52	113	*20*	42	123	47	133	21
ATRAZINE	61	116	*20*	52	125	36	151	25
ANTHRACENE	65	114	*20*	57	122	51	129	*20*
ANILINE	30	78	24	23	86	*10*	125	36
ACETOPHENONE	44	98	*20*	36	106	18	115	28
ACENAPHTHYLENE	55	119	*20*	44	129	52	122	*20*
ACENAPHTHENE	52	107	*20*	42	116	45	121	*20*
4-NITROPHENOL	*10*	53	*40*	*5*	61	*10*	86	*40*
4-NITROANILINE	53	135	*20*	39	149	13	172	27
4-CHLOROPHENYL-PHENYLETHER	58	115	*20*	49	124	47	132	*20*
4-CHLOROANILINE	43	104	*20*	32	114	14	127	34
4-CHLORO-3-METHYLPHENOL	50	105	*20*	41	114	19	138	25
4-BROMOPHENYL-PHENYLETHER	63	120	*20*	53	130	51	138	*20*
4,6-DINITRO-2-METHYLPHENOL	21	119	*40*	*10*	135	*10*	149	*40*
3-NITROANILINE	49	116	*20*	38	127	18	141	31
3,3-DICHLOROBENZIDINE	58	116	*20*	49	125	*10*	149	33
3&4-METHYLPHENOL	33	94	*20*	23	104	*10*	136	29
2-NITROPHENOL	40	112	22	28	125	25	130	31
2-NITROANILINE	56	122	*20*	46	132	47	136	20
2-METHYLPHENOL	35	84	*20*	27	92	*10*	104	39
2-METHYLNAPHTHALENE	46	105	*20*	36	115	46	113	21
2-CHLOROPHENOL	37	90	21	28	98	18	103	33
2-CHLORONAPHTHALENE	47	106	*20*	37	116	47	111	*20*
2,6-DINITROTOLUENE	57	110	*20*	48	118	52	123	*20*
2,4-DINITROTOLUENE	59	117	*20*	49	127	48	133	21
2,4-DINITROPHENOL	10	121	*40*	*5*	139	*10*	167	36
2,4-DIMETHYLPHENOL	47	108	*20*	37	118	*10*	154	*40*
2,4-DICHLOROPHENOL	46	105	*20*	37	114	17	132	30
2,4,6-TRICHLOROPHENOL	38	113	29	26	125	14	145	39

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Compound Name	LCS / LCSD Limits					MS/MSD Limits		
	Control Limits		RPD Limit	Marginal Excedence		Control Limits		RPD Limit
	Lower	Upper		Lower	Upper	Lower	Upper	
2,4,5-TRICHLOROPHENOL	41	125	27	27	139	27	143	34
1-METHYLNAPHTHALENE	45	100	*20*	36	109	42	106	20
1,4-DICHLOROBENZENE	28	94	25	18	105	28	95	30
1,3-DICHLOROBENZENE	27	94	25	16	105	28	94	30
1,2-DICHLOROBENZENE	30	96	24	19	106	30	97	28
1,2,4-TRICHLOROBENZENE	34	97	21	24	107	34	101	23
1,2,4,5-TETRACHLOROBENZENE	40	109	*20*	29	120	27	125	20
Surrogates								
P-TERPHENYL-D14	40	174						
PHENOL-D5	*10*	63						
NITROBENZENE-D5	17	119						
2-FLUOROPHENOL	*10*	75						
2-FLUOROBIPHENYL	29	127						
2,4,6-TRIBROMOPHENOL	16	147						

STATE NOTE: For all samples from South Carolina, the LCS/LCSD recovery must be within 70-130% of the expected target concentration with an RPD of <20%.

Attachment IVb 8270/625 - Common Calibration List & ESC-calculated Soil limits as of 7/28/11

Compound Name	LCS / LCSD Limits					MS/MSD Limits		
	Control Limits		RPD Limit	Marginal Excedence		Control Limits		RPD Limit
	Lower	Upper		Lower	Upper	Lower	Upper	
PYRIDINE	17	79	27	*10*	90	*10*	114	32
PYRENE	54	104	*20*	46	113	23	145	30
PHENOL	49	99	*20*	40	108	22	129	25
PHENANTHRENE	55	103	*20*	47	110	25	139	25
PENTACHLOROPHENOL	*10*	89	28	10	103	*10*	124	34
N-OCTADECANE	33	122	20	18	137	*10*	175	*40*
N-NITROSODIPHENYLAMINE	48	90	*20*	41	97	16	128	25
N-NITRODIPHENYLAMINE	57	121	*20*	46	132	19	164	27
N-NITROSODI-N-PROPYLAMINE	52	103	*20*	44	112	24	141	20
N-NITROSODIMETHYLAMINE	31	107	23	18	120	18	126	27
NITROBENZENE	47	92	*20*	40	100	22	122	20
N-DECANE	31	93	21	21	104	11	111	30
NAPHTHALENE	55	91	*20*	48	97	31	124	25
ISOPHORONE	51	99	*20*	43	107	26	134	20
INDENO(1,2,3-CD)PYRENE	50	110	*20*	40	120	*10*	139	32
HEXACHLOROETHANE	45	83	*20*	38	90	21	107	27
HEXACHLOROCYCLOPENTADIENE	36	117	20	23	130	*10*	124	33

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Compound Name	LCS / LCSD Limits					MS/MSD Limits		
	Control Limits		RPD Limit	Marginal Excedence		Control Limits		RPD Limit
	Lower	Upper		Lower	Upper	Lower	Upper	
HEXACHLOROBENZENE	50	108	*20*	41	118	26	136	20
HEXACHLORO-1,3-BUTADIENE	53	106	*20*	44	114	29	136	22
FLUORENE	59	100	*20*	52	107	30	138	22
FLUORANTHENE	59	108	*20*	50	116	24	145	29
DI-N-OCTYL PHTHALATE	51	119	22	40	131	14	164	24
DI-N-BUTYL PHTHALATE	59	114	*20*	50	120	24	149	24
DIMETHYL PHTHALATE	60	106	*20*	53	114	31	142	22
DIETHYL PHTHALATE	61	105	*20*	53	112	28	142	23
DIBENZOFURAN	56	98	*20*	50	105	30	131	20
DIBENZ(A,H)ANTHRACENE	49	111	*20*	39	122	*10*	137	29
CHRYSENE	55	102	*20*	48	109	20	139	23
CARBAZOLE	51	103	*20*	42	112	26	135	22
CAPROLACTAM	43	104	*20*	33	115	*10*	156	29
BIS(2-ETHYLHEXYL)PHTHALATE	56	120	*20*	46	130	20	163	24
BIS(2-CHLOROISOPROPYL)ETHER	56	95	*20*	49	101	32	128	22
BIS(2-CHLOROETHYL)ETHER	51	103	20	42	111	25	139	26
BIS(2-CHLORETHOXY)METHANE	58	104	*20*	50	112	32	141	*20*
BIPHENYL	55	93	*20*	48	99	30	125	20
BENZYLBUTYL PHTHALATE	61	118	*20*	51	127	20	168	23
BENZYL ALCOHOL	48	96	*20*	40	104	33	119	21
BENZOIC ACID	*10*	110	41	*10*	130	*10*	132	*40*
BENZO(K)FLUORANTHENE	53	104	*20*	44	112	15	152	22
BENZO(G,H,I)PERYLENE	47	112	*20*	37	122	*10*	137	32
BENZO(B)FLUORANTHENE	52	106	*20*	43	115	13	152	24
BENZO(A)PYRENE	57	103	*20*	50	111	16	148	21
BENZO(A)ANTHRACENE	56	103	*20*	48	111	22	139	22
BENZIDINE	10	55	40	5	67	10	55	36
BENZALDEHYDE	*10*	30	23	*10*	34	*10*	89	*40*
AZOBENZENE	49	105	*20*	39	115	28	131	21
ATRAZINE	55	101	*20*	47	108	19	145	29
ANTHRACENE	58	105	*20*	50	114	27	140	*20*
ANILINE	32	79	23	24	87	*10*	99	30
ACETOPHENONE	49	88	*20*	42	95	28	117	22
ACENAPHTHYLENE	61	107	*20*	54	114	31	144	24
ACENAPHTHENE	55	96	*20*	49	103	30	132	21
4-NITROPHENOL	34	101	26	23	112	*10*	166	35
4-NITROANILINE	41	105	20	30	116	13	145	28
4-CHLOROPHENYL-PHENYLETHER	59	103	*20*	52	111	27	142	21
4-CHLOROANILINE	38	89	20	30	97	*10*	116	*40*
4-CHLORO-3-METHYLPHENOL	58	98	*20*	51	105	24	140	22

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Compound Name	LCS / LCSD Limits					MS/MSD Limits		
	Control Limits		RPD Limit	Marginal Excedence		Control Limits		RPD Limit
	Lower	Upper		Lower	Upper	Lower	Upper	
4-BROMOPHENYL-PHENYLETHER	58	111	*20*	50	120	27	150	20
4,6-DINITRO-2-METHYLPHENOL	24	98	32	12	111	*10*	124	*40*
3-NITROANILINE	42	91	*20*	34	99	*10*	130	37
3,3-DICHLOROBENZIDINE	36	84	20	28	91	*10*	127	*40*
3&4-METHYLPHENOL	60	104	*20*	53	111	18	150	28
2-NITROPHENOL	55	106	*20*	46	115	*10*	156	24
2-NITROANILINE	55	110	*20*	46	119	15	154	23
2-METHYLPHENOL	52	90	*20*	46	96	19	126	24
2-METHYLNAPHTHALENE	57	94	*20*	51	100	32	128	20
2-CHLOROPHENOL	52	88	*20*	46	94	26	120	21
2-CHLORONAPHTHALENE	55	96	*20*	48	103	31	127	23
2,6-DINITROTOLUENE	53	99	*20*	46	106	*10*	150	23
2,4-DINITROTOLUENE	54	103	*20*	46	112	12	146	25
2,4-DINITROPHENOL	*10*	109	39	10	126	*10*	110	*40*
2,4-DIMETHYLPHENOL	52	101	*20*	44	109	13	147	27
2,4-DICHLOROPHENOL	56	96	*20*	49	102	30	134	23
2,4,6-TRICHLOROPHENOL	50	98	*20*	42	106	18	140	26
2,4,5-TRICHLOROPHENOL	48	103	*20*	40	112	21	147	23
1-METHYLNAPHTHALENE	54	90	*20*	48	97	31	121	*20*
1,4-DICHLOROBENZENE	47	84	*20*	41	91	27	110	25
1,3-DICHLOROBENZENE	47	84	*20*	40	90	26	110	25
1,2-DICHLOROBENZENE	48	86	*20*	42	92	27	134	25
1,2,4-TRICHLOROBENZENE	48	87	*20*	41	94	27	118	23
1,2,4,5-TETRACHLOROBENZENE	52	99	*20*	44	107	26	137	*20*
Surrogates								
P-TERPHENYL-D14	40	174						
PHENOL-D5	*10*	63						
NITROBENZENE-D5	17	119						
2-FLUOROPHENOL	*10*	75						
2-FLUOROBIPHENYL	29	127						
2,4,6-TRIBROMOPHENOL	16	147						

STATE NOTE: For all samples from South Carolina, the LCS/LCSD recovery must be within 70-130% of the expected target concentration with an RPD of <20%.

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TITLE: SEMI-VOLATILE ORGANICS BY GAS CHROMATOGRAPHY/MASS SPECTROMETRY USING CAPILLARY COLUMN (EPA METHOD 8270C, EPA 8270D, EPA METHOD 625, SM 6410B, 20TH ED.), INCLUDING PROVISIONS FOR ANALYSIS IN SIM MODE.

Attachment IVc 8270PAHSIM – Common Calibration List & ESC-calculated water limits for 3510C 1L extraction as of 7/28/11- *(may be updated without notice)*

Compound Name	LCS / LCSD Limits					MS/MSD Limits		
	Control Limits		RPD Limit	Marginal Excedence		Control Limits		RPD Limit
	Lower	Upper		Lower	Upper	Lower	Upper	
PYRENE	55	135	*20*	42	149	*10*	164	38
PHENANTHRENE	54	127	*20*	41	139	14	150	37
NAPHTHALENE	41	122	21	28	136	*10*	140	36
INDENO(1,2,3-CD)PYRENE	56	133	*20*	44	146	*10*	122	*40*
FLUORENE	52	125	*20*	40	137	27	135	31
FLUORANTHENE	57	134	*20*	45	146	11	167	*40*
DIBENZ(A,H)ANTHRACENE	57	131	*20*	44	144	*10*	101	40
CHRYSENE	58	132	*20*	45	144	14	143	36
BENZO(K)FLUORANTHENE	55	137	*20*	42	151	*10*	133	*40*
BENZO(G,H,I)PERYLENE	54	139	*20*	40	153	*10*	122	*40*
BENZO(B)FLUORANTHENE	53	131	*20*	41	144	*10*	126	*40*
BENZO(A)PYRENE	58	132	*20*	46	145	*10*	132	*40*
BENZO(A)ANTHRACENE	58	123	*20*	47	134	14	146	37
ANTHRACENE	61	133	*20*	48	145	32	134	29
ACENAPHTHYLENE	50	125	*20*	37	138	32	125	28
ACENAPHTHENE	51	121	*20*	39	133	27	128	32
2-METHYLNAPHTHALENE	43	126	21	29	140	*10*	148	*40*
2-CHLORONAPHTHALENE	47	129	21	33	143	23	131	35
1-METHYLNAPHTHALENE	48	126	21	34	140	20	132	37
Surrogates								
P-TERPHENYL-D14	38	132						
NITROBENZENE-D5	26	136						
2-FLUOROBIPHENYL	44	123						

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TITLE: SEMI-VOLATILE ORGANICS BY GAS CHROMATOGRAPHY/MASS SPECTROMETRY USING CAPILLARY COLUMN (EPA METHOD 8270C, EPA 8270D, EPA METHOD 625, SM 6410B, 20TH ED.), INCLUDING PROVISIONS FOR ANALYSIS IN SIM MODE.

Attachment IVd 8270PAHSIM – Common Calibration List & ESC-calculated Soil limits as of 7/28/11- *(may be updated without notice)*

Compound Name	LCS / LCSD Limits					MS/MSD Limits		
	Control Limits		RPD Limit	Marginal Excedence		Control Limits		RPD Limit
	Lower	Upper		Lower	Upper	Lower	Upper	
PYRENE	53	121	*20*	42	132	12	170	24
PHENANTHRENE	53	114	20	43	124	23	164	25
NAPHTHALENE	45	105	24	35	115	22	156	27
INDENO(1,2,3-CD)PYRENE	51	125	21	39	137	*10*	157	*40*
FLUORENE	54	109	20	45	119	44	143	23
FLUORANTHENE	58	118	20	48	128	23	160	22
DIBENZ(A,H)ANTHRACENE	53	122	20	41	134	*10*	160	39
CHRYSENE	57	118	*20*	47	129	26	146	30
BENZO(K)FLUORANTHENE	55	122	25	43	134	22	163	29
BENZO(G,H,I)PERYLENE	48	130	20	35	144	*10*	176	30
BENZO(B)FLUORANTHENE	55	114	20	45	124	*10*	188	33
BENZO(A)PYRENE	56	118	21	45	129	26	152	32
BENZO(A)ANTHRACENE	54	110	22	44	120	31	142	31
ANTHRACENE	58	120	20	48	130	38	153	27
ACENAPHTHYLENE	51	110	21	41	120	42	146	22
ACENAPHTHENE	52	108	22	42	118	43	133	26
2-METHYLNAPHTHALENE	44	109	24	33	120	22	172	29
2-CHLORONAPHTHALENE	51	114	24	40	125	31	153	22
1-METHYLNAPHTHALENE	48	113	24	37	124	25	155	27
Surrogates								
P-TERPHENYL-D14	25	139						
NITROBENZENE-D5	14	141						
2-FLUOROBIPHENYL	34	129						

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TITLE: SEMI-VOLATILE ORGANICS BY GAS CHROMATOGRAPHY/MASS SPECTROMETRY USING CAPILLARY COLUMN (EPA METHOD 8270C, EPA 8270D, EPA METHOD 625, SM 6410B, 20TH ED.), INCLUDING PROVISIONS FOR ANALYSIS IN SIM MODE.

Attachment IVe 8270 Missouri PAH & DRO – Common Calibration List & ESC-calculated water limits for 3510C 1L extraction as of 7/28/11- *(may be updated without notice)*

Compound Name	LCS / LCSD Limits					MS/MSD Limits		
	Control Limits		RPD Limit	Marginal Excedence		Control Limits		RPD Limit
	Lower	Upper		Lower	Upper	Lower	Upper	
PYRENE	64	127	*20*	54	137	64	127	*20*
PHENANTHRENE	63	133	*20*	52	145	63	133	*20*
NAPHTHALENE	44	124	29	30	138	44	124	29
INDENO(1,2,3-CD)PYRENE	53	135	*20*	40	148	53	135	*20*
FLUORENE	61	128	*20*	50	140	61	128	*20*
FLUORANTHENE	65	130	20	54	141	65	130	20
DIBENZ(A,H)ANTHRACENE	53	137	20	38	151	53	137	20
CHRYSENE	66	124	*20*	57	134	66	124	*20*
BENZO(K)FLUORANTHENE	60	129	25	49	140	60	129	25
BENZO(G,H,I)PERYLENE	58	133	*20*	46	145	58	133	*20*
BENZO(B)FLUORANTHENE	64	130	22	53	141	64	130	22
BENZO(A)PYRENE	62	126	*20*	52	137	62	126	*20*
BENZO(A)ANTHRACENE	66	118	*20*	58	127	66	118	*20*
ANTHRACENE	61	126	*20*	51	137	61	126	*20*
ACENAPHTHYLENE	58	126	*20*	46	138	58	126	*20*
ACENAPHTHENE	57	122	*20*	46	132	57	122	*20*
Surrogates								
P-TERPHENYL-D14	26	153						
NITROBENZENE-D5	23	139						
2-FLUOROBIPHENYL	31	148						
TPH (GC/FID) HIGH FRACTION	52	142	18	37	157	52	142	18
Surrogates								
P-TERPHENYL-D14	26	160						
NITROBENZENE-D5	35	137						
2-FLUOROBIPHENYL	43	145						

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Attachment IVf 8270 Missouri PAH & DRO – Common Calibration List & ESC-calculated Soil limits as of 7/28/11- *(may be updated without notice)*

Compound Name	LCS / LCSD Limits					MS/MSD Limits		
	Control Limits		RPD Limit	Marginal Excedence		Control Limits		RPD Limit
	Lower	Upper		Lower	Upper	Lower	Upper	
PYRENE	56	123	*20*	45	134	*10*	158	*40*
PHENANTHRENE	54	130	*20*	41	142	*10*	157	*40*
NAPHTHALENE	44	116	*20*	32	127	*10*	130	23
INDENO(1,2,3-CD)PYRENE	52	119	*20*	41	130	*10*	136	33
FLUORENE	58	127	*20*	46	139	10	137	22
FLUORANTHENE	56	127	*20*	44	139	*10*	157	*40*
DIBENZ(A,H)ANTHRACENE	54	117	20	43	128	*10*	128	24
CHRYSENE	58	121	*20*	47	132	12	137	23
BENZO(K)FLUORANTHENE	57	123	23	46	134	*10*	145	32
BENZO(G,H,I)PERYLENE	53	125	*20*	41	137	*10*	138	*40*
BENZO(B)FLUORANTHENE	59	128	20	47	139	*10*	159	*40*
BENZO(A)PYRENE	57	117	*20*	47	127	12	135	33
BENZO(A)ANTHRACENE	58	116	*20*	48	125	21	127	37
ANTHRACENE	54	126	*20*	41	138	13	135	*26*
ACENAPHTHYLENE	55	124	*20*	44	135	22	130	21
ACENAPHTHENE	53	120	*20*	42	131	20	125	23
Surrogates								
P-TERPHENYL-D14	39	148						
NITROBENZENE-D5	26	140						
2-FLUOROBIPHENYL	39	145						
TPH (GC/FID) HIGH FRACTION	61	145	*23*	47	159	*10*	187	*40*
Surrogates								
P-TERPHENYL-D14	37	155						
NITROBENZENE-D5	30	141						
2-FLUOROBIPHENYL	39	149						

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TITLE: SEMI-VOLATILE ORGANICS BY GAS CHROMATOGRAPHY/MASS SPECTROMETRY USING CAPILLARY COLUMN (EPA METHOD 8270C, EPA 8270D, EPA METHOD 625, SM 6410B, 20TH ED.), INCLUDING PROVISIONS FOR ANALYSIS IN SIM MODE.

Attachment V: Characteristic Masses (m/z) for Extractable Organic Compounds

(Reprinted from SW-846 Method 8270C /Dec. 1996)

Compound	Primary Characteristic Ion	Secondary Characteristic Ion(s)
Pyridine	79	52,78,51
N-Nitrosodimethylamine	42	74,44
2-Picoline	93	66,92
Aniline	93	66,65
Phenol	94	65,66
Benzaldehyde	105	106,77,51
Bis(2-chloroethyl) ether	93	63,95
2-Chlorophenol	128	64,130
1,3-Dichlorobenzene	146	148,111
1,4-Dichlorobenzene-d4 (ISTD)	152	150,115
1,4-Dichlorobenzene	146	148,111
Benzyl alcohol	108	79,77
1,2-Dichlorobenzene	146	148,111
N-Nitrosomethylethylamine	88	42,43,56
Bis(2-chloroisopropyl) ether	45	77,121
Methyl methanesulfonate	80	79,65,95
N-Nitrosodi-n-propylamine	70	42,101,130
Hexachloroethane	117	201,199
Nitrobenzene	77	123,65
Isophorone	82	95,138
N-Nitrosodiethylamine	102	42,57,44,56
2-Nitrophenol	139	109,65
2,4-Dimethylphenol	122	107,121
Bis(2-chloroethoxy)methane	93	95,123
Benzoic acid	122	105,77
2,4-Dichlorophenol	162	164,98
Ethyl methanesulfonate	79	109,97,45,65
1,2,4-Trichlorobenzene	180	182,145
Naphthalene-d8 (ISTD)	136	68
Naphthalene	128	129,127
Hexachlorobutadiene	225	223,227
Caprolactam	113	55,56,42
4-Chloro-3-methylphenol	107	144,142
2-Methylnaphthalene	142	141
1-Methylnaphthalene	142	141
2-Methylphenol	107	108,77,79,90

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Compound	Primary Characteristic Ion	Secondary Characteristic Ion(s)
Hexachloropropene	213	211,215,117,106,141
Hexachlorocyclopentadiene	237	235,272
N-Nitrosopyrrolidine	100	41,42,68,69
Acetophenone	105	71,51,120
4-Methylphenol	107	108,77,79,90
2,4,6-Trichlorophenol	196	198,200
2,4,5-Trichlorophenol	196	198,200
o-Toluidine	106	107,77,51,79
3-Methylphenol	107	108,77,79,90
2-Chloronaphthalene	162	127,164
N-Nitrosopiperidine	114	42,55,56,41
1-Chloronaphthalene	162	127,164
2-Nitroaniline	65	92,138
Dimethyl phthalate	163	194,164
Acenaphthylene	152	151,153
2,6-Dinitrotoluene	165	63,89
3-Nitroaniline	138	108,92
Acenaphthene-d10 (ISTD)	164	162,160
Acenaphthene	154	153,152
2,4-Dinitrophenol	184	63,154
2,6-Dinitrophenol	162	164,126,98,63
4-Chloroaniline	127	129,65,92
Isosafrole	162	131,104,77,51
Dibenzofuran	168	139
2,4-Dinitrotoluene	165	63,89
4-Nitrophenol	139	109,65
2-Naphthylamine	143	115,116
1,4-Naphthoquinone	158	104,102,76,50,130
Diethyl phthalate	149	177,150
Fluorene	166	165,167
N-Nitrosodi-n-butylamine	84	57,41,116,158
4-Chlorophenyl phenyl ether	204	206,141
Atrazine	200	215,58
4,6-Dinitro-2-methylphenol	198	51,105
N-Nitrosodiphenylamine	169	168,167
Safrole	162	104,77,103,135
Diphenylamine	169	168,167
1,2,4,5-Tetrachlorobenzene	216	214,179,108,143,218

TITLE: SEMI-VOLATILE ORGANICS BY GAS CHROMATOGRAPHY/MASS SPECTROMETRY USING CAPILLARY COLUMN (EPA METHOD 8270C, EPA 8270D, EPA METHOD 625, SM 6410B, 20TH ED.), INCLUDING PROVISIONS FOR ANALYSIS IN SIM MODE.

Compound	Primary Characteristic Ion	Secondary Characteristic Ion(s)
1-Naphthylamine	143	115,89,63
4-Bromophenyl phenyl ether	248	250,141
2,4,5-Trichlorophenol	196	198,97,132,99
Hexachlorobenzene	284	142,249
Pentachlorophenol	266	264,268
5-Nitro-o-toluidine	152	77,79,106,94
Thionazine	107	96,97,143,79,68
4-Nitroaniline	138	65,108,92,80,39
Phenanthrene-d10 (ISTD)	188	94,80
Phenanthrene	178	179,176
Anthracene	178	176,179
Carbazole	167	166,168,139
1,3-Dinitrobenzene	168	76,50,75,92,122
Diallate (cis or trans)	86	234,43,70
Pentachlorobenzene	250	252,108,248,215,254
Pentachloronitrobenzene	237	142,214,249,295,265
4-Nitroquinoline-1-oxide	174	101,128,75,116
Di-n-butyl phthalate	149	150,104
2,3,4,6-Tetrachlorophenol	232	131,230,166,234,168
Demeton-O	88	89,60,61,115,171
Fluoranthene	202	101,203
1,3,5-Trinitrobenzene	75	74,213,120,91,63
Benzidine	184	92,185
Pyrene	202	200,203
Phorate	75	121,97,93,260
Demeton-S	88	60,81,89,114,115
Phenacetin	108	180,179,109,137,80
Dimethoate	87	93,125,143,229
4-Aminobiphenyl	169	168,170,115
Dimethylphenylamine	58	91,65,134,42
Pronamide	173	175,145,109,147
Dinoseb	211	163,147,117,240
Disulfoton	88	97,89,142,186
Butyl benzyl phthalate	149	91,206
Methyl parathion	109	125,263,79,93
Dimethylaminoazobenzene	225	120,77,105,148,42
Benz(a)anthracene	228	229,226

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Compound	**Primary Characteristic Ion**	**Secondary Characteristic Ion(s)**
Chrysene-d12 (ISTD)	240	120,236
3,3'-Dichlorobenzidine	252	254,126
Chrysene	228	226,229
Kepone	272	274,237,178,143,270
Parathion	109	97,291,139,155
Bis(2-ethylhexyl) phthalate	149	167,279
3,3'-Dimethylbenzidine	212	106,196,180
Methapyrilene	97	50,191,71
Isodrin	193	66,195,263,265,147
Di-n-octyl phthalate	149	167,43
Aramite	185	191,319,334,197,321
Benzo(b)fluoranthene	252	253,125
Benzo(k)fluoranthene	252	253,125
Famphur	218	125,93,109,217
Benzo(a)pyrene	252	253,125
Perylene-d12 (ISTD)	264	260,265
7,12-Dimethylbenz(a)anthracene	256	241,239,120
2-Acetylaminofluorene	181	180,223,152
3-Methylcholanthrene	268	252,253,126,134,113
Dibenz(a,j)acridine	279	280,277,250
Indeno(1,2,3-cd)pyrene	276	138,227
Dibenz(a,h)anthracene	278	139,279
Benzo(g,h,i)perylene	276	138,277
Hexachlorophene	196	198,209,211,406,408
1,2-Diphenylhydrazine/Azobenzene	77	105,182
Surrogates		
2-Fluorobiphenyl (surr)	172	171
2-Fluorophenol (surr)	112	64
Nitrobenzene-d5 (surr)	82	128,54
Phenol-d6 (surr)	99	42,71
Terphenyl-d14 (surr)	244	122,212
2,4,6-Tribromophenol (surr)	330	332,141

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Attachment VI - QC Acceptance Criteria for Method 625

Compound	Test conc. (µg/L)	Limit for s (µg/L)	Range for x (µg/L)	Range p, p(s) (%)
Acenaphthene	100	27.6	60.1-132.3	47-145
Acenaphthylene	100	40.2	53.5-126.0	33-145
Aldrin	100	39	7.2-152.2	D-166
Anthracene	100	32	43.4-118.0	27-133
Benz(a)anthracene	100	27.6	41.8-133.0	33-143
Benzo(b)fluoranthene	100	38.8	42.0-140.4	24-159
Benzo(k)fluoranthene	100	32.3	25.2-145.7	11-162
Benzo(a)pyrene	100	39	31.7-148.0	17-163
Benzo(g,h,i)perylene	100	58.9	D-195.0	D-219
Benzyl butyl phthalate	100	23.4	D-139.9	D-152
beta-BHC	100	31.5	41.5-130.6	24-149
delta-BHC	100	21.6	D-100.0	D-110
Bis(2-chloroethyl) ether	100	55	42.9-126.0	12-158
Bis(2-chloroethoxy)methane	100	34.5	49.2-164.7	33-184
Bis(2-chloroisopropyl) ether	100	46.3	62.8-138.6	36-166
Bis(2-ethylhexyl) phthalate	100	41.1	28.9-136.8	8-158
4-Bromophenyl phenyl ether	100	23	64.9-114.4	53-127
2-Chloronaphthalene	100	13	64.5-113.5	60-118
4-Chlorophenyl phenyl ether	100	33.4	38.4-144.7	25-158
Chrysene	100	48.3	44.1-139.9	17-168
4,4'-DDD	100	31	D-134.5	D-145
4,4'-DDE	100	32	19.2-119.7	4-136
4,4'-DDT	100	61.6	D-170.6	D-203
Dibenzo(a,h)anthracene	100	70	D-199.7	D-227
Di-n-butyl phthalate	100	16.7	8.4-111.0	1-118
1,2-Dichlorobenzene	100	30.9	48.6-112.0	32-129
1,3-Dichlorobenzene	100	41.7	16.7-153.9	D-172
1,4-Dichlorobenzene	100	32.1	37.3-105.7	20-124
3,3'-Dichlorobenzidine	100	71.4	8.2-212.5	D-262
Dieldrin	100	30.7	44.3-119.3	29-136
Diethyl phthalate	100	26.5	D-100.0	D-114
Dimethyl phthalate	100	23.2	D-100.0	D-112
2,4-Dinitrotoluene	100	21.8	47.5-126.9	39-139
2,6-Dinitrotoluene	100	29.6	68.1-136.7	50-158
Di-n-octyl phthalate	100	31.4	18.6-131.8	4-146
Endosulfan sulfate	100	16.7	D-103.5	D-107
Endrin aldehyde	100	32.5	D-188.8	D-209
Fluoranthene	100	32.8	42.9-121.3	26-137

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Compound	Test conc. (µg/L)	Limit for s (µg/L)	Range for x (µg/L)	Range p, p(s) (%)
Fluorene	100	20.7	71.6-108.4	59-121
Heptachlor	100	37.2	D-172.2	D-192
Heptachlor epoxide	100	54.7	70.9-109.4	26.155
Hexachlorobenzene	100	24.9	7.8-141.5	D-152
Hexachlorobutadiene	100	26.3	37.8-102.2	24-116
Hexachloroethane	100	24.5	55.2-100.0	40-113
Indeno(1,2,3-cd)pyrene	100	44.6	D-150.9	D-171
Isophorone	100	63.3	46.6-180.2	21-196
Naphthalene	100	30.1	35.6-119.6	21-133
Nitrobenzene	100	39.3	54.3-157.6	35-180
N-Nitrosodi-n-propylamine	100	55.4	13.6-197.9	D-230
Aroclor 1260	100	54.2	19.3-121.0	D-164
Phenanthrene	100	20.6	65.2-108.7	54-120
Pyrene	100	25.2	69.6-100.0	52-115
1,2,4-Trichlorobenzene	100	28.1	57.3-129.2	44-142
4-Chloro-3-methylphenol	100	37.2	40.8-127.9	22-147
2-Chlorophenol	100	28.7	36.2-120.4	23-134
2,4-Chlorophenol	100	26.4	52.5-121.7	39-135
2,4-Dimethylphenol	100	26.1	41.8-109.0	32-119
2,4-Dinitrophenol	100	49.8	D-172.9	D-191
2-Methyl-4,6-dinitrophenol	100	93.2	53.0-100.0	D-181
2-Nitrophenol	100	35.2	45.0-166.7	29-182
4-Nitrophenol	100	47.2	13.0-106.5	D-132
Pentachlorophenol	100	48.9	38.1-151.8	14-176
Phenol	100	22.6	16.6-100.0	5-112
2,4,6-Trichlorophenol	100	31.7	52.4-129.2	37-144

(s) = Standard deviation of four recovery measurements, in µg/L

(x) = Average recovery for four recovery measurements, in µg/L

(p, p(s)) = Measured percent recovery

(D) = Detected; result must be greater than zero

(a) = Criteria from 40 CFR Part 136 for Method 625, using a packed GC column. These criteria are based directly on the method performance data. Where necessary, the limits for recovery have been broadened to assure applicability of the limits to concentrations below those used to develop method performance data. These values are for guidance only. Appropriate derivation of acceptance criteria for capillary columns should result in much narrower ranges. See Method 8000 for information on developing and updating acceptance criteria for method performance.

TITLE: SEMI-VOLATILE ORGANICS BY GAS CHROMATOGRAPHY/MASS SPECTROMETRY USING CAPILLARY COLUMN (EPA METHOD 8270C, EPA 8270D, EPA METHOD 625, SM 6410B, 20TH ED.), INCLUDING PROVISIONS FOR ANALYSIS IN SIM MODE.

Attachment VII - BNA Poor Performing Compounds

The following compounds are considered to be poor performing compounds.
Pyridine
Aniline
Benzoic Acid
n-Nitrosodimethylamine
Hexachlorocyclopentadiene
4-Chloroaniline
2-Nitroaniline
3-Nitroaniline
4-Nitroaniline
2,4-Dinitro-2-methylphenol
Pentachlorophenol
Carbazole
Benzidine
Atrazine
Acetophenone
Caprolactam
Benzaldehyde
1,2,4,5-Tetrachlorobenzene

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SOP/Document REVISION FORM

02/06/07 R.1.0

SOP/DOC# 330345 Current revision date & number: Rev 15, 6/12/12

Procedure/Method : Semi-Volatile Organics by Gas Chromatography/Mass Spectrometry using Capillary Column (EPA 8270C, EPA 8270D, EPA Method 625, SM 6410B, 20th Ed.), Including Provisions for Analysis in SIM Mode

Date	Analyst	Section	Revision	Reason*	Approvals	
					Supervisor	QA
12/10/12	SLP	8.3.1	Add: **Linear Regression Weighting**: As an alternative to calculating mean response factors and applying the RSD test, use the GC/MS data system software or other available software to generate a linear or second order regression calibration curve, by plotting A/A(is) vs. Q(x) using the equations found in section 9.4. Either equal weighting factors or 1/x regressions may be used.	**		
		11.4	Add: **STATE NOTE**: For South Carolina samples alternate recovery limits can only be used if they are more stringent than method criteria.	**		
		11.11.1	Add South Carolina to the state note regarding 10x criteria for method blank.	**		

12/13/12

*Comments: ** South Carolina audit

APPENDIX B

MGA SOPs

GENERAL FIELD SAMPLING GUIDELINES

SOP#: 2001
DATE: 08/11/94
REV. #: 0.0

1.0 SCOPE AND APPLICATION

The purpose of this Standard Operating Procedure (SOP) is to provide general field sampling guidelines that will assist REAC personnel in choosing sampling strategies, location, and frequency for proper assessment of site characteristics. This SOP is applicable to all field activities that involve sampling.

These are standard (i.e., typically applicable) operating procedures which may be varied or changed as required, dependent on site conditions, equipment limitations or limitations imposed by the procedure. In all instances, the ultimate procedures employed should be documented and associated with the final report.

Mention of trade names or commercial products does not constitute U.S. EPA endorsement or recommendation for use.

2.0 METHOD SUMMARY

Sampling is the selection of a representative portion of a larger population, universe, or body. Through examination of a sample, the characteristics of the larger body from which the sample was drawn can be inferred. In this manner, sampling can be a valuable tool for determining the presence, type, and extent of contamination by hazardous substances in the environment.

The primary objective of all sampling activities is to characterize a hazardous waste site accurately so that its impact on human health and the environment can be properly evaluated. It is only through sampling and analysis that site hazards can be measured and the job of cleanup and restoration can be accomplished effectively with minimal risk. The sampling itself must be conducted so that every sample collected retains its original physical form and chemical composition. In this way, sample integrity is insured, quality assurance standards are maintained, and the sample can accurately represent the larger body of material under investigation.

The extent to which valid inferences can be drawn from a sample depends on the degree to which the sampling effort conforms to the project's objectives. For example, as few as one sample may produce adequate, technically valid data to address the project's objectives. Meeting the project's objectives requires thorough planning of sampling activities, and implementation of the most appropriate sampling and analytical procedures. These issues will be discussed in this procedure.

3.0 SAMPLE PRESERVATION, CONTAINERS, HANDLING, AND STORAGE

The amount of sample to be collected, and the proper sample container type (i.e., glass, plastic), chemical preservation, and storage requirements are dependent on the matrix being sampled and the parameter(s) of interest. Sample preservation, containers, handling, and storage for air and waste samples are discussed in the specific SOPs for air and waste sampling techniques.

4.0 INTERFERENCES AND POTENTIAL PROBLEMS

The nature of the object or materials being sampled may be a potential problem to the sampler. If a material is homogeneous, it will generally have a uniform composition throughout. In this case, any sample increment can be considered representative of the material. On the other hand, heterogeneous samples present problems to the sampler because of changes in the material over distance, both laterally and vertically.

Samples of hazardous materials may pose a safety threat to both field and laboratory personnel. Proper health and safety precautions should be implemented when handling this type of sample.

Environmental conditions, weather conditions, or non-target chemicals may cause problems and/or interferences when performing sampling activities or when sampling for a specific parameter. Refer to the specific SOPs for sampling techniques.

5.0 EQUIPMENT/APPARATUS

The equipment/apparatus required to collect samples must be determined on a site specific basis. Due to the wide variety of sampling equipment available, refer to the specific SOPs for sampling techniques which include lists of the equipment/apparatus required for sampling.

6.0 REAGENTS

Reagents may be utilized for preservation of samples and for decontamination of sampling equipment. The preservatives required are specified by the analysis to be performed. Decontamination solutions are specified in ERT SOP #2006, Sampling Equipment Decontamination.

7.0 PROCEDURE

7.1 Types of Samples

In relation to the media to be sampled, two basic types of samples can be considered: the environmental sample and the hazardous sample.

Environmental samples are those collected from streams, ponds, lakes, wells, and are off-site samples that are not expected to be contaminated with hazardous materials. They usually do not require the special handling procedures typically used for concentrated wastes. However, in certain instances, environmental samples can contain elevated concentrations of pollutants and in such cases would have to be handled as hazardous samples.

Hazardous or concentrated samples are those collected from drums, tanks, lagoons, pits, waste piles, fresh spills, or areas previously identified as contaminated, and require special handling procedures because of their potential toxicity or hazard. These samples can be further subdivided based on their degree of hazard; however, care should be taken when handling and shipping any wastes believed to be concentrated regardless of the degree.

The importance of making the distinction between environmental and hazardous samples is two-fold:

(1) Personnel safety requirements: Any sample thought to contain enough hazardous materials to pose a safety threat should be designated as hazardous and handled in a manner which ensures the safety of both field and laboratory personnel.

(2) Transportation requirements: Hazardous samples must be packaged, labeled, and shipped according to the International Air Transport Association (IATA) Dangerous Goods Regulations or Department of Transportation (DOT) regulations and U.S. EPA guidelines.

7.2 Sample Collection Techniques

In general, two basic types of sample collection techniques are recognized, both of which can be used for either environmental or hazardous samples.

Grab Samples

A grab sample is defined as a discrete aliquot representative of a specific location at a given point in time. The sample is collected all at once at one particular point in the sample medium. The representativeness of such samples is defined by the nature of the materials being sampled. In general, as sources vary over time and distance, the representativeness of grab samples will decrease.

Composite Samples

Composites are nondiscrete samples composed of more than one specific aliquot collected at various sampling locations and/or different points in time. Analysis of this type of sample produces an average value and can in certain instances be used as an alternative to analyzing a number of individual grab samples and calculating an average value. It should be noted, however, that compositing can mask problems by diluting isolated concentrations of some hazardous compounds below detection limits.

Compositing is often used for environmental samples and may be used for hazardous samples under certain conditions. For example, compositing of hazardous waste is often performed after compatibility tests have

been completed to determine an average value over a number of different locations (group of drums). This procedure generates data that can be useful by providing an average concentration within a number of units, can serve to keep analytical costs down, and can provide information useful to transporters and waste disposal operations.

For sampling situations involving hazardous wastes, grab sampling techniques are generally preferred because grab sampling minimizes the amount of time sampling personnel must be in contact with the wastes, reduces risks associated with compositing unknowns, and eliminates chemical changes that might occur due to compositing.

7.3 Types of Sampling Strategies

The number of samples that should be collected and analyzed depends on the objective of the investigation. There are three basic sampling strategies: random, systematic, and judgmental sampling.

Random sampling involves collection of samples in a nonsystematic fashion from the entire site or a specific portion of a site. Systematic sampling involves collection of samples based on a grid or a pattern which has been previously established. When judgmental sampling is performed, samples are collected only from the portion(s) of the site most likely to be contaminated. Often, a combination of these strategies is the best approach depending on the type of the suspected/known contamination, the uniformity and size of the site, the level/type of information desired, etc.

7.4 QA Work Plans (QAWP)

A QAWP is required when it becomes evident that a field investigation is necessary. It should be initiated in conjunction with, or immediately following, notification of the field investigation. This plan should be clear and concise and should detail the following basic components, with regard to sampling activities:

- Objective and purpose of the investigation.
- Basis upon which data will be evaluated.
- Information known about the site including location, type and size of the facility, and length of operations/abandonment.
- Type and volume of contaminated material, contaminants of concern (including concentration), and basis of the information/data.
- Technical approach including media/matrix to be sampled, sampling equipment to be used, sample equipment decontamination (if necessary), sampling design and rationale, and SOPs or description of the procedure to be implemented.
- Project management and reporting, schedule, project organization and responsibilities, manpower and cost projections, and required deliverables.
- QA objectives and protocols including tables summarizing field sampling and QA/QC analysis and objectives.

Note that this list of QAWP components is not all-inclusive and that additional elements may be added or altered depending on the specific requirements of the field investigation. It should also be recognized that although a detailed QAWP is quite important, it may be impractical in some instances. Emergency responses and accidental spills are prime examples of such instances where time might prohibit the development of site-specific QAWPs prior to field activities. In such cases, investigators would have to rely on general guidelines and personal judgment, and the sampling or response plans might simply be a strategy based on preliminary information and finalized on site. In any event, a plan of action should be developed, no matter how concise or informal, to aid investigators in maintaining a logical and consistent order to the implementation of their task.

7.5 Legal Implications

The data derived from sampling activities are often introduced as critical evidence during litigation of a hazardous waste site cleanup. Legal issues in which sampling data are important may include cleanup cost recovery, identification of pollution sources and responsible parties, and technical validation of remedial design methodologies. Because of the potential for involvement in legal actions, strict adherence to technical and administrative SOPs is essential during both the development and implementation of sampling activities.

Technically valid sampling begins with thorough planning and continues through the sample collection and analytical procedures. Administrative requirements involve thorough, accurate

documentation of all sampling activities. Documentation requirements include maintenance of a chain of custody, as well as accurate records of field activities and analytical instructions. Failure to observe these procedures fully and consistently may result in data that are questionable, invalid and non-defensible in court, and the consequent loss of enforcement proceedings.

8.0 CALCULATIONS

Refer to the specific SOPs for any calculations which are associated with sampling techniques.

9.0 QUALITY ASSURANCE/ QUALITY CONTROL

Refer to the specific SOPs for the type and frequency of QA/QC samples to be analyzed, the acceptance criteria for the QA/QC samples, and any other QA/QC activities which are associated with sampling techniques.

10.0 DATA VALIDATION

Refer to the specific SOPs for data validation activities that are associated with sampling techniques.

11.0 HEALTH AND SAFETY

When working with potentially hazardous materials, follow U.S. EPA, OSHA, and corporate health and safety procedures.

STANDARD OPERATING PROCEDURE

ONE

FIELD SCREENING

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1.0 FIELD SCREENING OF SOILS

The South Dakota Department of Environment and Natural Resources has developed a field soil sampling and screening method patterned after the Minnesota Pollution Control Agency and research performed by John J. Fitzgerald. This methodology was developed for field soil screening to generate consistency and reliability of results when using PID/FID instruments. **Soil samples collected for field soil screening may not be used for laboratory analysis. Separate soil samples must be collected according to the soil sampling protocols outlined in this handbook.** The field soil sampling and screening procedures shall include the following:

1. Samples collected during drilling procedures must be taken in advance of the drill bit or auger.

2. No samples shall be collected from the auger flights.

3. When using PID/FID instrument the following procedure must be used:

   A. Half-fill **either** a glass jar, or a plastic whirl pack bag or Ziploc® baggie.

      1) When using glass jars:

         a. Fill jars with a total capacity of 8 oz. or 16 oz. Preferably 16 oz.

         b. Seal each jar with one (1) or two (2) sheets of aluminum foil with the screw cap applied to secure the aluminum foil.

      2) When using whirl pack bags or Ziploc® baggies:

         a. Half fill whirl pack bags from the split spoon or the excavation.

         b. Whirl and tie or zip to close.

   B. Vigorously shake the sample jars or bags for at least thirty (30) seconds once or twice in a 10-15 minute period to allow for headspace development.

   C. If ambient temperatures are below 32^0 Fahrenheit (0^0 Celsius) headspace development is to be within a heated vehicle or building.

   D. Quickly insert the PID/FID sampling probe through the aluminum foil. If plastic bags are used, unzip the corner of the bag approximately one to two inches and insert the probe or insert the probe through the plastic. Record the maximum meter response (should be within the first 2-5 seconds). Erratic responses should be discounted as a result of high organic vapor concentrations or conditions of elevated headspace moisture.

   E. Record headspace screening data from both jars or bags for comparison.

   F. PID/FID instruments shall be operated and calibrated to yield "total organic vapors" in parts per million as benzene. PID instruments should be operated with a 10.2 eV

lamp source. Calibration must be checked/adjusted daily. In addition, all manufacturers' requirements for instrument calibration must be followed.

G. If sample jars are to be re-used in the field, jars must be cleaned according to field decontamination procedures for cleaning of bailers. In addition, headspace readings must be taken to ensure no residual organic vapors exist in the cleaned sample jars. Plastic bags may not be re-used.

H. Any deviation(s) from these procedures must be noted and a basis stated for the deviation(s), with consideration of acceptance by the Department.

2.0 DETERMINATION OF ANALYTICAL RESULTS USING A MOBILE GAS CHROMATOGRAPH

Mobile Gas Chromatographs (GCs) may be used in the field to determine analytical contaminant levels of soil and groundwater. The Department will accept analytical results generated in this manner with the following conditions:

1. At least 20% of the samples analyzed by field GC are split and sent to a fixed laboratory for confirmation using an equivalent analytical method.

2. Acceptable analytical methods are used:

For Soil Samples -

	Method No.
Diesel Fuel Constituents (PAH Screen)	EPA 8100 or Equivalent
Gasoline Constituents (BTEX)	EPA 8020, 8015 or Equivalent
Waste Oil Constituents	SEE NOTE (A)
Total Petroleum Hydrocarbons (for hydrocarbon of concern)	California/USGS Method or Equivalent

For Water Samples -

Diesel Fuel Constituents (PAH Screen)	EPA 8100, 610
Gasoline Constituents (BTEX)	EPA 8020, 8015, 602 or Equivalent
Waste Oil Constituents	SEE NOTE
Total Petroleum Hydrocarbons (for hydrocarbons of concern)	California/USGS Method or Equivalent

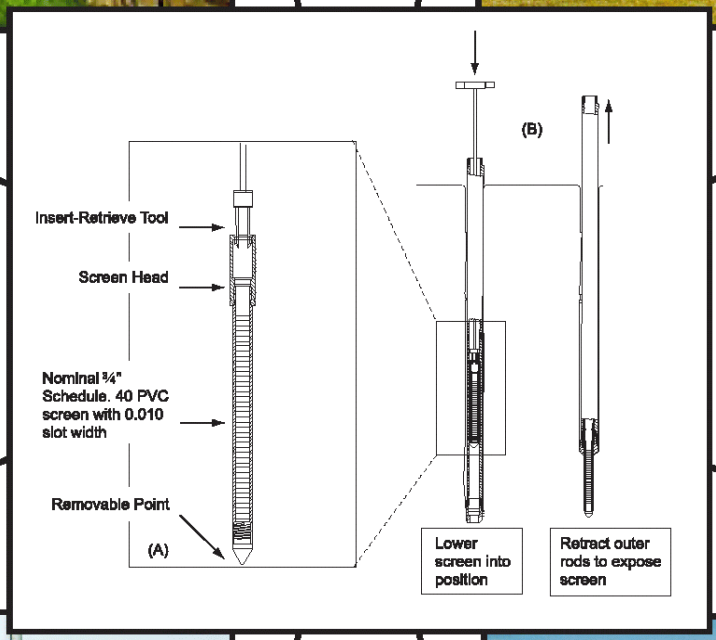
3. The integrity of the samples is protected using all Department recommended sampling, containment, shipping, etc.., procedures outlined in this handbook.

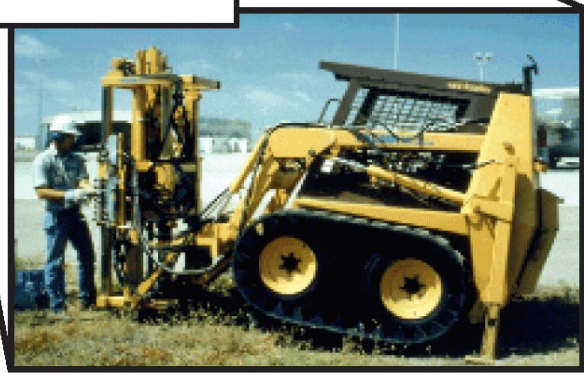
(A) NOTE: Should be based on contents of waste oil spill (i.e. solvents, metals, etc).

3.0 OTHER FIELD SCREENING METHODS

Other field screening methods will be considered by the Department for use at sites. All other field screening methods must follow the manufacture's instructions.

Groundwater Sampling and Monitoring with Direct Push Technologies





Solid Waste and
Emergency Response
(5204G)

OSWER No. 9200.1-51
EPA 540/R-04/005
August 2005
www.epa.gov

Groundwater Sampling and Monitoring with Direct Push Technologies

U.S. Environmental Protection Agency
Office of Solid Waste and Emergency Response
Washington, DC 20460

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Abbreviations

ASTM	American Society for Testing and Materials
CPT	cone penetrometer testing
CSP	centrifugal submersible pump
DCE	dichloroethene
DNAPL	dense non-aqueous phase liquid
DO	dissolved oxygen
DPT	direct push technology
DQO	data quality objectives
FID	flame ionization detector
GC	gas chromatograph
ID	inner diameter
LIF	laser-induced fluorescence
LNAPL	light non-aqueous phase liquid
MIP	membrane interface probe
OD	outer diameter
ORP	oxidation/reduction potential
PAH	polycyclic aromatic hydrocarbon
PCB	polychlorinated biphenyl
PE	polyethylene
PP	polypropylene
PTFE	polytetrafluoroethylene
PVC	polyvinyl chloride
QC	quality control
RCRA	Resource Conservation and Recovery Act
ROST	Rapid Optical Screening Tool
SC	specific conductivity
SVOC	semi-volatile organic compound
TCE	trichloroethene
EPA	United States Environmental Protection Agency
UV	ultraviolet
VOC	volatile organic compound

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Section 1: Introduction

Background

Direct push technology (DPT, also known as "direct drive," "drive point," or "push technology") refers to a growing family of tools used for performing subsurface investigations by driving, pushing, and/or vibrating small-diameter hollow steel rods into the ground. By attaching sampling tools to the end of the steel rods they can be used to collect soil, soil-gas, and groundwater samples. DPT rods can also be equipped with probes that provide continuous in-situ measurements of subsurface properties (e.g., geotechnical characteristics and contaminant distribution). Interest in understanding how DPT groundwater collection methods compare with traditional monitoring well sampling methods has steadily increased since the mid-1980s when DPT first started being used for this purpose. Although environmental professionals recognize that DPT provide a cost-effective alternative to conventional approaches to subsurface sampling, some have been reluctant to use it for groundwater sampling because of uncertainty regarding the quality of samples that the technology can provide. This guidance is designed to encourage more widespread consideration of DPT by clarifying how DPT can be used to meet a variety of data quality requirements for a variety of site conditions.

Intended Audience

The primary audience for this guidance is EPA regional folks working on CERCLA, RCRA, and other related programs. It also may be useful for environmental professionals who oversee or undertake the collection of groundwater samples at contaminated sites and have a basic scientific understanding of groundwater sampling. Information is provided on the application and limitations of DPT for groundwater sampling activities. Although this document is not intended to provide substantial background information, Section 2 provides a general overview of DPT groundwater sampling and an extensive list of resources is cited within the text and listed in the reference section.

Scope and Limitations

This document focuses on groundwater sampling issues related to DPT, in particular those regarding the quality and usability of the groundwater data. Two general types of DPT groundwater sampling methods are discussed: "point-in-time" or "grab" sampling and sampling with direct push installed monitoring wells. In order to provide a concise and readable document, references are provided so that readers can access more detailed information where needed. Other uses of DPT, such as soil sampling, soil-gas sampling, and deployment of continuous logging equipment, generally are not controversial; therefore, they are not discussed at length. In addition, this guidance assumes a basic level of understanding of DPT equipment. Readers unfamiliar with DPT equipment should refer to:

- Expedited Site Assessment Tools for Underground Storage Tank Sites: A Guide for Regulators (EPA, 1997). Chapter V of this guide, Direct Push Technologies, provides a

good overview of the tools and their capabilities. It is available at: http://www.epa.gov/swerust1/pubs/sam.htm.

- The Field Analytical Technology Encyclopedia (FATE) contains a section on Direct Push Platforms. It is available at: http://fate.clu-in.org.

- ASTM direct push standards, Standard Guide for Installation of Direct Push Ground Water Monitoring Wells, D 6724-01; Standard Practice for Direct Push Installation of Prepacked Screen Monitoring Wells in Unconsolidated Aquifers, D 6725-01; Standard Guide for Direct-Push Water Sampling for Geoenvironmental Investigations, D-6001; and Standard Guide for Direct Push Soil Sampling for Environmental Site Characterization, D-6282. They are available for purchase at: http://www.astm.org.

This guidance is not intended to replace the knowledge and advice of an experienced hydrogeologist. Site-specific situations may dictate that an expert familiar with site conditions and project goals be involved in the planning and implementation of any groundwater sampling event. Furthermore, Federal and State regulatory requirements can vary substantially among jurisdictions and the appropriate regulatory and State agencies must be consulted to ensure that legal requirements are met.

Advantages and Limitations of Direct Push Technologies

Direct push technologies are a valuable tool for environmental investigations because they can offer a number of advantages over conventional well installation and sampling methods and can provide many other types of data to a project team (e.g., in-situ detection of contaminants, real-time geotechnical data). Some of the typical advantages of using DPT over monitoring wells drilled and installed with conventional tools, such as hollow stem augers, include:

- Faster sampling capability that helps to provide more data, thereby improving site decision making and facilitates the use of a dynamic work plan strategy;
- In general, lower cost when greater data density is needed;
- Greater variety of equipment and methods resulting in greater flexibility in meeting project goals;
- Capability of collecting depth-discrete groundwater samples to locate contaminated layers;
- Better vertical profiling capability for generating three-dimensional profiles of a site that improve the conceptual site model; and
- Less investigation-derived waste generated, thereby saving additional time and money while minimizing the potential for exposure to hazardous substances.

However, DPT cannot completely replace the use of conventional monitoring wells. Rather, DPT provides environmental professionals with additional choices from which to select equipment and methods for collecting groundwater samples. Conventional methods still have a number of potential advantages over DPT, including:

- Fewer limitations for deployment in a variety of geologic and hydrogeologic settings. For example, conventional DPT may not be able to penetrate some caliches, bedrock, or unconsolidated layers with significant amounts of gravel or cobbles. DPT is not recommended where telescoped wells are needed to prevent contaminant migration below confining layers;
- Deeper limit of subsurface penetration than DP rigs in most geologic settings; and
- Easier collection of large sample volumes.

Consequently, DPT and conventional monitoring well technologies may both be useful for groundwater sampling. They can provide environmental professionals with a variety of options to collect data sufficient for decision making, even when high quality groundwater samples are needed.

How to Use This Guidance

This guidance is divided into four major sections designed to expose the reader to potential issues and solutions regarding groundwater sampling with DPT:

- *Section 2: Summary of Direct Push Technology Groundwater Sampling Methods*: provides an overview of the different types of equipment available with DPT to collect groundwater.
- *Section 3: Data Quality Objectives for Groundwater Sampling*: provides the reader with a summary of groundwater data quality issues that should be considered while planning a groundwater sample collection activity.
- *Section 4: Recommended Methods for Collecting Representative Groundwater Samples*: provides the reader with information on filter packs, well development, and low-flow sampling methods as they relate to DPT.
- *Section 5: Recommended Methods for Minimizing the Potential for Cross-Contamination*: provides the reader with information on drag-down, hydraulic conduits, decontaminating equipment, and decommissioning DPT boreholes.

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Section 2: Summary of Direct Push Technology Groundwater Sampling Methods

DPT groundwater sampling equipment generally falls into one of two broad categories:

- **Point-in-time groundwater samplers:** These tools or devices, also referred to as "temporary samplers" or "grab samplers," are used to rapidly collect samples to define groundwater conditions during one sampling event. They are usually less than two inches outside diameter (OD) and are generally constructed of steel or stainless steel. Direct push methods (percussion or static weight) are used to advance point-in-time samplers below the static water level in unconsolidated formations. Generally, groundwater flows into the sampler from an exposed screen under ambient hydrostatic pressure. Groundwater may be collected from the sampler using bailers or pumps, or the sampler may be retracted to the surface to obtain the water sample. Once sampling is completed, these devices are removed and the boring should be abandoned in accordance with local regulations.

- **DPT-installed groundwater monitoring wells:** These monitoring wells are installed by direct push methods to permit short-term or long-term monitoring of groundwater and are usually two inches in diameter or less and constructed of PVC and/or stainless steel. Since monitoring wells are installed for periods of several months to several years, the annulus of the boring around the well casing is usually sealed to prevent migration of contaminants into the aquifer. Surface protection is required to prevent tampering with the well. A slotted or screened section permits groundwater to flow into the well under ambient hydrostatic pressure. Groundwater may be collected from monitoring wells using bailers, various pumps, or passive sampling devices.

Point-in-time sampling tools are typically used during site characterization to identify plume boundaries or hot spots. They cannot be used for long-term monitoring or trend analysis since the boreholes need to be decommissioned upon completion of sampling. In contrast, temporary and permanent monitoring wells are typically used to provide trend analysis of contaminant groundwater concentrations over an extended period of time. DPT can be used to install small-diameter (e.g., up to 2 inches outside diameter [OD]) monitoring wells.

Ideally, both DPT point-in-time and monitoring well groundwater sampling equipment should be used together to maximize their effectiveness. Point-in-time sampling techniques are generally better for identifying plume boundaries, hot spots, preferred pathways, or other monitoring points of interest. Once this information is collected, DPT monitoring wells, as well as conventional monitoring wells, can be optimally placed to provide project teams with the most useful monitoring data.

This section summarizes the various types of point-in-time sampling tools and DPT monitoring well installation techniques. Since DPT groundwater sampling methods are often used to their best advantage in combination with other specialized DPT measurement and logging tools, these associated tools are also discussed at the end of this section. This information is intended to provide the reader with an easily accessible summary of available

DPT tools. Other resources listed in the *Introduction* could also be used for a more comprehensive survey of available tools and their capabilities. Table 2.1 provides a list of some of the major DPT equipment available for groundwater investigations. It summarizes the capabilities of the equipment and helps the reader to sort through the variety of tools and how they may be useful for specific project goals. Because this section provides a basic overview of existing equipment, readers already familiar with the capabilities of DPT equipment may wish to proceed to Section 3 for information on how these tools can be used to collect groundwater samples to meet project objectives.

Point-in-Time Sampling

A variety of point-in-time groundwater sampling tools are available for site characterization, including:

- Sealed-screen sampling;
- Multi-level sampling (or vertical profiling); and
- Open-hole sampling.

With these techniques, the time needed to retrieve the sample will vary according to the hydraulic conductivity of the sampling zone. In general, sampling within coarse-grained sediments takes minutes while fine-grained sediments can take several hours or more. In situations where slow recharge inhibits the timely collection of groundwater samples, the sampler may be left in place to recharge while the DPT rig is moved to a new sampling location.

Sealed-Screen Samplers

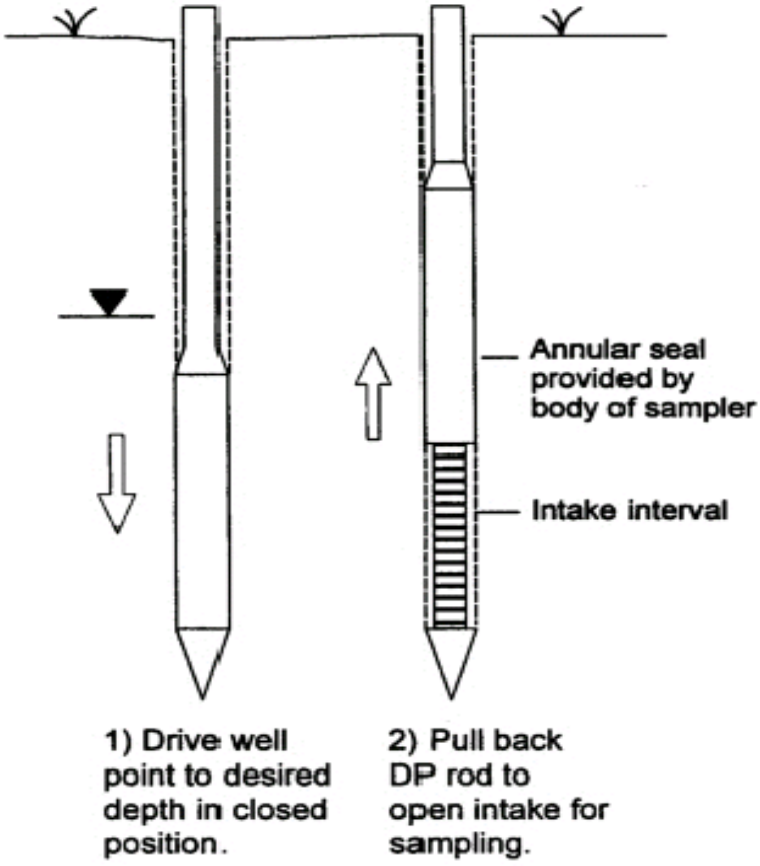
Sealed-screen samplers typically consist of a short (e.g., 6-inch to 3-foot) screen nested within a sealed, water-tight tool body (Figure 2.1). Because the screen is not exposed to the formation as the sampler is advanced into the subsurface, the screen does not become plugged or damaged. In addition, the potential for cross contamination is greatly reduced and a true depth-discrete sample that is representative of the target sampling zone can be collected. The sample volume collected with some sealed-screen samplers is limited by the volume of the sample chamber (e.g., 500 mL for the Hydropunch I™; 1.2 L for the Hydropunch II™; and 35 to 120 mL for each vial in the BAT™).

To collect the sample, the sealed-screen sampler is advanced to the target sampling depth and the protective outer rod is retracted, exposing the screen to groundwater. Groundwater flows through the screen under the hydraulic head conditions that exist at that depth and into the drive rods or sample chamber. O-ring seals placed between the drive tip and the tool body help ensure that the sampler is water tight as it is driven to the target sampling interval. The integrity of the seal can often be checked by lowering an electronic water level indicator into the sampler prior to retracting the protective outer rod.

Table 2.1
Comparison of Various Direct Push Technology Sampling and Data Collection Capabilities

EQUIPMENT	CAPABILITIES											
	Evaluate Strati-graphy	Measure Pore Pressure	Measure Soil Conduc-tivity	Detect Hydrocarbon	Detect VOC	Sample Soil	Sample Soil Gas	Sample Ground-water	Sample Pore Water	Evaluate Vadose Zone	Measure Water Level	Install Small Diameter Wells
Sealed-Screen Samplers												
BAT GMS								✓	✓			
BAT Enviroprobe							✓	✓				
Dual-Tube Samplers	✓					✓	✓	✓		✓	✓	✓
HydroPunch I and II™								✓				
PowerPunch™										✓	✓	✓
Screen Point 15								✓			✓	
SimulProbe®						✓	✓	✓				
Multi-Level Samplers												
Geoprobe	✓		✓		✓	✓	✓	✓		✓	✓	✓
Envirocore	✓					✓	✓	✓		✓	✓	✓
Vertek ConeSipper®	✓	✓	✓				✓	✓		✓	✓	
Waterloo Profiler							✓	✓		✓	✓	
Specialized Measurement and Logging Tools												
Cone Penetrometer	✓	✓	✓			✓	✓	✓		✓	✓	✓
Instrumented CPTs	✓	✓	✓	✓	✓	✓						

Figure 2-1
Sealed-Screen Sampler



Source: Cordry, 1995[1]

Sampling fine-grained formations may be difficult because of the long time it takes to fill the sampler with groundwater. Sample collection times in formations with low hydraulic conductivity may exceed several hours for some tools, compared to several minutes or tens of minutes in formations of high to moderate hydraulic conductivity (Zemo et al., 1994; Zemo et al., 1995). However, to avoid downtime, the samplers can be left in the borehole to recharge while the installing rig moves off the hole to another location to sample. To decrease sample collection time, samples can be collected from samplers with longer, 30- to 42-inch screens (e.g., Geoprobe® Screen Point 15) while the tool is downhole. A bailer or pump is needed to collect the sample from the target zone.

Sealed-screen samplers generally are limited to collecting one sample per advance of the sampler. However, depending upon the system used, multi-level sampling in a single borehole can be accomplished with sealed-screen samplers by retrieving the sampler and decontaminating it or replacing it with a clean sampler before reentering the hole to collect another sample.

Multi-Level Samplers

Multi-level samplers, most of which are exposed-screen samplers, are DPT equipment capable of collecting groundwater samples at multiple intervals as the sampling tool is advanced, without having to withdraw the tool for sample collection or decontamination. The terminal end of a typical multi-level sampling tool has a 6-inch- to 3-foot-long screen made up of fine-mesh, narrow slots, or small holes. The screen remains open to formation materials and water while the tool is advanced (Figure 2.2). This allows samples to be collected either continuously or periodically as the tool is advanced to vertically profile groundwater chemistry and aqueous-phase contaminant distribution.

Multi-level samplers can be used to measure water levels at discrete intervals within moderate- to high-yield formations to assist in defining vertical head distribution and gradient. Additionally, some of these tools can be used to conduct hydraulic tests at specific intervals to characterize the hydraulic conductivity in formation materials to identify possible preferential flow pathways and barriers to flow (Butler et al., 2000; and McCall et al., 2000).

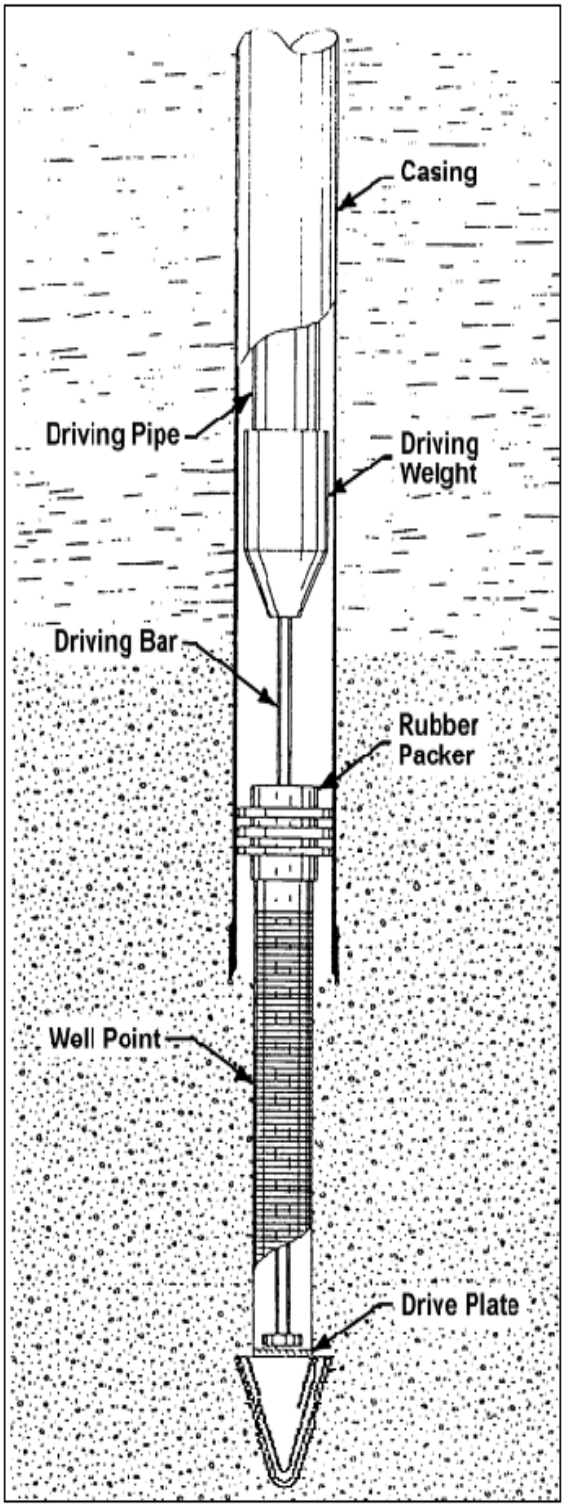
A drawback to multi-level sampling is the possible drag-down by the screen of contamination from zones above the desired sampling interval (Figure 2.3) (Pitkin et al., 1999). The Waterloo Profiler minimizes the potential for cross- contamination. It uses a 6-inch long, uniform diameter, stainless-steel sampling tool into which several inlets or sampling ports have been drilled and covered with fine-mesh screen. As the tool is advanced, distilled or deionized organic-free water is slowly pumped down tubing that runs inside the drive rod and leads to the sampling ports in the tool (Figure 2.4). The water keeps groundwater from entering the tool while it is advanced. A peristaltic pump is typically used for depths less than 25 feet; a double-valve pump can be used for sampling at greater depths.

After the first target interval is reached, the flow of the pump is reversed and the sampling tube is purged so water representative of the aquifer is obtained. After the sample is collected, the pump is reversed and distilled or deionized water is again pumped through the sampling ports. The tool is then advanced to the next target interval where the process is repeated (Figure 2.5).

Several field studies (Cherry, et al., 1992; Pitkin, et al., 1994; Pitkin, et al., 1999) have demonstrated that the Waterloo Profiler is capable of providing a very detailed view of contaminant plumes—particularly in complex stratified geological materials—without the effects of drag-down and the cross contamination of samples. However, because a peristaltic pump is typically used to collect samples when the sampling depth is less than 25 ft below ground surface (bgs), there may be a negative bias in samples collected for analysis of VOCs or dissolved gases. To avoid this potential bias, VOC samples should be collected in-line, ahead of the pump, and a sufficient volume of water should be pumped through the system to account for the initial filling of the containers when a negative head space was present.

Another multi-level sampler, the VERTEK ConeSipper®, attaches directly behind a standard cone penetrometer to collect groundwater as the cone penetrometer testing (CPT) is advanced. An inert gas flows to the ConeSipper® to control the rate of sample collection and to purge and decontaminate the device down hole. The ConeSipper® is equipped with two filters, which help minimize turbidity in the samples. The primary filter is a stainless steel screen whose openings can range in size from 51 to 254 µm. A secondary filter, which can be made from sintered stainless steel and comes with opening sizes ranging from 40 to 100 µm or regular

Figure 2.2
Exposed-Screen Sampler–Well Point Driven below the Base of a Borehole



Source: ASTM (2001e)

Figure 2.3
Schematic Illustration of Degrees of Drag Down Potentially Induced by Direct Push Sampling Devices

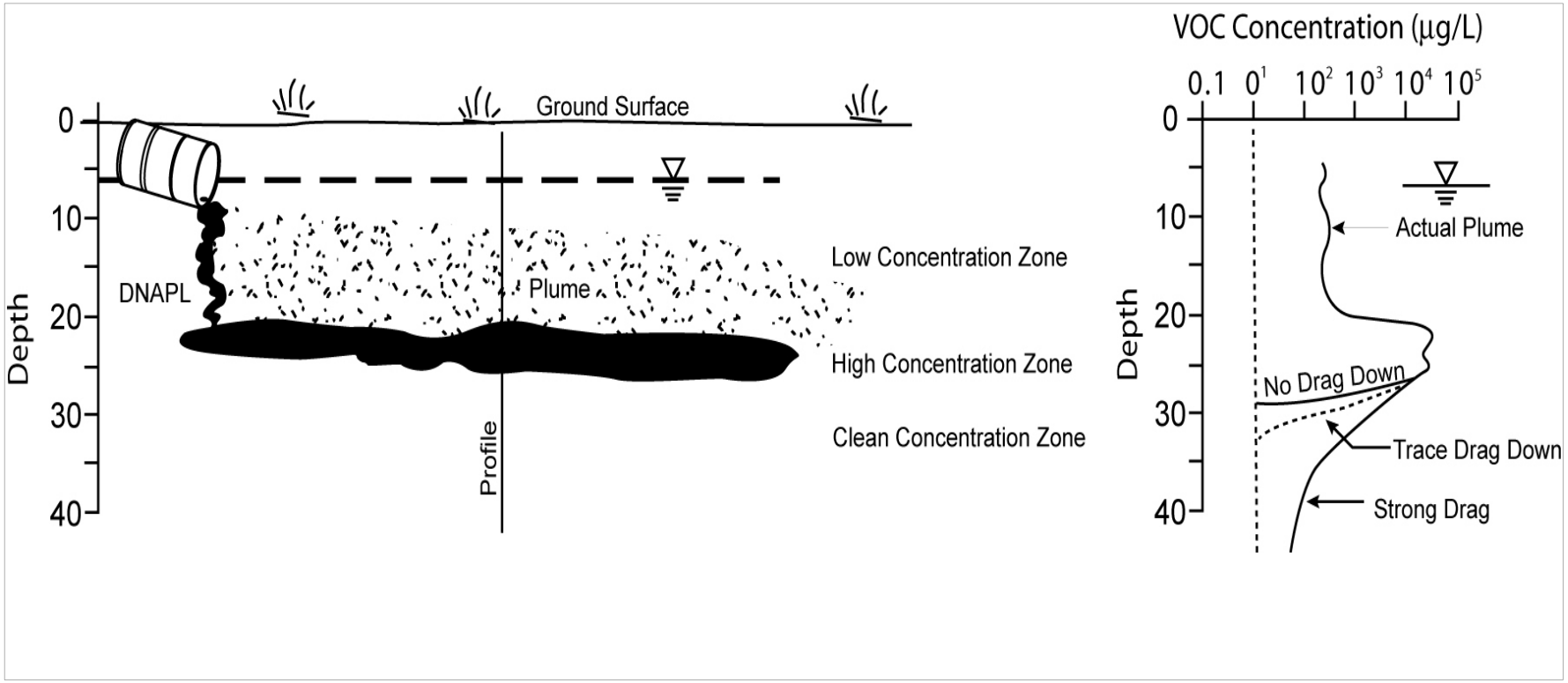
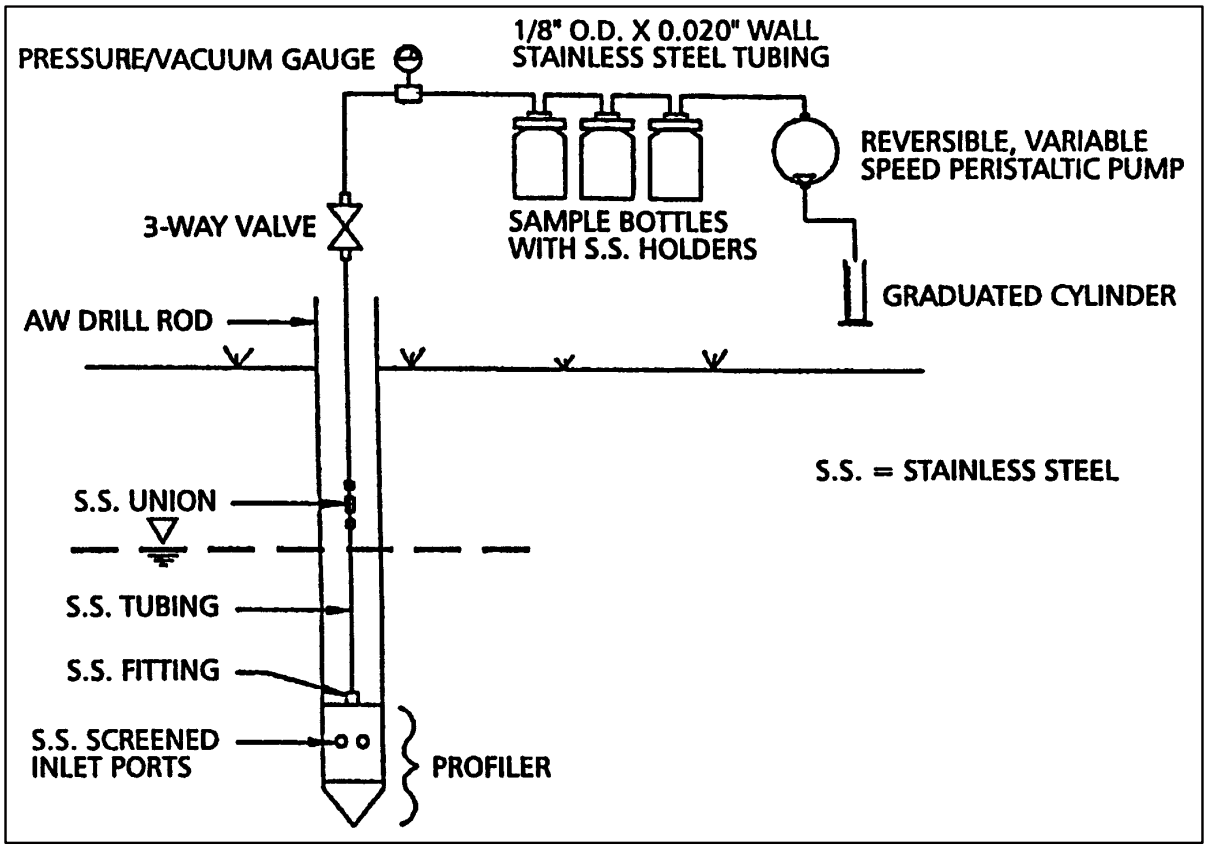


Figure 2.4
Operation of the Waterloo Profiler



Source: Pitkin et al. (1999)

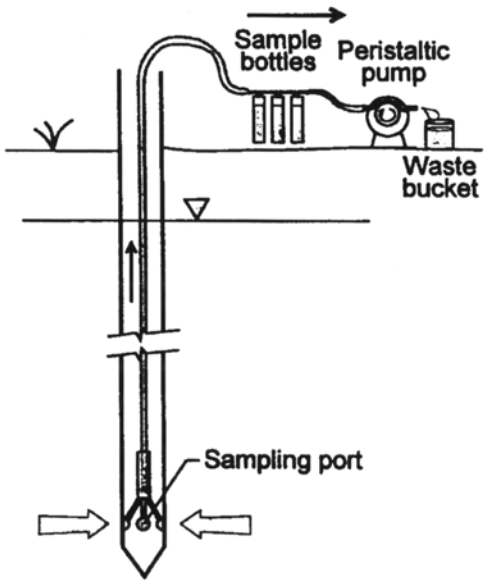
stainless steel with openings ranging in size from 38 to 74 µm, removes fines (Applied Research Associates, 2004).

Open-Hole Sampling Methods

Open-hole sampling is conducted by advancing drive rods with a drive point to the desired sampling depth. Upon reaching the sampling depth, the rods are withdrawn slightly which separates them from the drive tip and allow water to enter the rods. The water can be sampled by lowering a bailer into the rods or by pumping. The open-hole method is only feasible within formations that are fairly cohesive, otherwise the formation may flow upwards into the rods when they are withdrawn, preventing samples from being collected.

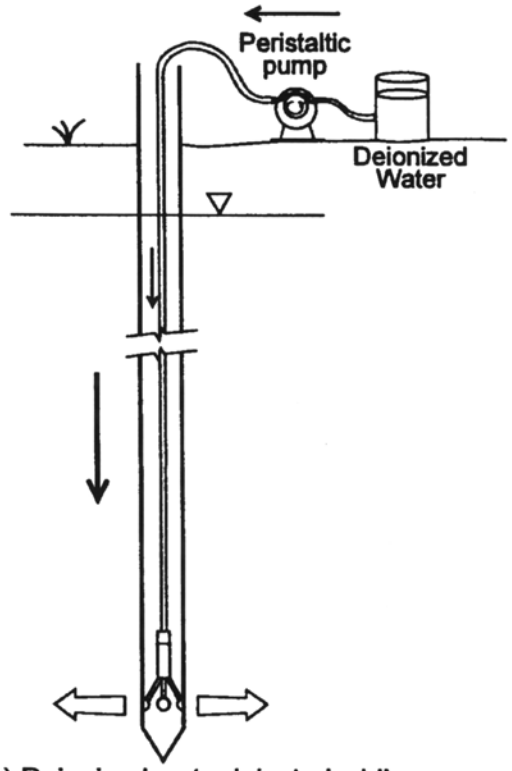
With single-rod systems, open-hole sampling can only be conducted at one depth within a borehole because the borehole cannot be flushed out between sampling intervals and cross-contamination may occur. Dual-tube systems, on the other hand, can be used to conduct multi-level sampling.

Figure 2.5
Collecting Samples From Discrete Depths (Profiling) Using the Waterloo Drive-Point Profiler

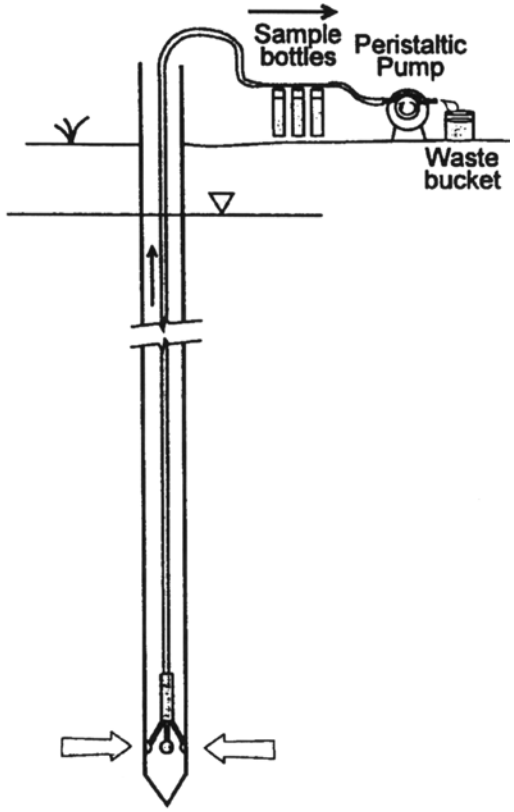


a) Groundwater sample collected from first target zone with peristaltic pump.

Source: Precision Sampling, Inc.



b) Deionized water injected while advancing tool to prevent sampling ports from becoming plugged.



c) Groundwater sample collected from second target zone with peristaltic pump.

Dual-tube samplers are typically advanced into the subsurface to collect continuous soil cores; however, groundwater samples can be collected at the end of each core run. Dual-tube samplers have an outer casing that is driven to the target soil coring depth. The outer casing holds the hole open and seals off the surrounding formation as an inner rod (with a sample liner for soil sampling) is lowered into the outer casing and both are driven into the undisturbed formation below. Once the soil core is retrieved, groundwater can be sampled by lowering a bailer or pump into the outer casing The borehole can continue to be advanced so that multiple groundwater samples can be retrieved from multiple depths in the same borehole. The water should be purged from the casing with subsequent advances of casing and inner rod so that groundwater from overlying intervals do not cross-contaminate the sample.

The amount of water that needs to be purged depends upon the type of sampling equipment that is used. For pumping systems, purging procedures similar to those designed for wells (low-flow purging) and described in Section 4, should be used. If bailers are used, then it is important that all the water contained in the outer casing be removed to ensure that the water the bailer is passing through comes from the interval of interest. The accepted procedure for traditionally completed wells when bailers are used is to remove at least three volumes of water and measure water quality indicators (e.g., pH, specific conductance) until they stabilize. The use of a bailer in this situation may preclude the collection of some parameters that may be sensitive to the iron in the outer casing (See Low-flow Purging Section 4.)

A dual-tube profiling system has been developed so that a simple screen can be inserted through the cutting shoe of a dual-tube soil sampling device (Figure 2.6). This system enables the operator to collect soil samples and then insert a screen at selected intervals, which they can then use for sampling or conducting slug tests to locate preferential migration pathways (Butler et al., 2000; McCall et al., 2000). This system also allows for bottom-up grouting to assure proper boring abandonment.

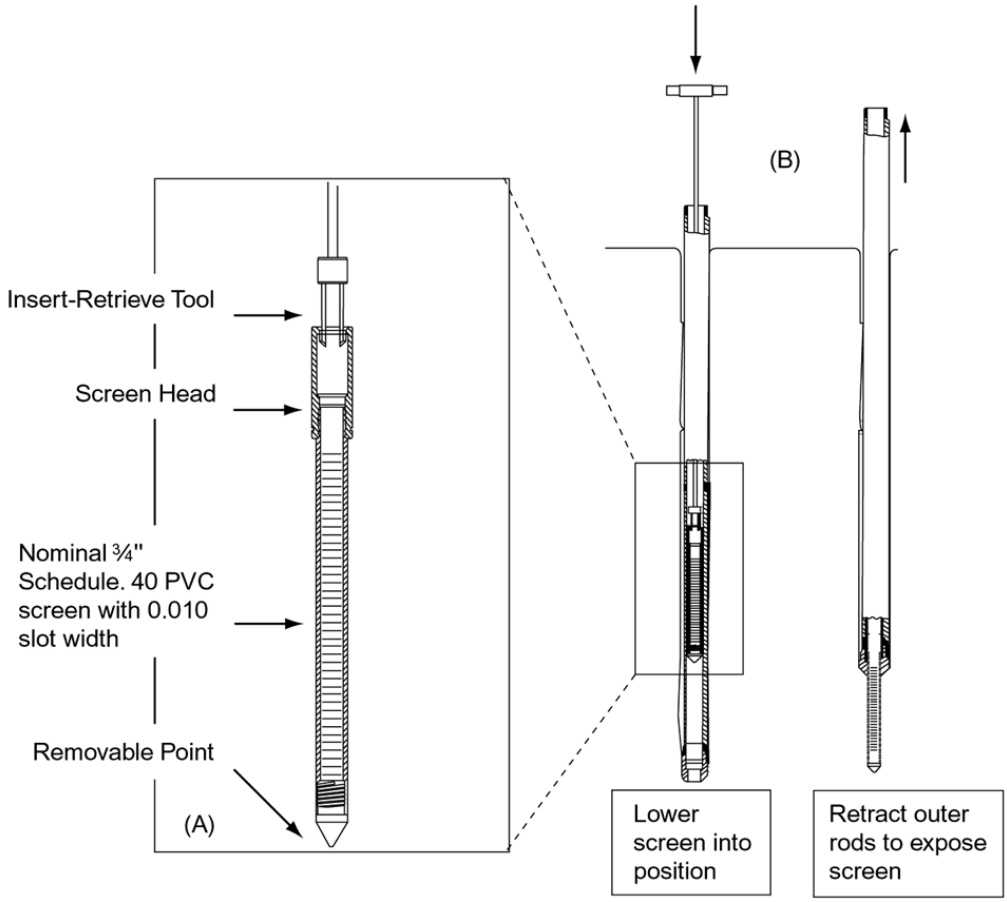
DPT Monitoring Well Installation

A variety of DPT methods are available for installing temporary or permanent monitoring wells. The two main installation methods used are exposed-screen and protected-screen wells. These methods are discussed in detail in ASTM D-6724 and D-6725 (ASTM, 2003a and 2003b) and are summarized here. As with conventional well installations, hydraulic connections should not be created between otherwise isolated water-bearing strata. In addition, precautions should be taken to minimize turbidity during the installation of filter packs and the development and sampling of wells (Section 4).

Exposed-Screen Well Installation Methods

With exposed-screen well installation methods, the well casing and screen are driven to the target depth using a single string of rods. Because the screen is exposed to formation materials while it is advanced, proper well development (as discussed in Section 4) is important to remove soil from screen slots. This method is not recommended for installing well screens within or beneath contaminated zones because drag-down of contaminants with the screen may cross-contaminate sampling zones and make acquisition of samples representative of the target zone impossible. Exposed-screen well installation methods should only be used in upgradient

Figure 2.6
Schematic of the Geoprobe® DT21 Profiler



A) Screen components for insertion through the dual-tube system for slug testing and sampling.
B) The profiling screen is lowered through the outer rods after the inner rods are removed. Once the screen is at the base of the outer rods, they are retracted as the screen is held in position for accurate placement.

Source: www.geoprobe.com

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areas that are known to be uncontaminated. Also, some states prohibit allowing the formation to collapse around a well screen in the construction of a monitoring well**.** Therefore, state regulations should be consulted before selecting exposed-screen techniques.

In one type of exposed-screen installation, the PVC well screen and casing are assembled and placed around a shaft of a drive rod connected to a metal drive tip. The casing and screen, which rest on top of the drive tip, are advanced to the target depth by driving the rod to avoid placing pressure on the screen. The drive tip slightly enlarges the hole to reduce friction between the formation and the well screen and casing, and remains in the hole plugging the bottom of the screen. The filter pack surrounding the well screen commonly is derived from formation materials that are allowed to collapse around the screen. Rigorous well development

improves the hydraulic connection between the screen and the formation and generally is necessary to remove formation fines and the effects of well installation, which may include borehole smearing or the compaction of formation materials. Due to the very small annulus (if any) that surrounds a well constructed using the exposed-screen method, it is not generally possible to introduce a filter pack or annular seal from the surface.

Exposed-screen methods also can be used to install well points—simple wells used for rapid collection of water level data, groundwater samples, and hydraulic test data in shallow unconfined aquifers. Well points are generally constructed of slotted steel pipe or continuous-wrap, wire-wound, steel screens with a tapered tip on the bottom. They can be driven into unconsolidated formations and used for either point-in-time sampling and decommissioned after the sample is collected, or left in place for the duration of the sampling program—possibly requiring the installation of a seal to prevent infiltration of water from the ground surface to the screened interval.

The optimum conditions for well point installations are shallow sandy materials. Predominantly fine-grained materials such as silt or clay can plug the screen slots as the well point is advanced. Because well points are driven directly into the ground with little or no annular space, the formation materials are allowed to collapse around the screen, and the well point needs to be developed to prepare it for sampling.

Protected-Screen Well Installation Methods

When installing a protected-screen well, the well casing and screen are either advanced within or lowered into a protective outer drive rod that has already been driven to the target depth. Once the well casing and screen are in place, the drive rod is removed. Alternatively, the casing, screen, and a retractable shield may be driven simultaneously to the target depth. Once in place, the screen is exposed and the entire unit remains in the ground. If there is sufficient clearance between the inside of the drive rod and the outside of the well casing and screen, a filter pack and annular seal may be installed by tremie from the surface as the drive casing is removed from the hole. Several filter packing and annular sealing approaches are available, depending on the equipment used for the installation (ASTM D5092 and D6725; ASTM, 2003b and 2003c). Regardless of the method of installation, the filter pack should be sized appropriately to retain most of the formation materials (refer to Driscoll, 1986 or ASTM D5092-02).

The most common protected-screen method for installing DPT wells is to advance an outer drive casing equipped with an expendable drive tip to the target depth. The well casing and screen are then assembled, lowered inside the drive casing, and anchored to the drive tip. The drive casing seals off the formations through which it has been advanced, protecting the well casing and screen from clogging and from passing through potentially contaminated intervals. The position and length of the screen should be selected to match the thickness of the monitoring zone, which can be determined by using additional information, such as CPT logs or continuous soil boring logs.

When DPT wells are installed in non-cohesive, coarse-grained formations, the formation can be allowed to collapse around the screen (if this technique is not prohibited by state well installation regulations) after it is placed at the target depth since turbidity problems are unlikely. When turbidity is likely to pose a problem for groundwater sample quality (see Section 3), a

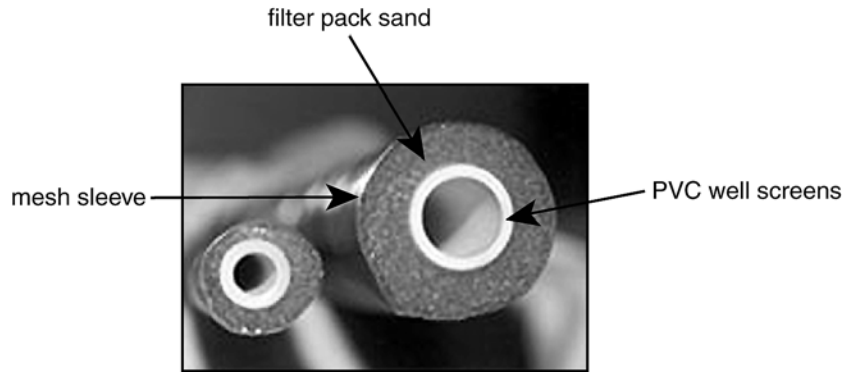
number of methods for installing filter packs are available. The filter pack can be poured or tremied into place as the drive casing is removed. Depending on the relative size of the drive casing and well, however, it may be difficult to introduce filter pack or annular seal materials downhole unless the hole is in a cohesive formation that will remain open as the drive casing is removed. Typical inside diameters of DPT wells range from 0.5-inch (schedule 80 PVC) to 2 inches (schedule 40 PVC), and the maximum inside diameter of drive casing is 3.5 inches. Table 2.2 provides a reference for understanding the relationship between inside diameters of DPT drive casing, the outside diameter of well casing and screen, and the annular space available for filter packs.

For the best control of filter pack placement and grain size, "sleeved" or "prepacked" well screens can be used (Figure 2.7). Pre-packed screens are generally composed of a rigid Type I PVC screen surrounded by a pre-sized filter pack. The filter pack is held in place by a stainless-steel wire mesh (for organic contaminants) or food-grade plastic mesh (for inorganic contaminants), such as polyethylene, that is anchored to the top and bottom of the screen. Sleeved screens consist of a stainless-steel wire mesh jacket filled with a pre-sized filter-pack material, which can be slipped over a PVC pipe base with slots of any size. Although sleeve thickness generally ranges from only 0.25 to 0.5 inch, it has been shown to provide an effective filter pack (Kram et al., 2000).

Table 2.2
Annular Space for Well Completion Based on Size of Well Casing and Screen

Inside Diameter of Well Casing and Screen (inches)	Outside Diameter of Well Casing and Screen (inches)	Annular Space with 1.5-inch Inside Diameter (1.8-inch OD) Drive Casing (inches)	Annular Space with 3-inch Inside Diameter (3.5-inch OD) Drive Casing (inches)
0.5	0.84	0.66	2.16
0.75	1.05	0.45	1.95
1	1.32	0.18	1.68
1.25	1.66	Not applicable	1.34

Figure 2.7
Photograph of Pre-Packed Well Screens



Courtesy Geoprobe® Systems, 1996.

Annular seals and grout should be placed above the filter pack to prevent infiltration of surface runoff and to maintain the hydraulic integrity of confining or semi-confining layers, where present. The sealing method used depends on the formation, the well installation method, and the regulatory requirements of state or local agencies. Most protected-screen installations tremie a high-solids (at least 20% solids) bentonite slurry or neat cement grout into place as the drive casing is removed from the hole. (Additional guidance on grout mixtures is available in ASTM D6725 (ASTM, 2003b).) A barrier of fine sand or granular or pelletized bentonite (where water is present) may be placed above the primary filter pack before grouting to protect it from grout infiltration, which could alter the water chemistry in the screened zone. Similar to the pre-packed and sleeved screens mentioned above, modular bentonite sleeves that attach to the well screens and are advanced with the well during installation are also available. As depicted in Figure 2.8, some manufacturers provide a foam seal that expands immediately when the casing is withdrawn to form a temporary seal above the screen[1]. A bentonite sleeve above the seal expands more slowly after the casing is withdrawn but forms a permanent seal once it hydrates.

To ensure a complete seal of the annular space from the top of the annular seal to the ground surface, the grout or slurry should be placed from the bottom up. By using a high pressure grout pump and nylon tremie tube (Figure 2.9) it is possible to perform bottom-up grouting in the small annular spaces of DPT equipment. Slurries of 20-30% bentonite or neat cement grout are most commonly used to meet state regulatory requirements.

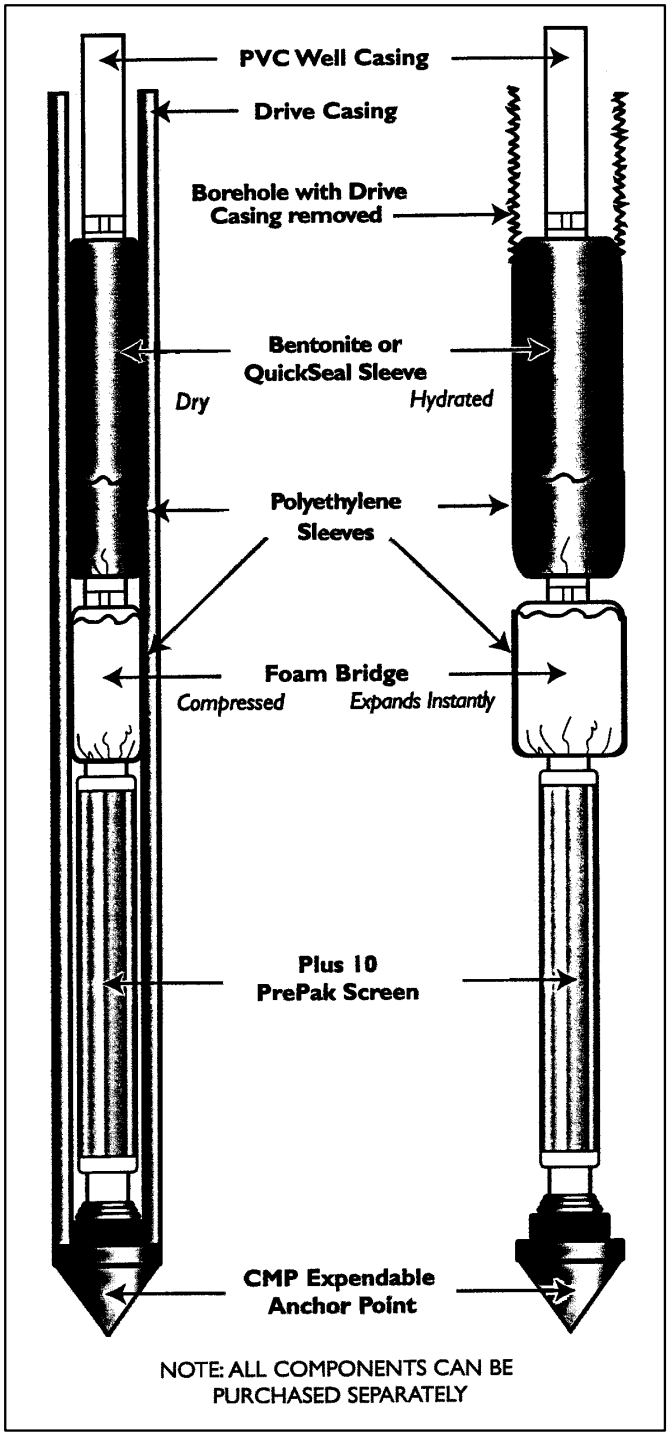
A properly constructed DPT-installed monitoring well (Figure 2.10) can provide representative water quality samples and protect groundwater resources. A 1997 study (McCall et al., 1997) demonstrated that DPT wells installed in this manner beneath highly contaminated source zones consistently provided non-detect values. In addition, as with conventional wells, a properly constructed DPT well should have a flush-mount or above-ground well protection to prevent physical damage or tampering of the well. Small locking well plugs are also available for even 0.5-inch nominal PVC casing.

Specialized Measurement and Logging Tools

There are a number of specialized measurement and logging tools available that can be used with DPT equipment to optimize the number and location of groundwater samples. These tools can estimate geotechnical, geophysical, hydrogeologic, and analytical parameters in the subsurface. They are particularly useful when the subsurface is highly stratified or contains laterally discontinuous layers. In such situations, characterizing or monitoring a dissolved-phase plume may require identifying preferred groundwater flow pathways, such as zones of high hydraulic conductivity, for sampling. For example, if the presence of DNAPL is suspected, then possible locations where DNAPL has pooled should be targeted by mapping the surface and areal

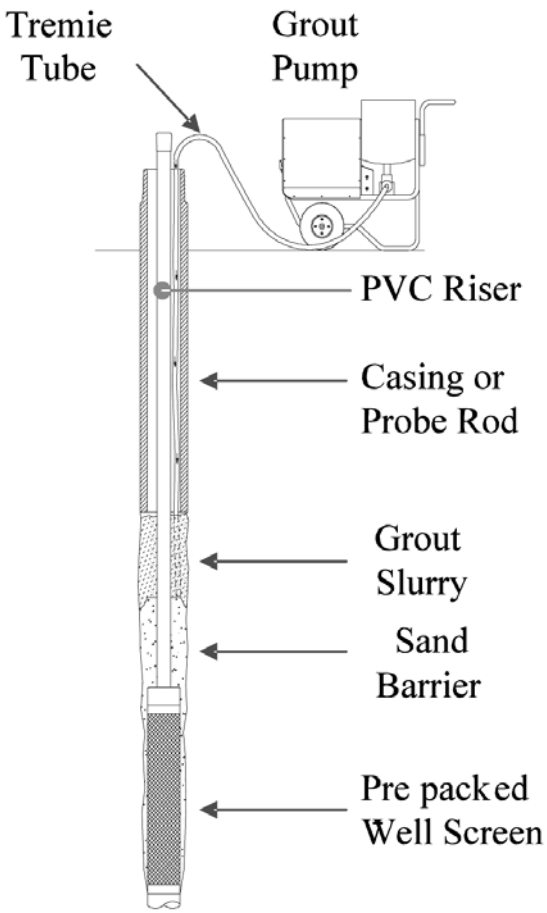
[1]The foam bridge is constructed of a polyethylene cover over polyurethane foam. In choosing to use this device, it should be kept in mind that polyethylene is permeable to many dissolved organic constituents and polyurethane foam will bind organic constituents that come in contact with it. Whether this will affect the quality of the sample is not known. However, purging the well should take care of any potential problems.

Figure 2.8
Small Diameter DPT Well Components



Source: GeoInsight Inc. product literature

Figure 2.9
Bottom-Up Method for Grouting Small Annular Spaces of DPT Wells



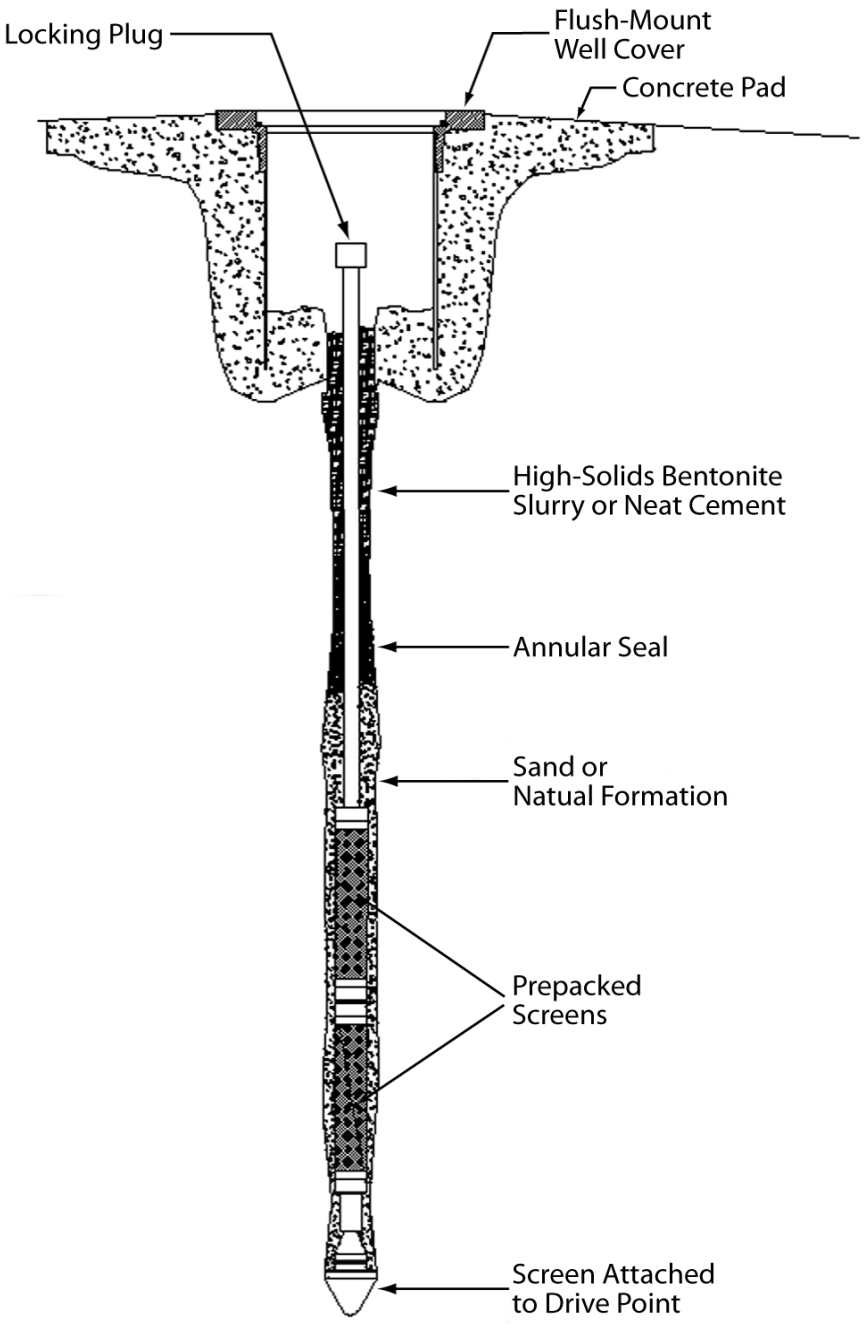
Adapted from Geoprobe®, 1996.

extent of an aquitard. The following section describes some of the specialized measurement and logging tools that are currently available. Since new tools are continually being developed, the list provided in this guide should not be considered complete and is directed at tools specifically concerned with groundwater quality.

Geotechnical

The most common type of DPT geotechnical measurements are conducted with a three-channel cone as part of a CPT rig. It simultaneously measures the tip resistance, sleeve resistance, and inclination of the cone. The ratio of sleeve resistance and tip resistance is used to interpret the soil behavior types encountered (Chiang et al., 1992). In general, sandy soils have high tip resistance and low sleeve resistance, whereas clayey soils have low tip resistance and higher sleeve resistance. The data are recorded in real time on a computer at the ground surface and compiled to generate logs that show soil behavior type and relative density with depth. Actual soil samples are needed to correlate CPT soil behavior data to site soil types.

Figure 2.10
Example of a Properly Constructed DPT Well Installation with Prepacked Well Screen



Adapted from Geoprobe®, 1996.

Geophysical

A number of geophysical measurements can be collected with probes or cones attached to direct push rods. The most common equipment is a conductivity probe that measures the bulk conductivity (or resistivity) of the adjacent soil as it is advanced. The differences in conductivity can be related to changes in stratigraphy. Although actual soil logs are important to correlate probe readings with actual site conditions, in general, finer-grained sediments (e.g., mineral clays) are more conductive than coarser sediments (e.g., sands, gravels). Conductivity probes are also affected by soil water content and ionic strength so they can be used to locate contaminant plumes that have a different salt content than naturally occurring water/soils. In addition, these instruments can sometimes be used for detecting DNAPL masses, which have low conductivities, when there is a sufficiently large conductivity difference between the DNAPL and the surrounding soil matrix. Although these probes will detect LNAPLs as well, there are generally simpler and more reliable ways of locating them than using conductivity probes. Electrical resistivity can also be measured with probes and cones to obtain similar information.

Hydrogeologic

CPT rigs can be equipped with piezocones that measure dynamic pore water pressure as the tool is advanced through the soil layers. The pore water pressure data can be used to determine the depth to the water table and the relative permeability of the layers. Advancement of the penetrometer can be paused at selected intervals to run dissipation tests to obtain estimates of hydraulic conductivity. The combined results of the CPT and piezocone tests can help identify potential preferential contaminant transport pathways in the subsurface. These pathways are especially useful for targeting groundwater sampling locations. Using point-in-time DPT sampling to identify which of these pathways are contaminated can further define optimum intervals for monitoring well screens.

Analytical

There are a number of probes that can be attached to DPT rigs to detect contaminants in the subsurface. These include induced fluorescence systems and volatilization and removal systems.

Induced Fluorescence Systems

Two widely available systems used with CPT rigs are the Site Characterization Analysis Penetrometer System, or SCAPS, and the Rapid Optical Screening Tool, or ROST™. Both use a CPT-deployed laser-induced fluorescence (LIF) probe to qualitatively identify the types and relative concentrations of petroleum hydrocarbons present. This is accomplished by transmitting ultraviolet (UV) light from a nitrogen laser through a sapphire window into the soil. The UV light causes polynuclear aromatic hydrocarbon (PAH) components to fluoresce, and the varying intensity of the fluorescence is indicative of the amounts of the PAHs present. The spectrum of the fluorescence describes the distribution of PAHs present in the hydrocarbon (or often contaminant mass), which can be used for rough fingerprinting of the type of hydrocarbon (Knowles, 1995).

Another induced fluorescence technology, sometimes referred to as a fuel fluorescence detector (FFD), is very similar to LIF except that it generally uses a mercury lamp as its light source, and the light is located in the probe at the sapphire window. This lamp provides a continuous source of light rather than the pulsed technique of the LIF. Although downhole detectors are available, fluorescence intensities from the soil are generally returned to the surface for measurement via fiber optic cable. It generally reads total fluorescence. Some vendors have filtering capabilities to limit wavelength reception to their detectors that allows some differentiation between contaminant types.

Volatilization and Removal Systems

There are two established systems for analysis of VOCs by volatilizing the contaminants in the subsurface and transporting them with a carrier gas to the surface for analysis. The membrane interface probe (MIP), used with percussion or hydraulic driven DPT rigs, heats the surrounding soil to promote diffusion of VOCs through a permeable membrane. Once VOCs enter the probe, they are transported to the surface to a detector (e.g., a photoionization detector or flame ionization detector) with a carrier gas. The probe is generally driven at a rate of one foot per minute to maintain operating temperatures. The presence or absence of VOCs and their relative distribution among sampling locations can be estimated. If more chemical specific information is needed, the MIP can be used in combination with a direct sampling ion trap mass spectrometer (DSITMS). Since this instrument does not have a separation column in front of it, it may not be able to differentiate between chemicals having the same major ion signature.

The SCAPS Hydrosparge™ can be used with either CPT or percussion rigs and is equipped with a module that is lowered into a sealed-screen sampler once the drive rods are retracted to expose the screen. The module uses helium gas at a calibrated flow rate to purge VOCs from the groundwater and transfer them via a Teflon tube directly into a detector at the surface for real time analysis. One sample per location can be analyzed. To collect additional samples at other depths, the sealed-screen sampler can be re-advanced at other locations adjacent to the sampled hole. Data from the hydrosparge is semi-quantitative because of uncertainty associated with sample volume measured.

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Section 3: Data Quality Objectives for Groundwater Sampling

Before selecting an approach to collecting environmental samples, EPA requires that EPA-funded projects use a systematic planning process to plan the collection of project data (EPA, 2000). To help planners select the best methods for obtaining data of the appropriate type, quality, and quantity for their intended use, EPA has developed a seven-step recommended data quality objective (DQO) process. DQOs are designed to provide qualitative and quantitative statements that clarify study objectives, define the appropriate type of data, and specify tolerable levels of potential decision errors that can be used as the basis for establishing the quality and quantity of data needed to support decisions. Because DQOs are intended to be project specific, they should be developed as part of the process for determining the goals for their activity. For some groundwater investigations, a DQO may be needed that describes the type of samples needed to characterize a groundwater plume; or a DQO may be needed to describe the type of samples required to establish that contamination is no longer a threat to drinking water. Although these DQOs may demand very different types of activities, the factors that should be evaluated for DPT groundwater sampling projects typically include:

- Determining the potential direction and degree of sampling bias;
- Evaluating whether the sample volume is sufficient for the selected analytical methods; and
- Minimizing the potential for contamination drag-down or creating a conduit for contaminant transport.

These factors are often issues for both DPT and conventional methods. Techniques exist for resolving problems for both methods, however, this section summarizes how they apply to DPT groundwater collection techniques.

Sample Bias

Sample bias generally is the systematic or persistent distortion of a measurement process that causes errors in one direction. In other words, sample measurements can be consistently different than the samples' true values. There are several potential sources of sample bias when sampling groundwater with any method, including DPT, but one of the most critical factors typically is the type of sampling equipment used to retrieve the sample (Nielsen and Yeates, 1985). Three of the most common sources of bias, due to sampling equipment and methods, include:

- Sample turbidity;
- Sample disturbance; and
- Sampling interval.

Sample turbidity can cause bias as a result of the adsorption of chemicals onto, or the release of chemicals from, the surface of particles in the sample. There also are several sources of bias that can result from sample disturbance. These sources are summarized in Table 3.1. Because sampling interval can have an impact on all analytes in a similar way, (i.e., errors do not necessarily result in one direction), it is not included in the table. The table helps to clarify what factors may have significant impacts on specific analytes and which would have little or no impact by indicating the direction of bias ("P" for positive bias and "N" for negative bias)

Table 3.1
Potential Impacts of Sources of Bias on Specific Analytes During Sampling

Analyte	Potential Source of Bias							
	Sample Disturbance						Turbidity	
	Pressure Decreases	Temperature Increases	Exposure to Atmospheric Conditions	Adsorption onto Sampler Materials (Plastics & Metals)	Desorption from Sampler Materials	Agitation/ Aeration During Sample Collection	Adsorption onto Particles (a)	Releases from Particles (a)
VOCs	N+++	N+++	N+++/P+++ (b)	N+	P+	N++	0	0
Dissolved Gases & ORP	N+++	N+++	N+++/P+++ (b)	N++ to N+++ (c)	P+	N++	0	0
Semi-Volatiles	N+	N++	N++	N+	P++	N+	N++	P++
Pesticides	N+	N+	N+	N+	0	N+	N++	P++
Trace Metals	N+	N+	N+++	N+ to N+++ (d)	P+	N+	N+++	P+++
Radionuclides	N+	N+	N+	N+	0	0	N+++	P++
Major Ions (Inorganic Anions & Cations)	0	0	N+	N+	0	0	N+	P+

Legend:

Bias Type:	Relative Degree of Impact:	
N = Negative	+	Weak
P = Positive	++	Moderate
0 = None	+++	Strong

(a) Adsorption to and release from particles is directly related to the level of turbidity and will also depend heavily on particle size and type. Adsorption is a greater factor when fine-grained, organic-rich particles are present.

(b) Depending on the analyte concentrations in the sample or the ambient atmosphere, concentrations in the sample may increase or decrease significantly.

(c) Reaction of the dissolved oxygen in groundwater with iron in the drive rods will significantly reduce measured dissolved oxygen and oxidation/reduction potential (ORP) due to oxidation of the zero valent iron under ambient conditions. Therefore, the sample must be isolated from steel drive rods to minimize this effect.

(d) Some trace metals can complex with hydrous iron oxides (rust) forming soluble ferrous iron. Therefore, the sample must be isolated from steel drive rods to minimize this effect.

and the degree of bias ("+" for weak, "++" for strong, and "+++" for very strong) when sampling for various analytes. For example, if investigators are interested in collecting groundwater samples for VOC analysis, they should be concerned about any changes in temperature or pressure of those samples because it would likely have a negative bias on results. However, if they were collecting the same samples for analysis of major, naturally occurring, inorganic anions and cations (e.g., Ca^{2+}, Na^{+}, K^{+}, SO_4^{2-}, CO_3^{2-}, NO^{3-}, Cl^{-}), they normally would not have to be concerned about pressure or temperature changes since they normally would not affect results.

The iron in steel drive rods of point-in-time samplers can have a significant affect on measured concentrations of analytes, such as dissolved oxygen, iron, and some trace metals as well as changing oxygen-reducing potentials. As discussed in Section 4, these affects can be minimized by placing the pump intake within the screened interval to be sampled and pumping at a low-flow rate to avoid drawdown of standing water in contact with the rods into the intake interval.

To minimize sampling bias, sampling equipment that meets project DQOs should be selected. For DPT, the primary difficulty in collecting groundwater samples typically is caused by the small inside diameter of many sampling points—generally 0.75-inch or less for the rods used with DPT sampling tools and 1 to 2 inches for DPT-installed wells. For DPT tools of these sizes, the available sampling devices are usually limited to less than 1-inch OD. For rods or well casing/screen with inside diameters less than 2 inches, sampling equipment usually is limited to bailers, inertial-lift pumps, suction-lift (e.g., peristaltic) pumps, gas-drive pumps, centrifugal pumps, and bladder pumps. For 2 inches and larger, additional devices available include gas-operated piston pumps and several designs of electric submersible pumps (e.g., gear-drive, helical rotor, or progressing cavity).

Bladder pumps (Pohlman et al., 1990; Unwin and Maltby, 1988; Parker, 1994; Barcelona et al., 1984), gear-drive electric submersible pumps (Imbrigiotta et al., 1988; Backhus et al., 1993), centrifugal pumps, and helical rotor pumps have consistently outperformed other pumps in their ability to deliver a representative sample for a wide variety of analytes under a wide range of field conditions. These devices are recommended for use in collecting samples for all classes of analytes. Each of the other devices has limitations that may affect the representativeness of samples for one or more classes of analytes (Nielsen and Yeates, 1985; Herzog et al., 1991; Parker, 1994; Pohlman et al., 1994; Pohlman and Hess, 1988; Pearsall and Eckhardt, 1897; Unwin and Maltby, 1988; Imbrigiotta et al., 1988; Pohlman et al., 1990). However, some of these devices may be appropriate for collecting samples for some sets of analytes.

A detailed discussion of the operational characteristics of sampling devices is provided in the Appendix. In addition, Table 3.2 provides a summary of some important operational characteristics related to DPT applications and the appropriateness of each device for sampling analyte classes. For example, the table shows that bailers are adequate for sampling narrow diameter wells when the analytes of concern are inorganic ions. If trace VOCs are being analyzed, bailers may not be as reliable for providing high quality samples as other methods. In addition, choosing a sampling tool is often related to the specific use the data acquired will be put. For example, bailers might be completely acceptable when sampling VOCs to locate a DNAPL source zone when the dissolved values in the water are very high and marginal losses are not important.

Table 3.2
Typical Operational Characteristics and Appropriateness of Groundwater Sampling Devices for Specific Analytes

Device	Approx. Minimum Well Diameter	Approx. Maximum Useful Depth to Groundwater	Approx. Minimum/ Maximum Sample Delivery Rate	Field Indicators				Inorganics			Organics		Radioactive		Biological
				SC	pH	ORP	DO	Major Ions	Trace Metals	Salts	VOCs	SVOCs	Radio-nuclides	Gamma α/β	Coliform
Bailer	½"	unlimited	highly variable	✓				✓		✓			✓		✓
Inertial-Lift Pump	½"	unlimited	highly variable	✓				✓		✓			✓		✓
Suction-Lift Pump (Peristaltic)	½"	25'	50 mL - 4 L/min	✓				✓		✓			✓		✓
Gas-Drive Pump*	½"	250'	50 mL - 20 L/min	✓	✓			✓	✓	✓		✓	✓	✓	✓
Bladder Pump	½"	300'	25 mL - 8 L/min	✓	✓	✓	✓	✓	✓	✓	✓	✓	✓	✓	✓
Piston Pump	2"	1000'	100 mL - 8 L/min	✓	✓			✓	✓	✓		✓	✓	✓	✓
Electric Submersible Pumps:															
Gear-Drive	2"	300'	50 mL - 12 L/min	✓	✓	✓	✓	✓	✓	✓	✓	✓	✓	✓	✓
Helical Rotor	2"	180'	100 mL - 6 L/min	✓	✓	✓	✓	✓	✓	✓	✓	✓	✓	✓	✓
Centrifugal	1.75"	220'	100 mL - 34 L/min	✓	✓	✓	✓	✓	✓	✓	✓	✓	✓	✓	✓

✓ = Device compatible with analyte

* Presumes use of inert drive gas

Abbreviations

SC - specific conductivity
ORP - oxidation/reduction potential
DO - dissolved oxygen
VOCs - volatile organic compounds
SVOCs - semivolatile organic compounds
α - alpha
β - beta

Sample Turbidity

In groundwater sampling, turbidity generally refers to the presence of suspended particles in the sample. These particles may be entrained in the groundwater when the subsurface is disturbed, such as when a DPT sampling tool or well is advanced and installed. The disturbance may cause some compaction and disaggregation of granular material as well as the breakage of grain coatings and cementing agents. The largest particles (i.e., silts) typically will settle out quickly but much can remain suspended in the water column. Depending on their type and size, some particles are neutrally buoyant, remaining suspended once they have become suspended. Turbidity can also be generated during sampling activities due to the relatively high entrance velocity of groundwater into the well when water is withdrawn by sampling tools, such as high-speed submersible pumps, or sealed-screen samplers when the screen is opened to the formation.

Although turbidity can be present in samples from sand and gravel formations, it particularly can be a problem when sampling in fine-grained formations. High turbidity also can be associated with the DPT sampling tools that lack screens or filter packs to keep the fines from entering the sampling tool or well. Conventional wells can also produce samples with high turbidity, especially if the slot size of the screen or the grain size of the filter pack are incorrectly sized for the formation.

The term “artifactual turbidity” is sometimes used to distinguish particles stirred up during drilling and sampling, which would not be mobile under ambient groundwater flow conditions, from those particles that are mobile under ambient conditions—colloids. Colloids are typically clays, hydroxyls, and humic materials that are 1 to 1000 µm in diameter. Although colloidal transport may be considered important in formations made up of materials coarser than fine sand (Dragun, 1988; Mason, 1991), under most ambient conditions, colloids are immobile in the subsurface. Because most turbidity is artifactual in origin, for the purpose of this discussion it is just referred to as “turbidity”.

Sample turbidity can be important because it can be a common source of significant bias, both negative and positive, in groundwater samples, particularly when metals and semi-volatile organic compounds (SVOCs) are the analytes of concern (see Table 3.1). For example, some clay colloids can artificially increase the measured concentration of dissolved metals because metals are found in their structure (e.g., aluminum, magnesium, and iron) and as similarly sized impurities associated with them. The surficial negative charge of the colloids and impurities attracts and loosely typically binds the positively charged metal cations in groundwater. The risk of positive bias can be further increased when acid is used to preserve a sample because it will cause the metals to dissolve back into the sample. On the other hand, if the dissolved metals bind to the colloids, and the sample is then filtered to remove high turbidity levels, the concentration of dissolved metals in the groundwater may be significantly reduced and the sample normally would not be representative of actual conditions.

In a similar way, colloids can become a “source” or a “sink” for organic constituents. Although organic chemicals of concern generally do not occur naturally in clays, they can sorb to colloids and naturally occurring organic matter. The level of sorption that will occur depends on the individual chemical, whether it was in equilibrium with the suspended colloids before being collected, and the analytical preparation method used. If groundwater sampling creates or occurs under non-equilibrium conditions, then significant sorption may occur. In addition, if organic constituents in groundwater have bound to clays or humic acids in the subsurface,

causing them to become immobile, the sampling process can disturb these immobile constituents and cause them to become dissolved in the groundwater sample.

One method of determining whether the level of turbidity will significantly impact the concentration of an organic constituent by causing it to become bound to colloids is to examine the logarithm of the *n*-octanol/water partition coefficient (log K_{ow}) because the log K_{ow} is a measure of a compound's tendency to remain dissolved in water. The higher the K_{ow} value, the more likely the chemical is to partition from the water onto suspended organic particles. Table 3.3 contains the log K_{ow} of a number of common organic chemicals.

This information can also be used as a quality control check on groundwater sample results. In general, the compounds with the higher log K_{ow} levels are SVOCs. If the groundwater concentrations for these chemicals approach or exceed their solubility, then the measured concentrations may have been artificially inflated by turbidity and other sampling data should be evaluated (e.g., nephelometric turbidity units [NTU], a measure of turbidity, dissolved oxygen). Alternatively, turbidity may not be a source of bias in a sample if the constituents of concern are organic compounds with low log K_{ow} values (e.g., < 2.5). In this situation, other issues, such as stability of groundwater indicator parameters during sampling, may be more important in evaluating groundwater sample quality. A paper by Paul and Puls (1997) helps to illustrate the significance of these numbers. In the study, which included analysis of both laboratory and field samples spiked with kaolinite and sodium montmorillonite clays, the researchers demonstrated that TCE, *cis*-DCE, and vinyl chloride concentrations were statistically unaffected by turbidity levels. This group of chemicals has low log K_{ow} values ($\leq$ 2.42). The report concluded that the presence of solids in the groundwater samples had little or no effect on the VOC concentrations evaluated in the study. Losses of VOC due to volatilization during the sampling process were thought to have a greater effect on concentrations. As a word of caution, however, matrix effects can affect the tendency of some compounds to sorb more than their low log K_{ow} values would indicate. This increased level of sorption generally occurs when the soil matrix contains a substantial amount of organic materials, which can range from humic and fulvic acids to organic debris (roots leaves) and peat. Site-specific factors, such as total organic carbon (TOC), should be considered when deciding to take measures to reduce turbidity in groundwater samples. High levels of TOC in groundwater samples can yield analytical results that indicate higher levels of dissolved VOCs than are actually present. These higher levels can drive a risk assessment, even though the VOCs are actually sorbed to particles and are immobile.

When turbidity is a concern for groundwater sampling, steps can be taken to minimize it, provided adequate quality control (QC) procedures, described in Section 4, are followed, such as installation of filter packs (also discussed in Section 2), developing the well, and using low-flow sampling. In addition, DPT methods that promote high turbidity levels, such as open-hole sampling or exposed-screen methods, should be avoided. A representative sample cannot be salvaged with filtering if inappropriate sampling techniques have already compromised the sample. If filtering is justified by a project's DQOs, proper filtering techniques should be used. For more information on filtering, readers should refer to *Ground-Water Sampling Guidelines for Superfund and RCRA Project Managers* (EPA, 2002).

Table 3.3
Log *n*-Octanol/Water Partition Coefficients (Log K_{ow}) of Common Organic Contaminants

Chemical	Log K_{ow} [a]	Chemical	Log K_{ow}
Acenaphthene	4.07	Ethylbenzene	3.13
Acetone	-0.24	Ethylene Glycol	-1.36
Aldrin	5.52	Fluoranthene	5.22
Anthracene	4.45	Fluorene	4.12
Arochlor 1221	2.8 estimated	Heptachlor	5.44
Arochlor 1242	5.58	Hexachlorobutadiene	4.78
Arochlor 1260	6.91	Indeno(1,2,3-cd)pyrene	7.7
Benzene	2.13	Lindane	3.7
Benzo(a)anthracene	5.61	Methoxychlor	4.3
Benzo(k)fluoranthene	6.85	Methyl Ethyl Ketone	0.29
Benzo(g,h,i)perylene	7.1	Methyl t-Butyl Ether	1.24
Benzo(a)pyrene	5.99	Naphthalene	3.59
Bis(2-ethylhexyl)phthalate	4.20, 5.11	Pentachlorophenol	5.01
Carbon Tetrachloride	2.83	Phenanthrene	4.468
Chlordane	6	Phenol	1.48
Chlorobenzene	2.84	Pyrene	5.18
Chloroform	1.97	Styrene	2.95
Chrysene	5.6	1,1,2,2-Tetrachloroethane	2.39
p, p-DDT	6.36	Tetrachloroethene (PCE)	3.4
Dibenz(a,h)anthracene	6.36	Toluene	2.73
Dibenzofuran	4.17	Toxaphene	3.3
1,1-Dichloroethane	1.79	1,1,1-Trichloroethane	2.49
1,2-Dichloroethane	1.48	1,1,2-Trichloroethane	2.07
cis-1,2-Dichloroethene	1.86	Trichloroethene	2.42
Dieldrin	5.16	Tetrahydrofuran1,4-Dioxane	-0.27
1,4-Dioxane	-0.27	Vinyl Chloride	0.6
Endrin	5.16	m-Xylene	3.2

[a] Log K_{ow} values taken from Howard et al., 1993a and 1993b and from Montgomery and Welkom, 1991.

Sample Disturbance

If groundwater samples are disturbed during the collection process (e.g., agitation, allowing sample temperature to increase, or creating a situation for sample materials to adsorb or desorb analytes) there is a risk of negative impacts on groundwater sample quality, beyond the issue of generating turbidity, because it may result in the:

- Volatilization of any VOCs that are present;
- Dissolution of dissolved gases; or
- Oxidation/reduction of metals.

Sampling methods should be carefully selected in order to minimize disturbance during sampling. As shown in Table 3.2, bladder pumps and to a lesser extent centrifugal pumps generally are the best equipment for all analytes when sampling rods or wells less than 2-inches in diameter and where high quality samples are needed. Research has shown that suction lift pumps (e.g., peristaltic pumps—see Appendix) cause a negative bias in VOC and dissolved gas measurements because of the negative pressure generated by the pumping action. When point-in-time sampling is conducted in situations where volatilization of VOCs is a concern, sealed-screen samplers, such as the BAT Enviroprobe™ or Hydropunch™, which maintain in-situ pressure conditions, should be considered.

Sampling Interval

The most appropriate sampling interval to use at a site should be determined because the location and length of a sampling interval can bias a sample. Short or long sampling screens can be used with both DPT and conventional monitoring wells; however, DPT point-in-time methods are generally more economical and better designed to target smaller sampling intervals than conventional wells. When selecting discrete sampling intervals (e.g., 6-inch interval), there is a risk of missing contaminants that may be migrating through sections of the aquifer that do not fall within the screened interval(s). When selecting long sampling intervals (e.g., 5 to 15 feet), there is a risk that a highly contaminated but discrete interval will be diluted by larger uncontaminated intervals.

To determine the most appropriate sampling interval, a number of sources of existing information should be evaluated and, if necessary, additional subsurface data collected. DPT equipment can provide many cost-effective and comprehensive methods for collecting subsurface data for this purpose, including continuous soil logs, detailed stratigraphic logs using specialized measurement and logging tools (e.g., cone penetrometer testing, membrane interface probe, induced fluorescence), multi-level discrete groundwater samples to create a vertical profile of contamination, and piezometric data over a wide area to determine groundwater flow direction (see U.S. EPA, 1997 for more details). Based on this information, as well as slug tests and/or aquifer tests to estimate hydraulic conductivity for specific stratigraphic zones, contaminant source locations can be estimated. At this point the appropriate sampling intervals can be selected, thereby minimizing the installation of extraneous and ineffective wells or the risk of missing or diluting important transport pathways.

Sample Volume

There are three sample volume issues that can be particularly relevant to collecting groundwater samples with DPT:

- Point-in-time methods can be extremely slow in fine-grained formations (e.g., several hours or more).
- Sample chamber volume of some sealed-screen, point-in-time samplers is quite small (e.g., less than one liter), which can make it difficult to collect the larger volumes needed for some types of analyses (e.g., SVOCs, PCBs/pesticides).
- Both DPT wells and point-in-time samplers often have a smaller diameter than their conventional well counterparts. The smaller volume of the DPT wells may require lower-flow purge rates to avoid significant drawdown during sampling.

Consequently, subsurface conditions and the required analytical suite should be considered when selecting a DPT method to sample groundwater. Depending on the site conditions and site DQOs, larger volume samplers or low-flow purge equipment capable of very low-flow rates (discussed in Section 4) may be needed, or a sampler can be left in place to recharge while other locations are sampled. If a sampler is left in place to recharge, sample quality can be compromised if the sample container is filled in increments collected over a period of time. This is a concern primarily when filling sample vials for volatile organic analysis, which need to be filled completely with one sampling effort. Otherwise, volatile compounds may partition into the headspace above the sample.

Sample Cross-Contamination

Any groundwater sampling method can cause cross-contamination that affects groundwater sample quality and/or long-term water quality in at least three ways:

- Causing contaminant drag-down;
- Creating hydraulic conduits; and
- Biasing samples from improperly decontaminated equipment.

In evaluating the potential for cross-contamination and developing a sampling plan, the site geology, the types of contaminants present, and the sampling methods and equipment used should be examined. For example, drilling a hole through an aquitard creates a potential conduit for contaminant migration. If DNAPLs are perched on top of the aquitard and precautions are not taken, they may migrate down the borehole and contaminate a previously uncontaminated aquifer.

Although cross-contamination can be a serious concern that may pose additional challenges when using DPT equipment, DPT methods also provide many strategies for minimizing or eliminating the risk of cross-contamination. Section 5 reviews these methods and additional resources that can provide specific details needed in the appropriate use of DPT equipment.

Selecting a DPT Groundwater Sampling Tool

DPT tools can be considered for a wide range of groundwater field applications, and they can meet project DQOs in a broad variety of cases. Sampling bias, sample volume, and cross-contamination are potential problems whether DPT or conventional monitoring wells are used. It is important to understand the limitations of equipment being used and how they relate to project needs. The first step in selecting equipment should be narrowing down the categories of appropriate tools. With DPT equipment, that can be done by deciding whether qualitative, semi-quantitative, or quantitative data are needed. Table 3.4 provides a summary of the applications of DPT tools, emphasizing the basic concept that the project objectives should be considered when selecting equipment and methods.

Table 3.4
Recommended DPT Groundwater Tools for Various Field Applications

General Field Application	Specialized Measurement and Logging Tools (a)	Point-in-Time Groundwater Sampler	DPT-Installed Monitoring Well
Presence of contamination (i.e., qualitative sampling goals)	✓	✓	
Approximation of contaminant zone/level (i.e., semi-quantitative sampling goals)	✓	✓	
Define specific contaminants and accurate concentrations (i.e., quantitative sampling goals)		✓	✓
Long-term monitoring			✓

(a) Includes induced fluorescence and volatilization and removal systems.

Section 4: Recommended Methods for Collecting Representative Groundwater Samples

Collecting groundwater samples that are representative of in-situ aquifer conditions generally is important in any groundwater investigation. One of the most important factors in meeting this goal, for many analytes, typically is to minimize turbidity. This is because particles from formation materials that are suspended in a sample, but are not normally suspended in groundwater, can provide a substrate for various analytes to adsorb or desorb. This process can create a positive or negative bias for analytical results. In addition, although the causes of turbidity and their solutions for both DPT and conventional groundwater sampling methods generally are similar, the relatively narrow rod diameters of DPT systems can create additional hurdles. This section focuses on the activities that can minimize turbidity, specifically for DPT systems.

As discussed in Section 3, turbidity can cause substantial bias, both negative and positive, when sampling metals and SVOCs, but typically is much less an issue when sampling VOCs. There are several precautions that can be taken to minimize turbidity in DPT groundwater samples. Important techniques to consider include:

- Installation of a filter pack;
- Well development; and
- Low-flow purging or passive sampling protocols.

Each of these techniques can be applied to monitoring wells installed with DPT, but not all are possible with point-in-time samplers. For example, filter packs normally cannot be installed when using point-in-time samplers. Furthermore, well development and low-flow/passive sampling protocols normally can only be used with those point-in-time samplers that provide access to the sample location from the surface (See Section 2). Usually, samplers that rely on a sealed sample chamber to retrieve groundwater cannot be developed, nor can low-flow or passive sampling protocols be followed. However, there are some samplers in this category that may work, such as one of the BAT™ system samplers, that sample water through a ceramic or polymer tip that acts as an in-situ filter to prevent turbidity. The decision to use a specific type of point-in-time sampler should be weighed against project DQOs.

Installation of a Filter Pack

As mentioned in Section 2, installing a filter pack in monitoring wells can be an important means of minimizing sample turbidity. However, its installation is not always possible or necessary, depending on project objectives. A common construction technique for DPT wells is to let the formation collapse around the screened interval, rather than installing a filter pack. In fact, this is the only option for exposed-screen well installations since there is no annular space between the drive rods and the borehole walls to accommodate a filter pack. Similarly, DPT point-in-time sampling techniques do not allow for the installation of filter packs due to the lack of annular space between the drive rods/sample tool and the formation. Wells installed using protected-screen methods, however, may have adequate annular space for a filter pack and should be selected where data quality objectives dictate. Please refer back to Section 2 for more

information on filter pack installation techniques, including the use of pre-packed and sleeved well screens.

Well Development

Wells should be developed after completing the installation and allowing sufficient time for the annular seal to completely set (typically two weeks, but not less than 48 hours). The purpose of well development or development of a point-in-time sampler generally is to repair borehole damage caused by advancement and installation procedures, such as the smearing of fine-grained particles along the borehole walls and the generation of turbidity. Development generally is designed to remove these particles to improve the hydraulic connection between the well and formation so that groundwater can enter more freely. Development also is designed to remove the groundwater impacted by well installation so that groundwater representative of ambient conditions can be sampled.

Like conventionally installed wells, DPT-installed monitoring wells should be developed. This process typically involves block surging and pumping or bailing groundwater until certain water quality parameters (e.g., pH, specific conductance, dissolved oxygen, redox potential, and temperature) have stabilized and turbidity has been removed or decreased as much as possible. Wells are surged by raising and lowering a surge block (any tool that is slightly smaller than the inside diameter of the well casing) within the screened interval to mechanically backwash the well screen. These activities help to dislodge particles smeared within the borehole and the particles clogging the screen so they can be removed. In-line turbidimeters can be used during development or purging procedures to judge turbidity levels and their potential impact on sample quality.

Due to the way some point-in-time samplers are constructed and used, they may not accommodate a pump or bailer for development. Those samplers that can accommodate a pump or bailer can be developed similar to the method described above for wells, although due to the generally smaller diameters of point-in-time samplers, smaller diameter surge blocks, pumps, and bailers usually will be needed. In general, if DQOs recommend development for groundwater samples to meet quality standards, then many point-in-time samplers may not be appropriate for the situation.

Low-Flow Purging and Sampling

Low-flow purging, also referred to as low-stress purging, low-impact purging, and minimal drawdown purging, is a method of preparing a well for sampling which, unlike traditional purging methods, does not require the removal of large volumes of water from the well. The term “low-flow” refers to the velocity at which groundwater moves through the pore spaces of the formation adjacent to the screen during pumping. It does not necessarily reflect the flow rate of the water discharged by the pump at the ground surface. The focus of low-flow purging and sampling is on collecting high-quality samples by minimizing the impact of pumping on well hydraulics and aquifer chemistry. Because the flow rate used for purging is, in many cases, the same as or only slightly higher than the flow rate used for sampling, the process

is a continuum and is referred to as "low-flow purging and sampling." Although minimizing the disturbance of sampling on the aquifer is important for all types of groundwater sampling devices, it can be particularly important for DPT point-in-time samplers and exposed screen wells because installation of a filter pack generally is not feasible with these tools.

Low-flow purging and sampling generally are appropriate for collecting groundwater samples in a wide variety of situations. It can be used to sample all categories of aqueous phase contaminants and naturally occurring analytes, including VOCs, SVOCs, trace metals and other inorganics, pesticides, PCBs, radionuclides, and microbiological constituents and often is particularly appropriate for situations where colloidal transport is an issue (i.e., radionuclides, metals, and hydrophobic compounds). However, low-flow methods generally are not applicable to the collection of NAPLs because they do not respond to the effects of pumping in the same manner as groundwater.

Theoretical and Research Basis for Low-Flow Purging and Sampling

Groundwater sampling research has demonstrated that water standing in a well casing for a protracted time is not representative of ambient groundwater quality (Gillham et al., 1985; Miller, 1982; Marsh and Lloyd, 1980; Barcelona and Helfrich, 1996). Hence, this water should not be collected as part of the sample for analysis. In addition, the water within the screened interval of nearly all wells generally is representative, provided that the well has been designed, installed, developed, and maintained properly and the aquifer has a sufficient flow rate to ensure the water in the screened interval is being replaced.

Based on these findings, recommended low-flow sampling protocols have been developed so that groundwater can be collected from the screened interval without significant mixing with the water standing in the casing. Research has shown that this method does indeed provide high quality, representative groundwater samples (Backhus et al., 1993; Bangsund et al., 1994; Barcelona et al., 1994; Karklins, 1996; Kearl et al., 1994; Kearl et al., 1992; McCarthy and Shevenell, 1998; Puls and Paul, 1995; Puls and Barcelona, 1996; Puls and Barcelona, 1989; Puls and Powell, 1992; Puls et al., 1992; Puls et al., 1991; Serlin and Kaplan, 1996; Shanklin et al., 1995).

Because they are sampled almost immediately, point-in-time DPT tools should not develop a stagnant water column that will affect most analytical results. However, a standing water column for even a short period of time can affect some inorganic analytes (e.g., iron, nitrogen, and hexavalent chromium) and sensitive geochemical parameters (e.g., dissolved oxygen, oxygen reducing potential, and pH) if the water is in contact with steel drive rods. The zero valent iron in the rods can quickly react with any dissolved oxygen in the groundwater causing alteration of the analytes and parameters and potentially complexing with metal analytes. As a result, water being collected should be isolated from contact with the drive rods when sampling for these analytes or parameters. Placing the pump intake within the screened interval and pumping at a low-flow rate to avoid drawdown of water in contact with the drive rods should help isolate the sample from the rods. Although there is little concern about the water column affecting organic analytes when sampling with point-in-time DPT samplers, when

turbidity is a concern, low-flow sampling may help to lower turbidity to acceptable levels (McCall et al., 1997; EPA, 1996a).

Low-Flow Purging and Sampling Protocols

To conduct low-flow purging and sampling, a pump that can be operated at a low-flow rate normally is needed. For large wells (e.g., 2-inch diameter or greater), less than 500 mL/min is often needed; for small diameter DPT wells and point-in-time samplers, flow rates as low as 100 mL/min may be needed. As a result of their design, bailers generally are inappropriate for low-flow purging and sampling. Inertial-lift pumps, or other well sampling devices that agitate the water column also generally cannot be used for low-flow purging and sampling

Before purging and sampling can begin, the hydraulic conductivity of the screened interval normally needs to be evaluated to ensure that low-stress pumping is maintained. To begin, the water level should be measured to determine when drawdown in the well stabilizes. As water is purged from the well, water quality indicator parameters (e.g., pH, temperature, specific conductance, dissolved oxygen, redox potential, and in some cases turbidity) generally need to be measured to determine when the readings stabilize and samples can be collected. The results can then be used to define well-specific, low-flow procedures.

Passive Sampling Protocols

In situations where a sampling point cannot yield sufficient water to support low-flow sampling, a passive sampling (also referred to as micropurging) approach generally is preferred (Powell and Puls, 1993; Puls and Barcelona, 1996). Passive sampling involves placing the pump intake within the screened interval and purging a minimal volume of groundwater from the well or sampler—a single volume of the pump chamber and discharge tubing, rather than the greater volumes purged by low-flow sampling or the multiple well volumes purged using other techniques. The goal of passive sampling generally is to obtain groundwater within the well screen or sampler screen, which is most representative of ambient groundwater quality, without disturbing the water column and introducing the stagnant water above. The sample should be collected immediately after purging the small volume of groundwater.

Passive sampling methods may be appropriate for use in a variety of situations and can be applied to most wells in which there is sufficient water to ensure that a pump intake is submerged throughout purging and sample collection. They are most often applied to wells and samplers installed in low-yield formations. In very low yield formations, however, the water in the screened interval may be of equivalent quality to that in the casing above and not representative of formation water. The application of passive sampling methods, therefore, should be evaluated on a site-specific basis.

Passive sampling typically is easiest to apply when dedicated pumps are used. The flow rates used for passive sampling are lower than those used for low-flow purging and sampling—generally less than 100 mL/min. Because very low hydraulic conductivity formations do not yield sufficient water to satisfy the demands of a pump, even at these low-flow rates, drawdown may occur. Thus, to determine the volume of water available for sampling, the volume of water within the well screen above the pump intake should be calculated. Only this

volume, which normally will be very small for most DPT wells, should be collected. Sampling should not continue once water from the top of the screen is drawn close to the pump because casing water should not be collected as part of the sample. Since indicator parameters are not typically analyzed, this method does not normally provide any evidence that the sample taken is representative of formation water. When non-dedicated equipment is used, there is a higher risk of mixing casing and screened interval water which can add to the uncertainty of the representativeness of the sample. Table 4.1 provides a comparison between low-flow and passive sampling methods.

Table 4.1
Comparison of Some Key Elements of Low-Flow and Passive Sampling

	Low-Flow Sampling	Passive Sampling
Hydraulic Conductivity	Sufficient to maintain steady water level during sampling	Too low to allow low-flow sampling
Analyte Applicability	All analytes except NAPLs	All analytes except NAPLs
Pump Discharge Rate	500 mL/min to100 mL/min depending on well/sampling point size and hydraulic conductivity.	<100 mL/min
Purge Volume	Continuous until parameters (e.g., specific conductance, turbidity, O_2, redox) stabilize	Greater than the volume of the pump and the submerged tubing

Section 5: Recommended Methods for Minimizing Potential for Cross-Contamination

As mentioned in Section 3, the potential for cross-contamination should be considered when advancing any type of groundwater sampling tool or monitoring well into the subsurface because it can result in sample bias, incorrect decisions, or the spread of contaminants. Methods for avoiding cross-contamination should be discussed and accounted for in project planning with respect to:

- Avoiding drag-down;
- Avoiding the creation of hydraulic conduits;
- Decontaminating equipment; and,
- Decommissioning DPT wells and borings.

These issues typically apply to both DPT and conventional wells; however, because of the different methods of construction, the solutions often differ even though the results are often the same. This section provides guidance on how these issues can relate to DPT methods because the methods for conventional wells are well established.

Avoiding Drag-Down

Drag-down of contamination is commonly considered to be less of a problem with DPT methods than with conventional well drilling techniques, such as hollow stem augering, where contaminants have a better chance of sticking to the augers as they advance. As DPT rods are advanced, the action of pushing the drive rods through new soil generally wipes away old soil. In fact, researchers have demonstrated the lack of drag-down with DPT in a number of studies (Cherry, et al. 1992; Pitkin et al., 1994; McCall et al., 1997; Pitkin et al., 1999); however, as with conventional drilling techniques, it is unlikely that DNAPL or certain soils, such as sticky clays, would be completely wiped clean as the rods advance. In addition, certain DPT methods can result in drag-down if used in inappropriate situations. This is primarily a problem with advancing multi-level samplers in contaminated fine-grained soil, which can clog the screens, and with advancing exposed screen monitoring well installations, which can carry shallow contaminated soil and groundwater to the target sampling depth. Thus, exposed screen well installations should not be considered for use in contaminated areas.

Where drag-down is a concern, use of DPT equipment that will minimize drag-down potential, such as protected-screen point-in-time samplers, the Waterloo Profiler, or sealed-screen monitoring wells should be considered. In addition, the DPT sampler or well should be properly developed to remove the affected soil and groundwater.

Avoiding the Creation of Hydraulic Conduits

Creation of hydraulic conduits that allow the downward flow of groundwater and contaminants can be avoided by sealing off the borehole annulus—the space between the borehole wall and the rod string. This issue is of particular concern when the borehole hydraulically connects previously unconnected hydrogeologic units, or when DNAPLs with low viscosity are present that can migrate downward along the vertical conduit. In addition to using grout to seal the annulus, an important method of reducing downward migration along the rod string is to minimize the borehole annulus. Two important considerations include:

- Using a drive tip that is the same diameter or smaller than the drive rods; and
- Using rods and samplers with the same diameter.

The absence of an annulus, however, does not necessarily prohibit contaminant migration because there may not be an effective seal between the steel rods and the borehole wall. This issue is not unique to DPT methods and should also be considered when drilling boreholes using conventional techniques.

DPT techniques are not recommended for installing monitoring wells with screens set in an interval below a confining layer if there is a real danger of contaminating the lower layer. Instead, construction of a "telescoped" monitoring well using conventional drilling methods should be used. This type of well involves drilling into the top of the aquitard, placing a steel casing in the hole and grouting it into place by tremieing grout into the annulus. The hole is then advanced using a drill bit that fits inside the steel casing. Upon reaching the target depth, the well screen and casing are lowered into place and the well completion materials are installed as the casing is slowly retracted. Multiple casings can be telescoped inside the other if more than one aquitard is present. DPT cannot be used for telescoped well construction because the annulus is too narrow to allow for an adequate grout seal to be installed along the drive casing.

Decontaminating Equipment

As with all groundwater sampling equipment, DPT equipment should be decontaminated before sampling at a new location to avoid cross-contamination. In addition, disposable material should be discarded properly. Some sampling tools, by virtue of their design, may be difficult to disassemble for cleaning. In these cases, when replacing associated tubing, hose, or pipe is not feasible, it may be more practical to clean these tools by circulating cleaning solutions and rinses through the device in accordance with appropriate guidance (e.g., ASTM D5088, ASTM, 2001g; RCRA Ground-Water Monitoring Draft Technical Guidance, EPA, 1992). Where field decontamination is not practical or possible, it may be simpler to use dedicated sampling devices or take a number of portable sampling devices into the field and decontaminate them later at a more appropriate location. Following any cleaning procedure, equipment rinseate blanks should be collected to assess the effectiveness of the cleaning procedure.

Decommissioning DPT Wells and Borings

As with conventional soil borings or abandoned monitoring wells, DPT boreholes for point-in-time sampling and abandoned DPT monitoring wells should be decommissioned to avoid creating a conduit for contaminant migration, either from the surface or between subsurface geologic units. Several methods are available for decommissioning DPT holes, but the method chosen should be capable of backfilling the hole completely with grout or a bentonite slurry, from bottom to top and without gaps. The most appropriate method will depend on a number of factors, including the type and size of DPT equipment being used, site-specific subsurface conditions, and state and/or local regulations. The type of slurry selected may also depend on the remedial action anticipated at the site. For example, a silica flour grout mixture may be selected for sites that may be treated with in-situ thermal technology.

The methods available for decommissioning DPT boreholes include:

- Retraction grouting;
- Re entry grouting; and
- Surface pouring.

These methods are detailed in ASTM D6001 (ASTM, 2001e), EPA (1997), and Lutenegger and DeGroot (1995). Figure 5.1 illustrates the use of these methods, and the following paragraphs summarize the techniques and their applications.

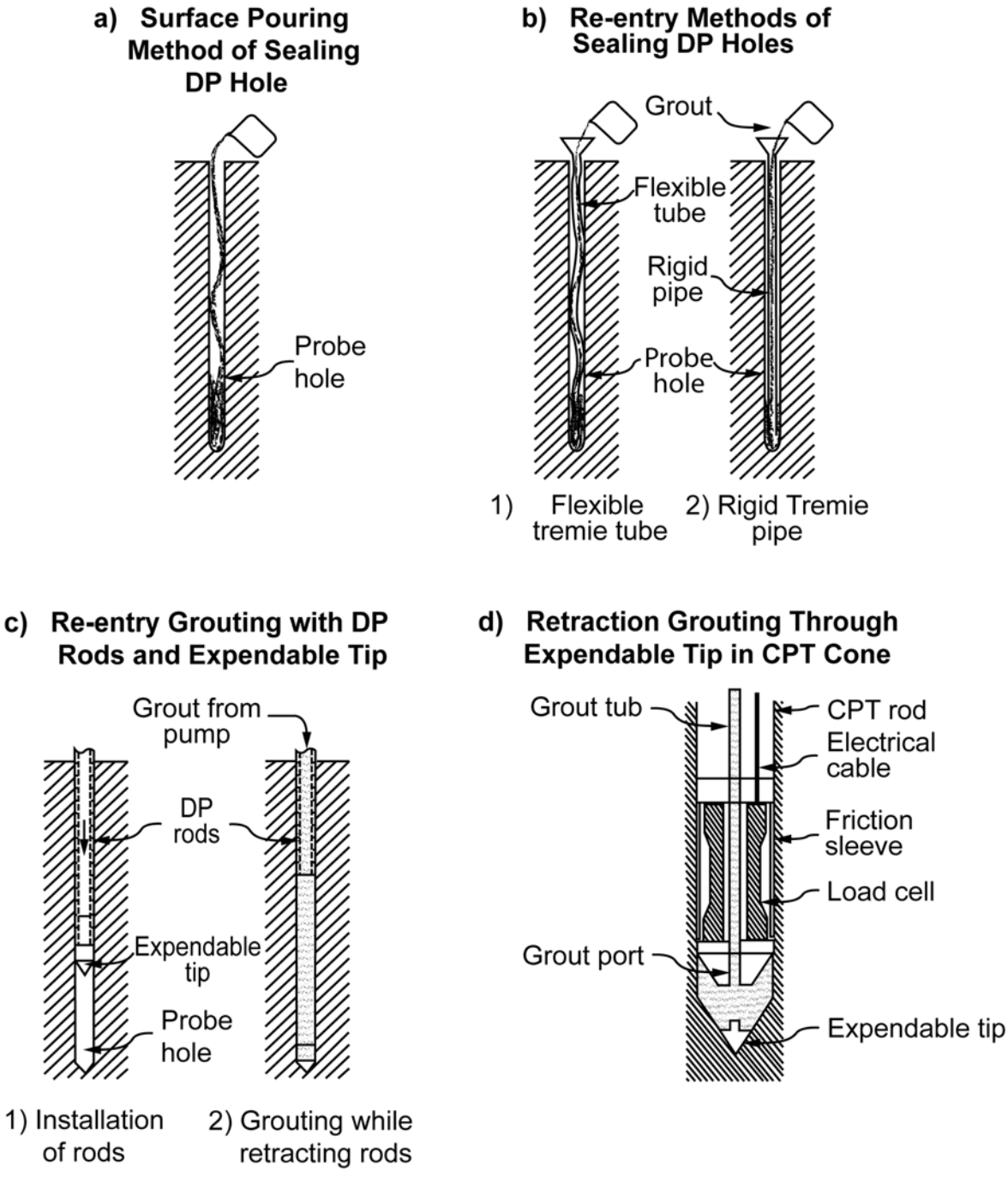
Retraction Grouting

Retraction grouting typically involves pumping a high-solids bentonite and water mixture or a neat cement grout through the rod and tool string and out the bottom of the sampling tool as the tool is withdrawn from the hole. To use this method, a port is needed at the end or sides of the tool or an expendable tip is necessary on the terminal end of the tool through which the grout can be pumped. Because the hole is grouted as the tool is withdrawn, this method ensures that the borehole is sealed throughout its length. Retraction grouting is generally considered to be the most reliable borehole sealing technique.

Re entry Grouting

Re entry grouting typically involves pumping grout through a tremie pipe (a rigid pipe, usually Schedule 40 or 80 Type I PVC) inserted into the borehole immediately following withdrawal of the rod string. Alternatively, the rod string may be reinstalled in the borehole, without the sampling tool, so that grout may be pumped through the open rods. The grout should be pumped continuously from the bottom of the hole to the top as the tremie pipe (or rod string) is withdrawn to avoid gaps and bridging (i.e., plugging the hole before grout reaches total depth) of the grout. Typically, re entry grouting is effective only if the hole remains open until tremie pipe or rods can be extended to the bottom of the borehole. If a portion of the borehole collapses, the tremie pipe or rods will not penetrate to the total depth of the hole. In this situation, it may be necessary to put an expendable tip on the end of the rod string, push the string to the total depth of the hole,

Figure 5.1
Sample Methods for Sealing Direct Push Technology Holes



Source: Adapted from Lutenegger & DeGroot, 1995

knock out the tip, and pump grout through the rods as they are withdrawn. Re entry grouting by this method may not provide a reliable seal if the DPT rods do not follow the original borehole, but the original borehole should provide the path of least resistance under most conditions.

By using a high pressure grout pump and nylon tremie tube it is possible to perform bottom-up grouting in the small annular spaces of DPT equipment. Slurries of 20-30% bentonite or neat cement grout are most commonly used to meet state regulatory requirements.

Surface Pouring

Surface pouring normally is the simplest method of borehole decommissioning; however, it may not be as effective as the other methods in most situations. It involves pouring either dry bentonite (granules, chips, or pellets), bentonite slurry, or neat cement grout from the surface down the open borehole after the rod string and tool are removed. Surface pouring may be effective if the borehole does not collapse after the rods are removed, and if the borehole is relatively shallow (less than about 10 or 15 feet). Where dry bentonite materials are proposed for use, it normally will only be effective if the bentonite is either hydrated from the surface immediately after installation or if it is installed beneath the water table. Maintaining a seal in the borehole requires that the soil moisture content be sufficient to keep the bentonite hydrated after installation. In deep holes, dry bentonite products can bridge, resulting in an incomplete seal. In situations where boreholes partially collapse, materials poured from the surface will not seal the borehole properly. This method can be improved by using a flexible tremie tube or a rigid tremie pipe to reenter the hole and fill it from the bottom up, however, if the hole collapses before the tremie can be installed, the tremie may not be effective either.

Section 6: Conclusions

Direct push technologies can be valuable tools for environmental investigations and facilitate the use of a dynamic work plan strategy. They have a number of potential advantages over conventional groundwater sampling methods and they can have the added benefit of being able to provide numerous other types of detailed subsurface data, such as geophysical, analytical, and hydrogeologic data. The diversity and capabilities of DPT equipment and methods are large enough that under many situations DPT can be used to provide the level of groundwater data quality needed for projects where the subsurface conditions and depth of investigation are amenable to pushing techniques. When techniques common to conventional well installation are followed, such as adequate well development, low-flow purging and sampling, proper decontamination of equipment, and preventing the creation of a hydraulic conduit, quality groundwater samples can be obtained.

DPT will not be appropriate for all situations. DPT methods typically are more limited in their depth of penetration and the types of materials they can penetrate than conventional drilling methods. Some methods may not be able to provide sufficient sample volume or sufficiently low turbidity. Use of DPT may be limited in lower yield formations. Conventional wells with larger diameters may be required to minimize the affect of lower yield formations. Lastly, DPT methods cannot always be used where confining layers are present and there is a danger of creating a vertical hydraulic conduit that could contaminate underlying layers. In these instances, telescoped wells may be needed to prevent downward migration of contaminants beneath a confining layer. As a result, DPT cannot completely replace the use of conventional monitoring wells. Rather, DPT provides additional choices to select equipment and methods for collecting environmental data.

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Appendix: Purging and Sampling Devices

A wide variety of purging and sampling equipment is available for use in DPT sampling points. Available devices can be classified into five general categories:

- Grab mechanisms (including bailers, thief samplers, and syringes);
- Suction-lift mechanisms (including surface centrifugal and peristaltic pumps);
- Centrifugal submersible pumps;
- Positive displacement mechanisms (including gas displacement pumps, bladder pumps, piston pumps, progressing cavity pumps and gear pumps); and
- Inertial-lift pumps.

Though frequently used in the groundwater industry for well development, the gas-lift method is generally considered unsuitable for purging and sampling because the extensive mixing of drive gas and water is likely to strip dissolved gasses from the groundwater and alter the concentration of other dissolved constituents (Gillham et al., 1983). This method is not discussed for this reason.

Grab Sampling Devices

Bailers, thief samplers (e.g., messenger samplers), and syringes are all examples of grab sampling devices. These devices are lowered into the sampling point on a cable, rope, string, chain, or tubing to the desired sampling depth and then retrieved for purge water discharge, sample transfer, or direct transport of the device to the laboratory for sample transfer and analysis.

The most commonly used grab samplers are bailers, which are available in both single check valve and dual check valve designs. The single check valve bailer is lowered to the sampling point, and water entering the bailer opens the check valve and fills the bailer. During retrieval, gravity and the weight of the water inside the bailer closes the check valve. There is some potential for the contents of the bailer to mix with the surrounding water column during retrieval. If mixing is not desirable, then a dual check valve bailer is advisable. Dual check valve bailers are intended to prevent mixing of the sample with the water column upon retrieval. Water passes through the dual check valve bailer as it is lowered. Upon retrieval, both check valves seat, retaining the aliquot of water inside the bailer. Groundwater investigators can minimize mixing by raising the bailer in a steady upward motion with no pausing or slight downward motion, which can occur if the retrieval is done manually.

The thief sampler, employs a mechanical, electrical, or pneumatic trigger to actuate plugs or valves at either end of an open tube to open and/or close the chamber after lowering it to the desired sampling depth, thus sampling from a discrete interval within the well.

The syringe sampler is divided into two chambers by a moveable piston or float. The upper chamber is attached to a flexible air line that extends to the ground surface. The lower chamber is the sample chamber. The device is lowered into the sampling point and activated by

applying a suction to the upper chamber, thereby drawing the piston or float upward and allowing water to enter the lower chamber. In situations where the pressure exerted on the lower chamber by submergence is great enough to cause the piston or float to move upward prior to achieving the desired sampling depth, the upper chamber can be pressurized to prevent piston movement. The device is then activated by slowly releasing the pressure from the upper chamber, allowing water to fill the lower chamber.

Samples collected with grab samplers, especially various types of bailers, exhibit variable accuracy and precision in sample chemistry, often due to operator technique (Puls et al., 1992; Barcelona et al., 1984; Gillham et al., 1983; Pohlmann et al., 1991; Unwin and Maltby, 1988; Tai et al., 1991). Grab samplers can aerate and/or agitate a sample, causing sample oxidation, degassing, and stripping of VOCs from the sample. Care should be taken to avoid sample agitation during transfer of the sample from a grab sampler to the sample container. Pouring water from the top of a bailer either directly into the sample container or to a transfer vessel may agitate/aerate the sample and cause alteration of sample chemistry. These devices can also increase the turbidity of a sample through the surging action created in the well as the device moves through the water column. Grab samplers generally do not subject the sample to pressure changes, though some change may be imparted to a sample when using a syringe sampler activated with a suction. A potential for sample contamination exists due to exposure of the grab sampling device to the surface environment during repeated removal and reinsertion of the device during use. Also, the suspension cord or cable used with grab samplers could contribute contaminants to groundwater samples (Canova and Muthig, 1991).

Suction-Lift Pumps

Surface centrifugal pumps and peristaltic pumps are two common suction-lift pumps. These pumps are usually placed at or above ground level during purging and sampling. They draw water to the surface by applying suction to an intake line through the use of impellers or rotors typically driven by an electric motor. Surface centrifugal pumps use impellers that are typically constructed of brass or mild steel, plastic, or synthetic rubber. A peristaltic pump consists of a rotor with rollers that squeeze flexible tubing as they revolve within a stator housing. This action generates a reduced pressure at one end of the tubing and an increased pressure at the other end. Several types of elastomeric material can be used for the tubing, although silicone rubber is the most common.

Suction-lift pumps may be unacceptable for some groundwater sampling applications. Exertion of a reduced pressure on the sample can cause volatilization or may result in degassing, which can cause changes in the pH, redox potential, and other gas-sensitive parameters (Barcelona et al., 1983; Ho, 1983; Barker and Dickhout, 1988). Peristaltic pumps may be satisfactory for some analytes that are not affected by changes in the sample that can be caused by application of reduced pressure when used under low-flow rate and low lift conditions (Barcelona et al., 1983; Puls and Powell, 1992; Backhus et al., 1993).

Because surface centrifugal pumps can cause cavitation, they are not appropriate for collection of samples to be analyzed for dissolved gases, VOCs, or gas-sensitive parameters such as trace metals. Because the pumped water contacts the pump mechanism, artifacts from sample

contact with these materials should be considered when evaluating these pumps for sampling. In addition, these pumps can mix air from small leaks in the suction circuit into a sample, which can cause sample bias. These pumps are typically difficult to adequately decontaminate between uses. To avoid the limitations posed by the effects of pumping or undesirable pump materials, an intermediate vessel could be used on the suction side of the pump circuit.

Peristaltic pumps do not usually cause cavitation but, as in all suction-lift pumps, the exertion of a reduced pressure on the sample can bias the sample. The flexible tubing required for use in a peristaltic pump mechanism may also cause sample bias.[2]

Centrifugal Submersible Pumps

A centrifugal submersible pump (CSP) consists of impellers housed within diffuser chambers that are attached to a sealed electric motor, which drives the impellers through a shaft and seal arrangement. Water enters the CSP by pressure of submergence, is pressurized by centrifugal force generated by the impellers, and discharged to the surface through tubing, hose, or pipe. A CSP is suspended in a well by its discharge line and/or a support line. Electric power is supplied to the motor through a braided or flat multiple-conductor insulated cable.

Flow rate and depth capability for all designs are wide ranging. For variable-speed CSPs, the discharge rate can be reduced by regulating the frequency of the electrical power supply and controlling the motor speed to reduce flow rate.

While there is no available peer-reviewed literature addressing the sampling effects of small-diameter variable-speed CSPs on dissolved gases or VOCs, one study found these pumps produced samples for some dissolved metals that were comparable to samples from bladder pumps (Pohlmann et al., 1994). With all CSPs, heat generated by the motor could increase sample temperature, which could result in loss of dissolved gases and VOCs from the sample.

CSPs are only available in diameters that will fit into sampling points 1.75 inches or larger in diameter. CSPs can be damaged when used in silty or sandy water, requiring repair or replacement of pump components and/or motor. If overheating occurs, there are three possible consequences. First, where the motor has internal water or oil in it for improved cooling characteristics, some of this liquid could be released into the sampling point, which could potentially contaminate the sampling point or samples. Because of this, motors that contain oil should not be used if the oil could interfere with the analytes of interest. Further, water used in motors should be of known chemistry. Second, when this type of motor eventually cools, it can draw in water from the sampling point, which could cause future cross-contamination problems. Proper decontamination of the pump should include changing internal cooling fluid if the pump is

[2]For example, the plasticizers in flexible PVC can contaminate samples with phthalate esters. The use of silicone rubber tubing, which contains no plasticizers, can obviate this problem; however, the potential for sample bias due to sorption/desorption exists with both materials (Barcelona et al., 1985). These pumps can be used with the intermediate vessel system described above, so that the sample contacts only the intake tubing and vessel, avoiding contact with the pump mechanism tubing. Alternatively, using silicone rubber tubing at the pump head only can minimize this problem (Ho, 1983; Barker and Dickhout, 1988).

to be used in non-dedicated applications. As an alternative, dry sealed motors can be used to avoid these potential problems. Third, extensive or long-term overheating problems may result in motor failure, usually requiring replacement of the motor. CSPs should not be allowed to operate dry, or damage may occur to the pump seals and/or motor. Some CSP designs may be difficult to disassemble in the field for cleaning or repair. For these pumps, if used portably, cleaning is usually performed by flushing the pump and discharge line and washing the exterior surfaces in accordance with ASTM D5088 (ASTM, 2001h).

Gas-Drive Pumps

Gas-drive or gas-displacement pumps are distinguished from gas-lift pumps by the method of water transport. A gas displacement pump forces a discrete column of water to the surface via pressure-induced lift without the extensive mixing of drive gas and water produced by gas-lift devices. Hydrostatic pressure opens the inlet check valve and fills the pump chamber (fill cycle). The inlet check valve closes by gravity after the chamber is filled. Pressurized gas is applied to the chamber, displacing the water up the discharge line (discharge cycle). By releasing the pressure, the cycle can be repeated. A check valve in the discharge line maintains the water in the line above the pump. A pneumatic logic unit, or controller, is used to control the application and release of the drive gas pressure. The lift capability of a gas-displacement pump is directly related to the pressure of the drive gas used.

Although there is a limited interface in gas-displacement pumps between the drive gas and the water, the potential exists for loss of dissolved gases and VOCs across this interface (Barcelona et al., 1983; Gillham et al., 1983). This potential greatly increases if the pump is allowed to discharge completely, which would cause drive gas to be blown up the discharge line. Contamination of the sample may also result from impurities in the drive gas. Typical lifts for gas displacement pumps rarely exceed 250 feet using single-stage compressors; greater lifts can be achieved using two-stage compressors or compressed-gas cylinders. Gas-displacement pumps are available for sampling points as small as 1/2 inch in diameter.

Bladder Pumps

Bladder pumps, also known as gas-operated squeeze pumps or diaphragm pumps, consist of a flexible membrane (bladder) enclosed by a rigid housing. Water enters the bladder under hydrostatic pressure through a check valve at the pump bottom. The inlet check valve closes by gravity after the bladder is filled. Compressed gas is applied to the annular space between the outside of the bladder and pump housing, which squeezes the bladder. This action forces the water out of the bladder and up the discharge line to the surface. By releasing the gas pressure, this cycle can be repeated; a check valve in the discharge line prevents discharged water from re-entering the bladder. In some bladder pump designs, the water and air chambers are reversed, with water entering the annular space between the pump housing and bladder; the bladder is then inflated to displace the water. A pneumatic logic controller controls the application and release of drive gas pressure to the pump. The lift capability of bladder pumps is directly related to the pressure of the drive gas source.

Bladder pumps provide representative samples under a wide range of field conditions. There is no contact between the drive gas and the water in a bladder pump, eliminating the potential for stripping of dissolved gasses and VOCs and the potential for sample contamination by the drive gas. Pressure gradients applied to the sample can be controlled by reducing the drive gas pressure applied to the bladder, thus minimizing disturbance to the sample chemistry. Bladder pumps are recommended for sampling all parameters under a wide variety of field conditions (Parker, 1994; Kearl et al., 1992; Puls et al., 1992; Barcelona et al., 1983; Pohlmann et al., 1991; Unwin and Maltby, 1988; Tai et al., 1991; Pohlmann et al., 1994).

Bladder pump designs are available for use in sampling points as small as 1/2 inch in diameter. Bladder pump flow rates are controlled by adjusting the drive gas pressure or the discharge and refill cycle timing. Where maximum flow rates are too low for purging, secondary purging pumps or packers can be used in conjunction with bladder sampling pumps in order to reduce purge time requirements.

Dual-Acting Piston Pumps

Dual-acting piston pumps consist of a plunger or set of plungers (pistons) moving inside a stationary submerged barrel (cylinder). As the piston travels back and forth in the cylinder, it alternately draws water into the cylinder under suction, then displaces the water from the cylinder. In a dual-acting piston pump, water is simultaneously discharged and drawn in both directions of piston travel. A check valve in each discharge port or in the discharge line is used to prevent discharge water from re-entering the pump. The piston can be cycled manually, or through the use of a pneumatic or mechanical actuator.

Piston pumps can provide representative samples for some parameters (Barcelona et al., 1983; Knobel and Mann, 1993). Samples may be altered due to the suction produced during refill of the pump; this effect is reduced as the pump cycling rate is decreased. Likewise, reducing the pump cycling rate also reduces the pressure applied to the sample, minimizing the potential for sample alteration. If a flow restrictor or valve is used to reduce the discharge rate, the resultant pressure changes could alter sample chemistry (Barcelona et al., 1983; Gillham et al., 1983).

Currently available designs of dual-acting piston pumps will only fit in sampling points that are 2 inches in diameter or larger. The flow rate of a piston pump depends on the inside diameter of the pump cylinder and the stroke length and rate. The ability to control the minimum flow rate for sampling is dependent on the degree to which the stroke rate can be controlled.

Helical Rotor Pumps

Helical rotor pumps, also referred to as progressing cavity pumps, utilize a down-hole rotor and stator assembly driven by an electric motor to displace water through a discharge line to ground surface. Rotation of the helical rotor causes the cavity between the rotor and stator to progress upward, thereby pushing water in a continuous flow upward through the discharge line. In some progressing cavity pumps, the discharge rate can be varied by adjusting the speed of the pump motor between 50 and 500 rpm. The progressing cavity pump is typically suspended in a

well by its discharge line or by a suspension cable. A two-conductor electric cable supplies power from a 12-volt DC power supply and control box to the pump motor.

The operating principle of progressing cavity pumps makes them suitable for collection of samples to be analyzed for VOCs (Imbrigiotta et al., 1988). There is some evidence these pumps may not be suitable for sampling trace metals and other inorganic analytes at higher flow rates due to increased turbidity (Barcelona et al., 1983); to control turbidity, a variable speed pump controller should be used to reduce flow rate. The pressure applied to a sample is directly related to the motor speed, and can be controlled in designs using variable-speed motor controls. Overheating of the motor may raise the temperature of the sample (Parker, 1994).

Progressing cavity pumps require sampling point diameters of at least 2 inches. The relatively low discharge rates attainable with most progressing cavity pumps makes them most useful in applications where purging does not require removal of large volumes of water from monitoring wells. With variable flow rate progressing cavity pumps, once purging is complete the discharge rate may be reduced before samples are collected.

Gear-Drive Pumps

Another type of positive displacement electric submersible pump is the gear-drive pump. In this type of pump, an electric motor drives a pair of PTFE gears. As these gears rotate, their advancing teeth draw water into the pump through the pump intake port and push it through the gears in a continuous flow up the discharge line. The discharge rate can be varied by using the pump controls to adjust the speed of the pump motor. As with many other submersible pumps, the gear-drive pump is usually suspended in a well by its discharge line. Electric power is supplied to the 24-volt DC motor through a cable from the power source and control box at ground surface.

Gear-drive pumps provide good recoveries of dissolved gases, VOCs, trace metals and other inorganics, and mobile colloids (Backhus et al., 1993; Imbrigiotta et al., 1988). However cavitation may occur if the pump is run at high rpm, which could affect dissolved gases or VOCs. The potential for cavitation can be reduced by controlling motor speed. The pressure applied by a gear-drive pump to a sample is directly related to the motor speed, and can be controlled by using the variable-speed motor controls. Gear-drive pumps are constructed of materials acceptable for sampling sensitive groundwater parameters; pump bodies are commonly constructed entirely of stainless steel materials while the gears are constructed of PTFE.

Gear-drive pumps require a sampling point diameter of at least 2 inches. Maximum discharge rates for gear-drive pumps range from more than 3 gallons per minute at lifts of less than 20 feet to 0.25 gallons per minute at lifts of 250 feet. Discharge rates are easily controlled by using the flow control, which adjusts the power supplied to run the pump motor; pump discharge can be adjusted to less than 50 ml/min.

Inertial Lift Pumps

Inertial lift pumps consist of a discharge line (either flexible tubing or rigid pipe) with a ball-check foot valve attached to the lower end of this line. In operation, the pump is lowered into a water column and cycled through reciprocating motion, either through manual action or through the use of a reciprocating mechanical arm mechanism driven by an electric motor or internal combustion engine, to achieve discharge of water. As the pump is moved upward, water that has entered the pump under hydrostatic pressure is lifted upward, held in the pump by the seated foot valve. When the upward motion of the pump is stopped, the inertia of the water column inside the pump carries it up and out of the discharge line. As the pump is pushed downward, the foot valve opens, allowing the pump to refill, and the cycle is repeated to pump water from the sampling point.

Inertial lift pumps can be constructed of any flexible tubing material or rigid discharge pipe that has sufficient strength to tolerate the pump cycling. Typically, these materials include rigid and flexible PVC, PE, PP, and PTFE. Tubing diameters of ¼ inch or ⅜ inch can be used to collect samples from sampling points as small as ½ inch in diameter.

If inertial-lift pumps are cycled rapidly prior to or during sample collection, some loss of VOCs and/or dissolved gasses could occur in the discharge stream. Inertial lift pumps do not cause pressure changes in the sample. However, the cycling action of an inertial lift pump in a sampling point can significantly increase sample turbidity and agitate and aerate the water column within the sampling point. This can result in alteration of concentrations of a wide variety of analytes (including dissolved gases, VOCs, and trace metals) and interference with analytical determinations in the laboratory.

The flow rate of an inertial lift pump is directly related to the cycling rate of the pump. Flexing of the tubing in the sampling point can cause the flow rate to drop. To achieve discharge rates suitable for sample collection, it is necessary to insert a short length of small-diameter flexible tubing into the discharge line to divert a portion of the discharge stream into sample containers.

SAMPLING EQUIPMENT DECONTAMINATION

SOP#: 2006
DATE: 08/11/94
REV. #: 0.0

1.0 SCOPE AND APPLICATION

The purpose of this Standard Operating Procedure (SOP) is to provide a description of the methods used for preventing, minimizing, or limiting cross-contamination of samples due to inappropriate or inadequate equipment decontamination and to provide general guidelines for developing decontamination procedures for sampling equipment to be used during hazardous waste operations as per 29 Code of Federal Regulations (CFR) 1910.120. This SOP does not address personnel decontamination.

These are standard (i.e. typically applicable) operating procedures which may be varied or changed as required, dependent upon site conditions, equipment limitation, or limitations imposed by the procedure. In all instances, the ultimate procedures employed should be documented and associated with the final report.

Mention of trade names or commercial products does not constitute U.S. Environmental Protection Agency (U.S. EPA) endorsement or recommendation for use.

2.0 METHOD SUMMARY

Removing or neutralizing contaminants from equipment minimizes the likelihood of sample cross contamination, reduces or eliminates transfer of contaminants to clean areas, and prevents the mixing of incompatible substances.

Gross contamination can be removed by physical decontamination procedures. These abrasive and non-abrasive methods include the use of brushes, air and wet blasting, and high and low pressure water cleaning.

The first step, a soap and water wash, removes all visible particulate matter and residual oils and grease. This may be preceded by a steam or high pressure water wash to facilitate residuals removal. The second step involves a tap water rinse and a distilled/deionized water rinse to remove the detergent. An acid rinse provides a low pH media for trace metals removal and is included in the decontamination process if metal samples are to be collected. It is followed by another distilled/deionized water rinse. If sample analysis does not include metals, the acid rinse step can be omitted. Next, a high purity solvent rinse is performed for trace organics removal if organics are a concern at the site. Typical solvents used for removal of organic contaminants include acetone, hexane, or water. Acetone is typically chosen because it is an excellent solvent, miscible in water, and not a target analyte on the Priority Pollutant List. If acetone is known to be a contaminant of concern at a given site or if Target Compound List analysis (which includes acetone) is to be performed, another solvent may be substituted. The solvent must be allowed to evaporate completely and then a final distilled/deionized water rinse is performed. This rinse removes any residual traces of the solvent.

The decontamination procedure described above may be summarized as follows:

1. Physical removal
2. Non-phosphate detergent wash
3. Tap water rinse
4. Distilled/deionized water rinse
5. 10% nitric acid rinse
6. Distilled/deionized water rinse
7. Solvent rinse (pesticide grade)
8. Air dry
9. Distilled/deionized water rinse

If a particular contaminant fraction is not present at the site, the nine (9) step decontamination procedure specified above may be modified for site specificity. For example, the nitric acid rinse may be eliminated if metals are not of concern at a site. Similarly, the solvent rinse may be eliminated if organics are not of

concern at a site. Modifications to the standard procedure should be documented in the site specific work plan or subsequent report.

3.0 SAMPLE PRESERVATION, CONTAINERS, HANDLING, AND STORAGE

The amount of sample to be collected and the proper sample container type (i.e., glass, plastic), chemical preservation, and storage requirements are dependent on the matrix being sampled and the parameter(s) of interest.

More specifically, sample collection and analysis of decontamination waste may be required before beginning proper disposal of decontamination liquids and solids generated at a site. This should be determined prior to initiation of site activities.

4.0 INTERFERENCES AND POTENTIAL PROBLEMS

- The use of distilled/deionized water commonly available from commercial vendors may be acceptable for decontamination of sampling equipment provided that it has been verified by laboratory analysis to be analyte free (specifically for the contaminants of concern).

- The use of an untreated potable water supply is not an acceptable substitute for tap water. Tap water may be used from any municipal or industrial water treatment system.

- If acids or solvents are utilized in decontamination they raise health and safety, and waste disposal concerns.

- Damage can be incurred by acid and solvent washing of complex and sophisticated sampling equipment.

5.0 EQUIPMENT/APPARATUS

Decontamination equipment, materials, and supplies are generally selected based on availability. Other considerations include the ease of decontaminating or disposing of the equipment. Most equipment and supplies can be easily procured. For example, soft-bristle scrub brushes or long-handled bottle brushes can be used to remove contaminants. Large galvanized wash tubs, stock tanks, or buckets can hold wash and rinse solutions. Children's wading pools can also be used. Large plastic garbage cans or other similar containers lined with plastic bags can help segregate contaminated equipment. Contaminated liquid can be stored temporarily in metal or plastic cans or drums.

The following standard materials and equipment are recommended for decontamination activities:

5.1 Decontamination Solutions

- Non-phosphate detergent
- Selected solvents (acetone, hexane, nitric acid, etc.)
- Tap water
- Distilled or deionized water

5.2 Decontamination Tools/Supplies

- Long and short handled brushes
- Bottle brushes
- Drop cloth/plastic sheeting
- Paper towels
- Plastic or galvanized tubs or buckets
- Pressurized sprayers (H_2O)
- Solvent sprayers
- Aluminum foil

5.3 Health and Safety Equipment

Appropriate personal protective equipment (i.e., safety glasses or splash shield, appropriate gloves, aprons or coveralls, respirator, emergency eye wash)

5.4 Waste Disposal

- Trash bags
- Trash containers
- 55-gallon drums
- Metal/plastic buckets/containers for storage and disposal of decontamination solutions

6.0 REAGENTS

There are no reagents used in this procedure aside from the actual decontamination solutions. Table 1 (Appendix A) lists solvent rinses which may be required for elimination of particular chemicals. In

general, the following solvents are typically utilized for decontamination purposes:

- 10% nitric acid is typically used for inorganic compounds such as metals. An acid rinse may not be required if inorganics are not a contaminant of concern.
- Acetone (pesticide grade)[(1)]
- Hexane (pesticide grade)[(1)]
- Methanol[(1)]

[(1)] - Only if sample is to be analyzed for organics.

7.0 PROCEDURES

As part of the health and safety plan, a decontamination plan should be developed and reviewed. The decontamination line should be set up before any personnel or equipment enter the areas of potential exposure. The equipment decontamination plan should include:

- The number, location, and layout of decontamination stations.
- Decontamination equipment needed.
- Appropriate decontamination methods.
- Methods for disposal of contaminated clothing, equipment, and solutions.
- Procedures can be established to minimize the potential for contamination. This may include: (1) work practices that minimize contact with potential contaminants; (2) using remote sampling techniques; (3) covering monitoring and sampling equipment with plastic, aluminum foil, or other protective material; (4) watering down dusty areas; (5) avoiding laying down equipment in areas of obvious contamination; and (6) use of disposable sampling equipment.

7.1 Decontamination Methods

All samples and equipment leaving the contaminated area of a site must be decontaminated to remove any contamination that may have adhered to equipment. Various decontamination methods will remove contaminants by: (1) flushing or other physical action, or (2) chemical complexing to inactivate contaminants by neutralization, chemical reaction, disinfection, or sterilization.

Physical decontamination techniques can be grouped into two categories: abrasive methods and non-abrasive methods, as follows:

7.1.1 Abrasive Cleaning Methods

Abrasive cleaning methods work by rubbing and wearing away the top layer of the surface containing the contaminant. The mechanical abrasive cleaning methods are most commonly used at hazardous waste sites. The following abrasive methods are available:

Mechanical

Mechanical methods of decontamination include using metal or nylon brushes. The amount and type of contaminants removed will vary with the hardness of bristles, length of time brushed, degree of brush contact, degree of contamination, nature of the surface being cleaned, and degree of contaminant adherence to the surface.

Air Blasting

Air blasting equipment uses compressed air to force abrasive material through a nozzle at high velocities. The distance between nozzle and surface cleaned, air pressure, time of application, and angle at which the abrasive strikes the surface will dictate cleaning efficiency. Disadvantages of this method are the inability to control the amount of material removed and the large amount of waste generated.

Wet Blasting

Wet blast cleaning involves use of a suspended fine abrasive. The abrasive/water mixture is delivered by compressed air to the contaminated area. By using a very fine abrasive, the amount of materials removed can be carefully controlled.

7.1.2 Non-Abrasive Cleaning Methods

Non-abrasive cleaning methods work by forcing the contaminant off a surface with pressure. In general, the equipment surface is not removed using non-abrasive methods.

Low-Pressure Water

This method consists of a container which is filled with water. The user pumps air out of the container to create a vacuum. A slender nozzle and hose allow the user to spray in hard-to-reach places.

High-Pressure Water

This method consists of a high-pressure pump, an operator controlled directional nozzle, and a high-pressure hose. Operating pressure usually ranges from 340 to 680 atmospheres (atm) and flow rates usually range from 20 to 140 liters per minute.

Ultra-High-Pressure Water

This system produces a water jet that is pressured from 1,000 to 4,000 atmospheres. This ultra-high-pressure spray can remove tightly-adhered surface films. The water velocity ranges from 500 meters/second (m/s) (1,000 atm) to 900 m/s (4,000 atm). Additives can be used to enhance the cleaning action.

Rinsing

Contaminants are removed by rinsing through dilution, physical attraction, and solubilization.

Damp Cloth Removal

In some instances, due to sensitive, non-waterproof equipment or due to the unlikelihood of equipment being contaminated, it is not necessary to conduct an extensive decontamination procedure. For example, air sampling pumps hooked on a fence, placed on a drum, or wrapped in plastic bags are not likely to become heavily contaminated. A damp cloth should be used to wipe off contaminants which may have adhered to equipment through airborne contaminants or from surfaces upon which the equipment was set.

Disinfection/Sterilization

Disinfectants are a practical means of inactivating infectious agents. Unfortunately, standard sterilization methods are impractical for large equipment. This method of decontamination is typically performed off-site.

7.2 Field Sampling Equipment Decontamination Procedures

The decontamination line is setup so that the first station is used to clean the most contaminated item. It progresses to the last station where the least contaminated item is cleaned. The spread of contaminants is further reduced by separating each decontamination station by a minimum of three (3) feet. Ideally, the contamination should decrease as the equipment progresses from one station to another farther along in the line.

A site is typically divided up into the following boundaries: Hot Zone or Exclusion Zone (EZ), the Contamination Reduction Zone (CRZ), and the Support or Safe Zone (SZ). The decontamination line should be setup in the Contamination Reduction Corridor (CRC) which is in the CRZ. Figure 1 (Appendix B) shows a typical contaminant reduction zone layout. The CRC controls access into and out of the exclusion zone and confines decontamination activities to a limited area. The CRC boundaries should be conspicuously marked. The far end is the hotline, the boundary between the exclusion zone and the contamination reduction zone. The size of the decontamination corridor depends on the number of stations in the decontamination process, overall dimensions of the work zones, and amount of space available at the site. Whenever possible, it should be a straight line.

Anyone in the CRC should be wearing the level of protection designated for the decontamination crew. Another corridor may be required for the entry and exit of heavy equipment. Sampling and monitoring equipment and sampling supplies are all maintained outside of the CRC. Personnel don their equipment away from the CRC and enter the exclusion zone through a separate access control point at the hotline. One person (or more) dedicated to decontaminating equipment is recommended.

7.2.1 Decontamination Setup

Starting with the most contaminated station, the decontamination setup should be as follows:

Station 1: Segregate Equipment Drop

Place plastic sheeting on the ground (Figure 2, Appendix B). Size will depend on amount of

equipment to be decontaminated. Provide containers lined with plastic if equipment is to be segregated. Segregation may be required if sensitive equipment or mildly contaminated equipment is used at the same time as equipment which is likely to be heavily contaminated.

Station 2: Physical Removal With A High-Pressure Washer (Optional)

As indicated in 7.1.2, a high-pressure wash may be required for compounds which are difficult to remove by washing with brushes. The elevated temperature of the water from the high-pressure washers is excellent at removing greasy/oily compounds. High pressure washers require water and electricity.

A decontamination pad may be required for the high-pressure wash area. An example of a wash pad may consist of an approximately 1 1/2 foot-deep basin lined with plastic sheeting and sloped to a sump at one corner. A layer of sand can be placed over the plastic and the basin is filled with gravel or shell. The sump is also lined with visqueen and a barrel is placed in the hole to prevent collapse. A sump pump is used to remove the water from the sump for transfer into a drum.

Typically heavy machinery is decontaminated at the end of the day unless site sampling requires that the machinery be decontaminated frequently. A separate decontamination pad may be required for heavy equipment.

Station 3: Physical Removal With Brushes And A Wash Basin

Prior to setting up Station 3, place plastic sheeting on the ground to cover areas under Station 3 through Station 10.
Fill a wash basin, a large bucket, or child's swimming pool with non-phosphate detergent and tap water. Several bottle and bristle brushes to physically remove contamination should be dedicated to this station . Approximately 10 - 50 gallons of water may be required initially depending upon the amount of equipment to decontaminate and the amount of gross contamination.

Station 4: Water Basin

Fill a wash basin, a large bucket, or child's swimming pool with tap water. Several bottle and bristle brushes should be dedicated to this station. Approximately 10-50 gallons of water may be required initially depending upon the amount of equipment to decontaminate and the amount of gross contamination.

Station 5: Low-Pressure Sprayers

Fill a low-pressure sprayer with distilled/deionized water. Provide a 5-gallon bucket or basin to contain the water during the rinsing process. Approximately 10-20 gallons of water may be required initially depending upon the amount of equipment to decontaminate and the amount of gross contamination.

Station 6: Nitric Acid Sprayers

Fill a spray bottle with 10% nitric acid. An acid rinse may not be required if inorganics are not a contaminant of concern. The amount of acid will depend on the amount of equipment to be decontaminated. Provide a 5-gallon bucket or basin to collect acid during the rinsing process.

Station 7: Low-Pressure Sprayers

Fill a low-pressure sprayer with distilled/deionized water. Provide a 5-gallon bucket or basin to collect water during the rinsate process.

Station 8: Organic Solvent Sprayers

Fill a spray bottle with an organic solvent. After each solvent rinse, the equipment should be rinsed with distilled/deionized water and air dried. Amount of solvent will depend on the amount of equipment to decontaminate. Provide a 5-gallon bucket or basin to collect the solvent during the rinsing process.

Solvent rinses may not be required unless organics are a contaminant of concern, and may be eliminated from the station sequence.

Station 9: Low-Pressure Sprayers

Fill a low-pressure sprayer with distilled/deionized water. Provide a 5-gallon bucket or basin to collect water during the rinsate process.

Station 10: Clean Equipment Drop

Lay a clean piece of plastic sheeting over the bottom

plastic layer. This will allow easy removal of the plastic in the event that it becomes dirty. Provide aluminum foil, plastic, or other protective material to wrap clean equipment.

7.2.2 Decontamination Procedures

Station 1: Segregate Equipment Drop

Deposit equipment used on-site (i.e., tools, sampling devices and containers, monitoring instruments radios, clipboards, etc.) on the plastic drop cloth/sheet or in different containers with plastic liners. Each will be contaminated to a different degree. Segregation at the drop reduces the probability of cross contamination. Loose leaf sampling data sheets or maps can be placed in plastic zip lock bags if contamination is evident.

Station 2: Physical Removal With A High-Pressure Washer (Optional)

Use high pressure wash on grossly contaminated equipment. Do not use high- pressure wash on sensitive or non-waterproof equipment.

Station 3: Physical Removal With Brushes And A Wash Basin

Scrub equipment with soap and water using bottle and bristle brushes. Only sensitive equipment (i.e., radios, air monitoring and sampling equipment) which is waterproof should be washed. Equipment which is not waterproof should have plastic bags removed and wiped down with a damp cloth. Acids and organic rinses may also ruin sensitive equipment. Consult the manufacturers for recommended decontamination solutions.

Station 4: Equipment Rinse

Wash soap off of equipment with water by immersing the equipment in the water while brushing. Repeat as many times as necessary.

Station 5: Low-Pressure Rinse

Rinse sampling equipment with distilled/deionized water with a low-pressure sprayer.

Station 6: Nitric Acid Sprayers (required only if metals are a contaminant of concern)

Using a spray bottle rinse sampling equipment with nitric acid. Begin spraying (inside and outside) at one end of the equipment allowing the acid to drip to the other end into a 5-gallon bucket. A rinsate blank may be required at this station. Refer to Section 9.

Station 7: Low-Pressure Sprayers

Rinse sampling equipment with distilled/deionized water with a low-pressure sprayer.

Station 8: Organic Solvent Sprayers

Rinse sampling equipment with a solvent. Begin spraying (inside and outside) at one end of the equipment allowing the solvent to drip to the other end into a 5-gallon bucket. Allow the solvent to evaporate from the equipment before going to the next station. A QC rinsate sample may be required at this station.

Station 9: Low-Pressure Sprayers

Rinse sampling equipment with distilled/deionized water with a low-pressure washer.

Station 10 : Clean Equipment Drop

Lay clean equipment on plastic sheeting. Once air dried, wrap sampling equipment with aluminum foil, plastic, or other protective material.

7.2.3 Post Decontamination Procedures

1. Collect high-pressure pad and heavy equipment decontamination area liquid and waste and store in appropriate drum or container. A sump pump can aid in the collection process. Refer to the Department of Transportation (DOT) requirements for appropriate containers based on the contaminant of concern.

2. Collect high-pressure pad and heavy equipment decontamination area solid waste and store in appropriate drum or container. Refer to the DOT requirements for appropriate containers based on the contaminant of concern.

3. Empty soap and water liquid wastes from basins and buckets and store in appropriate

drum or container. Refer to the DOT requirements for appropriate containers based on the contaminant of concern.

4. Empty acid rinse waste and place in appropriate container or neutralize with a base and place in appropriate drum. pH paper or an equivalent pH test is required for neutralization. Consult DOT requirements for appropriate drum for acid rinse waste.

5. Empty solvent rinse sprayer and solvent waste into an appropriate container. Consult DOT requirements for appropriate drum for solvent rinse waste.

6. Using low-pressure sprayers, rinse basins, and brushes. Place liquid generated from this process into the wash water rinse container.

7. Empty low-pressure sprayer water onto the ground.

8. Place all solid waste materials generated from the decontamination area (i.e., gloves and plastic sheeting, etc.) in an approved DOT drum. Refer to the DOT requirements for appropriate containers based on the contaminant of concern.

9. Write appropriate labels for waste and make arrangements for disposal. Consult DOT regulations for the appropriate label for each drum generated from the decontamination process.

8.0 CALCULATIONS

This section is not applicable to this SOP.

9.0 QUALITYASSURANCE/ QUALITY CONTROL

A rinsate blank is one specific type of quality control sample associated with the field decontamination process. This sample will provide information on the effectiveness of the decontamination process employed in the field.

Rinsate blanks are samples obtained by running analyte free water over decontaminated sampling equipment to test for residual contamination. The blank water is collected in sample containers for handling, shipment, and analysis. These samples are treated identical to samples collected that day. A rinsate blank is used to assess cross contamination brought about by improper decontamination procedures. Where dedicated sampling equipment is not utilized, collect one rinsate blank per day per type of sampling device samples to meet QA2 and QA3 objectives.

If sampling equipment requires the use of plastic tubing it should be disposed of as contaminated and replaced with clean tubing before additional sampling occurs.

10.0 DATA VALIDATION

Results of quality control samples will be evaluated for contamination. This information will be utilized to qualify the environmental sample results in accordance with the project's data quality objectives.

11.0 HEALTH AND SAFETY

When working with potentially hazardous materials, follow OSHA, U.S. EPA, corporate, and other applicable health and safety procedures.

Decontamination can pose hazards under certain circumstances. Hazardous substances may be incompatible with decontamination materials. For example, the decontamination solution may react with contaminants to produce heat, explosion, or toxic products. Also, vapors from decontamination solutions may pose a direct health hazard to workers by inhalation, contact, fire, or explosion.

The decontamination solutions must be determined to be acceptable before use. Decontamination materials may degrade protective clothing or equipment; some solvents can permeate protective clothing. If decontamination materials do pose a health hazard, measures should be taken to protect personnel or substitutions should be made to eliminate the hazard. The choice of respiratory protection based on contaminants of concern from the site may not be appropriate for solvents used in the decontamination process.

Safety considerations should be addressed when using abrasive and non-abrasive decontamination

equipment. Maximum air pressure produced by abrasive equipment could cause physical injury. Displaced material requires control mechanisms.

Material generated from decontamination activities requires proper handling, storage, and disposal. Personal Protective Equipment may be required for these activities.

Material safety data sheets are required for all decontamination solvents or solutions as required by the Hazard Communication Standard (i.e., acetone, alcohol, and trisodiumphosphate).

In some jurisdictions, phosphate containing detergents (i.e., TSP) are banned.

12.0 REFERENCES

Field Sampling Procedures Manual, New Jersey Department of Environmental Protection, February, 1988.

A Compendium of Superfund Field Operations Methods, EPA 540/p-87/001.

Engineering Support Branch Standard Operating Procedures and Quality Assurance Manual, USEPA Region IV, April 1, 1986.

Guidelines for the Selection of Chemical Protective Clothing, Volume 1, Third Edition, American Conference of Governmental Industrial Hygienists, Inc., February, 1987.

Occupational Safety and Health Guidance Manual for Hazardous Waste Site Activities, NIOSH/OSHA/USCG/EPA, October, 1985.

APPENDIX A

Table

Table 1. Soluble Contaminants and Recommended Solvent Rinse

TABLE 1 Soluble Contaminants and Recommended Solvent Rinse		
SOLVENT[1]	EXAMPLES OF SOLVENTS	SOLUBLE CONTAMINANTS
Water	Deionized water Tap water	Low-chain hydrocarbons Inorganic compounds Salts Some organic acids and other polar compounds
Dilute Acids	Nitric acid Acetic acid Boric acid	Basic (caustic) compounds (e.g., amines and hydrazines)
Dilute Bases	Sodium bicarbonate (e.g., soap detergent)	Acidic compounds Phenol Thiols Some nitro and sulfonic compounds
Organic Solvents [2]	Alcohols Ethers Ketones Aromatics Straight chain alkalines (e.g., hexane) Common petroleum products (e.g., fuel, oil, kerosene)	Nonpolar compounds (e.g., some organic compounds)
Organic Solvent[2]	Hexane	PCBs

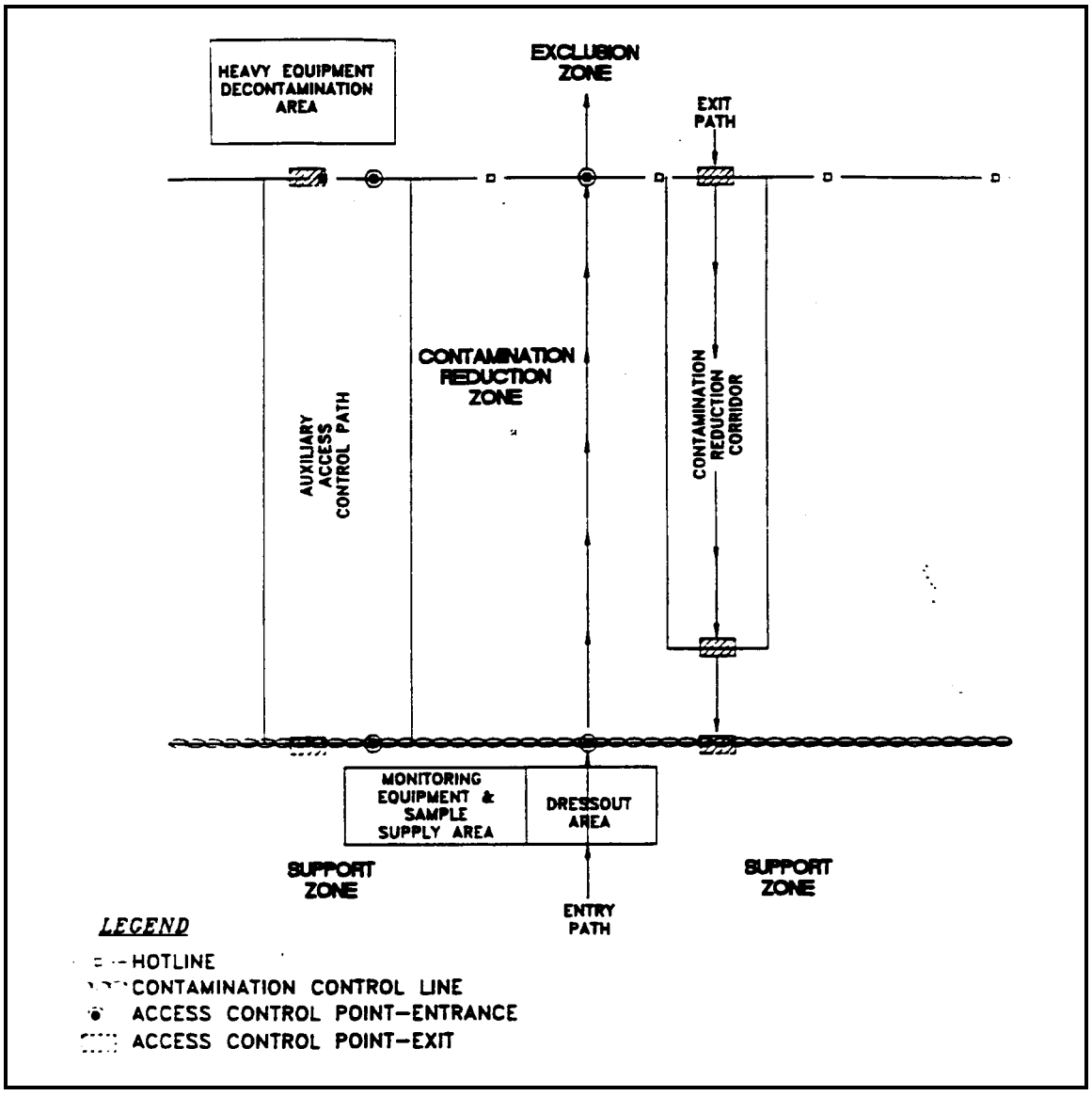
[1] - Material safety data sheets are required for all decontamination solvents or solutions as required by the Hazard Communication Standard

[2] - WARNING: Some organic solvents can permeate and/or degrade the protective clothing

APPENDIX B

Figures

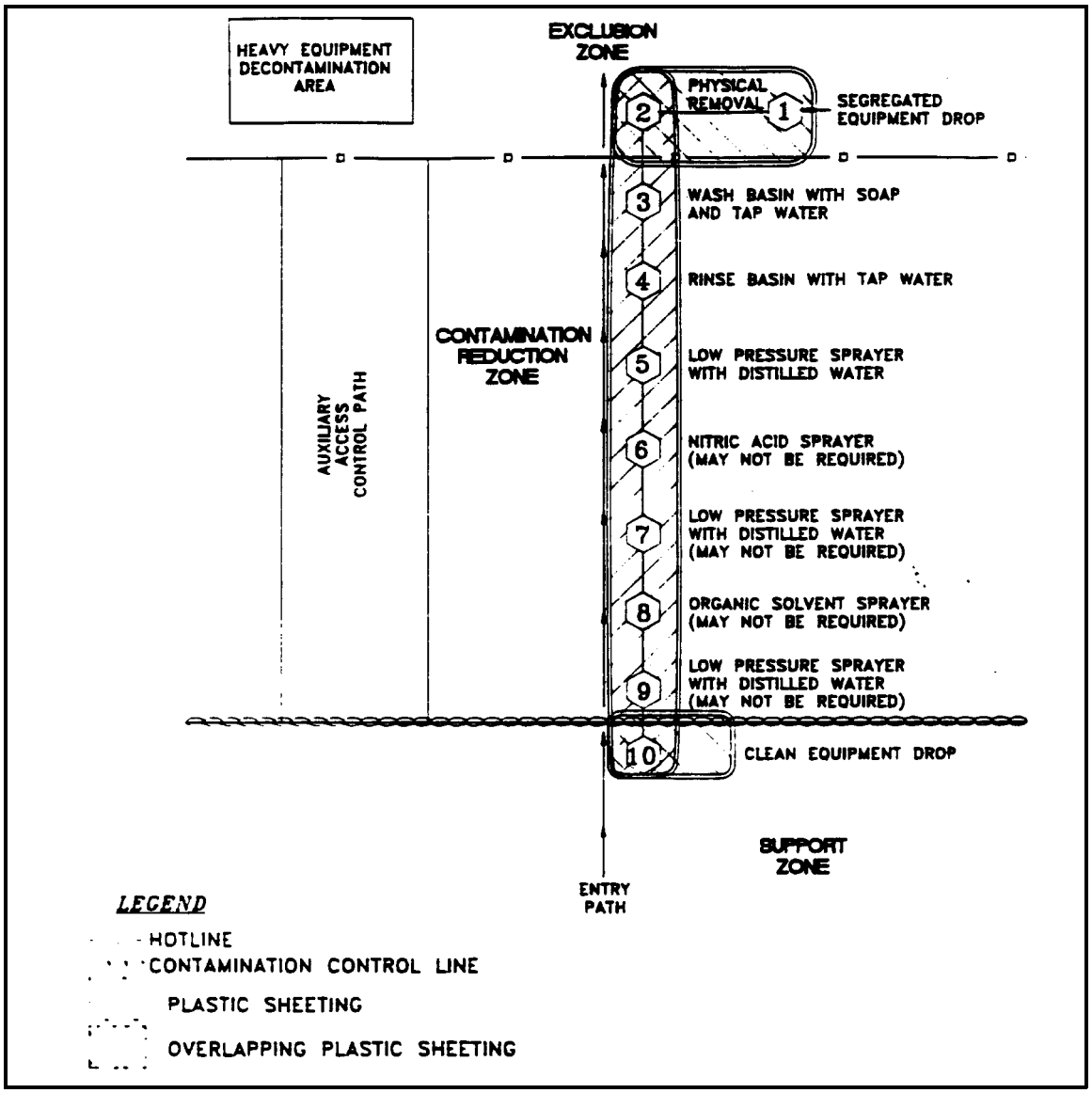
Figure 1. Contamination Reduction Zone Layout



APPENDIX B (Cont'd.)

Figures

Figure 2. Decontamination Layout



APPENDIX C

Chain-of-Custody Forms

Chain of Custody

Page 1 of 1

McGinley and Associates
6280 S Valley Blvd Ste604
Las Vegas, NV 89118
MCGINLVNV

Billing Information:
McGinley and Associates
6280 S Valley Blvd., Suite 604
Las Vegas, NV 89118

Report to: Brian Rakvica

Email to: brakvica@mcgin.com

Project Description:

City/Sate Collected

Phone: 702-260-4961
FAX:

Client Project #:

ESC Key: MCGINLVNV

Collected by:

Site/Facility ID#:

P.O.#:

Collected by (signature):

Immediately Packed on Ice N ____ Y ___

Rush? (Lab MUST Be Notified)
____ Same Day. 200%
____ Next Day.. . . . . 100%
____ Two Day 50%
Three Day 25%

Date Results Needed:

Email? __No__Yes
FAX? __No__Yes

No. of Cntrs

Analysis/Container/Preservative

SV8270PAHSIM - 8270SIM

12065 Lebanon Road
Mt. Juliet, TN 37122

Phone: (800) 767-5859
Phone: (615) 758-5858
Fax: (615) 758-5859

CoCode MCGINLVNV (lab use only)

Template/Prelogin

Shipped Via:

Sample ID	Comp/Grab	Matrix*	Depth	Date	Time	Remarks/Contaminant	Sample # (lab only)

*Matrix: **SS** - Soil/Solid **GW** - Groundwater **WW** - WasteWater **DW** - Drinking Water **OT** - Other_____

pH _____ Temp _____

Remarks:

Flow _____ Other _____

Relinquished by: (Signature)	Date:	Time:	Received by: (Signature)	Samples returned via: ☐ UPS ☐ FedEx ☐ Courier ☐ _____	Condition: (lab use only)	
Relinquished by: (Signature)	Date:	Time:	Received by: (Signature)	Temp:	Bottles Received:	CoC Seals Intact: ____ Y ____ N _____ NA
Relinquished by: (Signature)	Date:	Time:	Received for lab by: (Signature)	Date:	Time:	pH Checked: / NCF:

APPENDIX D

Sample Labels

Prepared by Environmental Science Corp

Project _____

Proj # _____

Sample Location/ID _____

Analysis Reqd _____

_____

Date _____ Time _____

APPENDIX E

Site Health and Safety Plan

McGinley & Associates

Reno Office
815 Maestro Drive
Reno, Nevada 89511
Ph: 775.829.2245

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6280 South Valley View Boulevard
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www.mcgin.com

- Site Remediation
- Soil & Groundwater Investigations
- Closure Optimization
- Brownfields Redevelopment
- Permitting & Compliance
- Phase I Assessments
- Storm Water & Spill Plans
- Underground Tank Services
- Toxic Release Inventory
- Geographic Information Systems
- Groundwater Modeling
- Litigation Support & Expert Witness

HEALTH AND SAFETY PLAN

Vacant 7-Acre Parcel
APN: 002-770-005
Wells
Elko County
Nevada
NDEP Contract #10-008-04
Task M02-13

Prepared for:

State of Nevada
Department of Conservation and Natural Resources
Division of Environmental Protection
Bureau of Corrective Actions
901 S. Stewart Street, Suite 4001
Carson City, Nevada 89701-5249

On behalf of:

City of Wells

January 24, 2013

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FIGURES

APPENCDICES

1. INTRODUCTION

McGinley & Associates Inc. (MGA) is pleased to submit this Health and Safety Plan (HASP) detailing personal safety precautions being performed on behalf of the Nevada Division of Environmental Protection (NDEP). This HASP addresses activities associated with the collection of soil and groundwater samples. The sampling activities will be conducted on a vacant seven-acre parcel of land located in Wells, Nevada.

Planned site activities will include:

- Site reconnaissance;
- GPR survey;
- Direct push advancement of borings to groundwater;
- Collection of soil samples for laboratory analysis; and
- Collection of groundwater samples for laboratory analysis.

1.1 Scope and Applicability of the Site Health and Safety Plan

The purpose of this HASP is to define the requirements and designate protocols to be followed for the site survey and sampling activities. Applicability extends to all MGA employees, contractors and subcontractors. Each person will also be expected to provide his or her own protective equipment.

All on-site personnel shall be informed of the site emergency response procedures and any potential fire, explosion, health, or safety hazards of the operation. This HASP summarizes hazards and defines protective measures planned for the site. This plan must be reviewed and signed by all site personnel prior to commencing with field activities. An agreement of compliance is provided in Appendix A.

During development of this plan, consideration was given to current safety standards as defined by EPA/OSHA/NIOSH, health effects and standards for known contaminants, and procedures designed to account for the potential for exposure to unknown substances. Specifically, the following reference sources have been consulted:

- OSHA 29 CFR parts 1910.120, 1910.134, 1926.350 and 1926.650;
- U.S. EPA, OERR ERT Standard Operating Safety Guides
- NIOSH/OSHA/USCG/EPA Occupational Health and Safety Guidelines
- (ACGIH) Threshold Limit Values

1.2 On-Site Personnel

All personnel entering the designated work areas at the Site are responsible for the following:

- Taking all reasonable precautions to prevent injury to themselves and to their fellow employees, and being alert to potentially harmful situations;
- Obeying all applicable laws and regulations relating to health and safety;
- Ensuring that activities do not impact the neighboring community;
- Performing only those tasks that they have been trained to complete and can do safely;
- Notifying their supervisor of any special medical conditions (i.e., allergies, contact lenses, diabetes) that may affect their ability to perform certain tasks;

- Notifying their supervisor of any prescription and/or non-prescription medication that they may be taking that might cause drowsiness, anxiety, or other unfavorable side-affects;
- Learning and complying with Site security requirements;
- Complying with the Site's prohibition on drug and alcohol use, smoking, horseplay, and restricted eating/drinking areas;
- Practicing good housekeeping by keeping the work areas neat, clean and orderly;
- Immediately reporting all injuries, incidents and near-misses to the HSO;
- Properly using PPE specified by this HASP.
- Properly maintaining their designated PPE per manufacturers' recommendations.
- Complying with the HASP and all health and safety recommendations and precautions.

In the event that a person does not adhere to the provisions of the HASP, he/she will be requested to leave the work area. All non-conformance incidents will be recorded in the site log.

2. KEY PERSONNEL

The Site Health and Safety Officer (HSO) is fully responsible for ensuring the provisions of this HASP are adequate and implemented in the field. Changing field conditions may require decisions to be made concerning adequate protection programs. Therefore, it is vital that personnel assigned as HSO be experienced and meet the additional training requirements specified by OSHA in 29 CFR 1910.120. The following personnel are critical to the planned activities at the Site. The organizational structure will be reviewed and updated periodically by the site supervisor.

Title/Responsibility	Name	Phone
City of Wells		
Site Contact	Jolene Supp	(775) 752-3355
McGinley and Associates, Inc.		
Project Manager – Project management, regulatory liaison, coordinate field activities, site safety, data review, report preparation.	Brett Bottenberg	(702) 232-5247
Environmental Scientist – Collect soil and groundwater samples.	Justin Fike	(702) 371-7864
Contractors/Vendors		
ESC Lab Sciences – Analysis of soil samples	Dave Veratti	(615) 758-5858
GPRS – Search for orphaned USTs	Jim Cox	(775) 560-8913
Earth Probe Environmental Field Services – Boring advancement	Patrick Casey	(801) 466-3752

2.1 Site Specific Health and Safety Personnel

The HSO is also responsible for conducting site inspections on a regular basis in order to ensure the effectiveness of this plan. The HSO at the site is Justin Fike, Staff Scientist for MGA. The designated alternate is George Hagan, Staff Technician for MGA.

2.2 Organizational Responsibility

City of Wells:	Party initiating investigation of soil impacts from previous dumping and staging activities.
MGA:	Primary agent for the City of Wells providing field services and project oversight of surveys and sampling.
Subcontractors:	Various companies and organizations providing services or skilled trades.

3. TASK/OPERATION SAFETY AND HEALTH RISK ANALYSIS

3.1 Historical Overview of Site

Currently, the site is a vacant and undeveloped parcel. Based on available historical information, it appears that the site was developed in the past. Aerial photographs suggest that the site may have been developed with several structures in 1967. However, the photograph is not clear and it is difficult to discern details of the structures.

In addition, ownership information provided by the Elko County Assessor's Office appears to indicate that the property may have formerly been a gas station. Chevron USA, Inc. is listed as an owner up until 2001. The number of years owned by Chevron prior to 2001 is unknown.

In May of 2012, MGA conducted a Phase I ESA on the study area. The ESA was conducted in compliance with the American Society for Testing and Materials (ASTM) Standard E-1527-05 to identify any recognized environmental conditions (RECs) at the Site. The proposed sampling assessment is based on the findings of this Phase I ESA which are previously presented in Section 1.5 of this Sampling and Analysis Plan.

3.2 Chemical Hazards

The following sections provide descriptions of the principal health hazards of the potential contaminants affecting this investigation and include:

- Volatile Organic Compounds (VOCs);
- Semi-VOCs (SVOCs); and/or
- Total Petroleum Hydrocarbons (TPH).

3.2.1 Organic Compounds

Volatile Organic Compounds (VOCs) and Semi Volatile Organic Compounds (SVOCs) are commonly found in oils, fuels, paints, solvents, and other chemicals that may have been disposed improperly during construction activities. Exposure to the vapors of these compounds above their respective OSHA permissible exposure limits (PELs) may produce irritation of the mucous membranes of the upper respiratory tract, nose, and mouth. Symptoms of such exposure include drowsiness, headache, fatigue, and "drunken-like" behaviors. Chronic and prolonged overexposure to the vapors of benzene may cause damage to the blood-forming organs and is known to cause leukemia in humans. In addition, these compounds may also present hazards through dermal absorption. As field activities normally involve subsurface disturbance for generally short periods of time, these pathways should be considered. Planning, development, and implementation of specific sampling protocol should be conducted to mitigate these potential concerns.

3.2.2 Petroleum Hydrocarbons

Petroleum hydrocarbons such as gasoline and diesel fuel are comprised of a wide range of substances, some of which may pose substantive human health hazards. Constituents including benzene, toluene, ethyl benzenes, and xylenes (BTEX) are generally a greater concern due to their potential exposure pathway through the lungs. In moderate exposures, BTEX compounds all produce similar acute effects including headaches, narcosis, and anesthesia. Among these compounds, benzene is the primary substance of concern due to its status as a known carcinogen and its association with leukemia and aplastic anemia in chronic exposure situations.

As field activities normally involve subsurface disturbance for generally short periods of time, these pathways should be considered. Planning, development, and implementation of specific sampling protocol should be conducted to mitigate these potential concerns.

3.3 Biological Hazards

The vacant parcel may contain spiders, snakes, and other types of natural hazards. Boots and protective clothing should be inspected for spiders prior to putting them on. Snakes should be avoided to prevent snakebites. If a spider or snake bit occurs, the HSO shall be notified immediately and the victim should be transported to Northeastern Nevada Regional Hospital in Elko, Nevada.

3.4 General Hazards

General hazards that may be encountered during sampling activities and preventative measures are described in the following sections and include:

- Slips, trips, and falls
- Elevated noise levels
- Hazards associated with lifting and carrying
- Extreme weather conditions

3.4.1 Slips, Trips, and Falls

Protection from slips and trips can be curbed by utilizing common sense and being aware of your surroundings. Falls are a leading cause of occupational fatalities. These fatalities are considered preventable with the use of fall protection systems. The following is a list of common fall hazards:

- Elevated work at > 6 feet above lower level with unprotected sides or edges

- Wall openings > 4 feet above lower level
- Floor/Roof openings (hatches)
- Floor/Roof holes (deterioration), i.e. failing roof
- Ramps, walkways, bridges
- Excavations

Protection from fall hazards can be achieved in one of three ways: 1) fixed position systems, 2) personal fall protection, and 3) safety monitoring systems. A combination of these three protection systems is often used to ensure the safety of site workers. Fixed position systems consist of guardrails, safety nets, and floor covers. Personal fall protection will consist of a full-body harness with a 6-foot shock-absorbing lanyard. Good housekeeping, proper PPE, and daily safety meetings can minimize injuries from falls.

3.4.2 Elevated Noise Levels

During on-site activities requiring the use of power equipment, hearing protection may be required to be worn for certain tasks or in designated areas where noise levels reach > 85 dBA. Training on proper use of hearing protection will be conducted prior to initiation of specified onsite work.

3.4.3 Hazards Associated with Lifting and Carrying

The human body is subject to severe damage in the form of back injury and/or hernia if caution is not observed in the handling process. General rules for minimizing injuries from manual lifting are:

- Get good footing.
- Place feet shoulder width apart.
- BEND AT KNEES to grasp object.
- Keep back straight.
- Get a good grip on object.
- Lift gradually by straightening the legs.
- GET HELP if object is too heavy for you to lift (usually 50-60 lbs lifting limit).

3.4.4 Extreme Weather Conditions

Extreme weather may occur at any time. Since the site activities are anticipated to be performed in the late winter or spring, it is anticipated that temperatures at the site during the proposed activities may exist at levels below freezing.

3.5 Task Hazard Analysis

3.5.1 Ground Penetrating Radar Survey

The ground penetrating radar (GPR) survey will consist of equipment rolling across the ground surface. Hazards from this task may include slips, trips, falls, and extreme weather conditions. In addition, biological hazards at the site may pose as a hazard to GPR technicians.

3.5.2 Boring Advancement

Boring advancement activities may disturb the soil in such a way that causes dust to become airborne. If this occurs, the risk of respiratory exposure goes up. In addition, dermal contact may occur if care is not taken to avoid contact with skin. Care should be taken to avoid the previously stated actions.

3.5.3 Collection of Soil and Groundwater Samples

Dermal contact may occur during collection of soil and groundwater samples if care is not taken to avoid contact with skin. Care should be taken to avoid the previously stated actions.

4. PERSONNEL TRAINING REQUIREMENTS

Consistent with OSHA's 29 CFR 1910.120, regulation covering Hazardous Waste Operations and Emergency Response and OSHA's 29 CFR 1926 Construction Industry Standards, workers are required to be trained in accordance with those standards. At a minimum, all personnel are required to be trained to recognize the hazards on-site and the provisions of this HASP.

4.1 Pre-assignment and Annual Refresher Training

Prior to arrival on site, each employer will be responsible for certifying that his/her employees meet the requirements of training, consistent with OSHA 29 CFR 1910.120 paragraph (e)(3) or (e)(9). The employer should be able to provide a document certifying that each general site worker has received 40 hours of instruction off the site, and 24 hours of training for any workers who are on site only occasionally for a specific task. If an individual employee has work experience and/or training that is equivalent to that provided in the initial training, an employer may waive the 40-hour training so long as that equivalent experience is documented or certified. All personnel must also receive 8 hours of refresher training annually.

4.2 Training and Briefing Topics

The following items may be discussed by a qualified individual at the site pre-entry briefing(s) and at periodic tailgate safety meetings.

Physical Hazards	Chemical Hazards
Emergency Response Plan	Air Monitoring
Training Requirements	Animal Bites and Stings
Respiratory Protection	Medical Surveillance
Site Control	Personal Protective Equipment
Heavy Machinery	

5. PERSONAL PROTECTIVE EQUIPMENT TO BE USED

This section describes the general requirements of the EPA designated Levels of Protection (A-D), and the specific levels of protection required for each task at the site.

5.1 Levels of Protection

Personnel wear protective equipment when response activities involve known or suspected atmospheric contamination vapors, gases, or particulate that may be generated by site activities, or when direct contact with skin-affecting substances may occur. The specific levels of protection and necessary components for each have been divided into four categories according to the degrees of protection afforded:

Level A: Should be worn when the highest level of respiratory, skin, and eye protection is needed.

Level B: Should be worn when the highest level of respiratory protection is needed, but a lesser level of skin protection. Level B is the primary level of choice when encountering unknown environments.

Level C: Should be worn when the criteria for using air-purifying respirators are met, and a lesser level of skin protection is needed.

Level D: Should be worn only as a work uniform and not in any area with respiratory or skin hazards. It provides minimal protection against chemical hazards.

Modifications of these levels are permitted, and routinely employed during site work activities to maximize efficiency. For example, Level C respiratory protection and Level D skin protection may be required for a given task. Likewise the type of chemical protective ensemble (i.e., material, format) will depend upon contaminants and degrees of contact. The Level of Protection selected is based upon the following:

- Type and measured concentration of the chemical substance in the ambient atmosphere and its toxicity.
- Potential for exposure to substances in air, liquids, or other direct contact with material due to work being done.
- Knowledge of chemicals on-site along with properties such as toxicity, route of exposure, contaminant matrix, and adequate warning properties.

In situations where the type of chemical, concentration, and possibilities of contact are not known, the appropriate Level of Protection must be selected based on professional experience and judgment until the hazards can be better identified. For all unknown situations on this site, Level D is the highest level anticipated.

5.2 Recommended Levels of Protection – Task Specific

The following specific personal protective ensembles are recommended for the site:

***GPR Survey / Site Reconnaissance* - (Level D)**

- Outer Gloves - Nitrile
- Outer Garment/Coveralls - Tyvek
- Hardhat
- Safety Glasses
- Steel-toed Boots

***Boring Advamcement* - (Level D)**

- Outer Gloves - Nitrile
- Outer Garment/Coveralls - Tyvek
- Hardhat
- Safety Glasses
- Steel-toed Boots

***Soil and Groundwater Sampling* - (Level D)**

- Outer Gloves - Nitrile
- Outer Garment/Coveralls - Tyvek
- Hardhat
- Safety Glasses
- Steel-toed Boots

5.3 Reassessment of Protection Program

The level of Protection provided by PPE selection shall be upgraded or downgraded based upon a change in site conditions or findings of investigations. When a significant change occurs, the hazards should be reassessed and the HASP updated. Some indicators of the need for reassessment are:

- Commencement of a new work phase, such as the start of unexpected sampling or work that begins on a different portion of the site;
- Change in job tasks during a work phase;
- Contaminants other than those previously identified are encountered;
- Change in ambient levels of contaminants;
- Change in work scope which affects the degree of contact with contaminants.

5.4 SOP for Personal Protective Equipment

Proper inspection of PPE features several sequences of inspection depending upon specific articles of PPE and its frequency of use. The different levels of inspection are as follows:

- Inspection and operational testing of equipment received from the factory or distributor;
- Inspection of equipment as it is issued to workers;
- Inspection after use or training and prior to maintenance;
- Periodic inspection of stored equipment; and
- Periodic inspection when a question arises concerning the appropriateness of the selected equipment, or when problems with similar equipment arise.

The primary inspection of PPE in use for activities at the site will occur prior to immediate use and will be conducted by the user. This ensures that the specific device or article has been checked-out by the user and that the user is familiar with its use.

6. MEDICAL SURVEILLANCE REQUIREMENTS

Medical monitoring programs are designed to track the physical condition of employees on a regular basis as well as survey pre-employment or baseline conditions prior to potential exposures. The medical surveillance program is a part of each employers Health and Safety program. Exposure to toxic materials is not anticipated at the Site.

6.1 Exposure/Injury/Medical Support

As a follow-up to an injury or possible exposure above established exposure limits, all employees are entitled to and encouraged to seek medical attention and physical testing. Depending upon the type of exposure, it is critical to perform follow-up testing within 24-28 hours. It will be up to the employer's medical consultant to advise the type of test required to accurately monitor for exposure effects.

7. EXPOSURE MONITORING/AIR MONITORING

Exposure monitoring will not take place at the Site.

8. SITE CONTROL MEASURES

The following section defines measures and procedures for maintaining site control. Site control is an essential component in the implementation of the site health and safety program.

8.1 Site Communications Plan

Successful communications between field teams and contact with personnel in the support zone is essential. The following communications systems will be available during activities at the site.

- Hand Signals
- Verbal
- Honk Vehicle Horn - Evacuate immediately

Signal	Definition
Hands on top of head	Need assistance
Thumbs up	OK/I am all right/I understand
Thumbs down	No/negative
Arms waving upright	Send backup support
Grip partners wrist	Exit area immediately

8.2 Safe Work Practices

The following is a list of standing orders for the duration of the project.

- No smoking, eating, or drinking in areas where there is a potential of cross contamination or risk of fire or explosion.
- No horse play.

- Implement the communications system.
- Line of sight must be in position.
- Wear the appropriate level of protection as defined in the Safety Plan.
- No unauthorized entry into hazardous work areas by unauthorized personnel l

9. DECONTAMINATION PLAN

Consistent with the levels of protection required, the decontamination process provides a step by step representation of the personnel decontamination steps for level D and C. These procedures should be modified to suit site conditions and protective ensembles in use. Decontamination involves the orderly controlled removal of contaminants. All site personnel should minimize contact with contaminants in order to minimize the need for extensive decontamination.

9.1 Personnel Decontamination

All workers exposed to COCs will be required to enact an orderly removal of contaminated PPE. This can be accomplished through repeated change of disposable garments and or PPE wash at the end of the shift. Workers shall be instructed to the importance of decontamination to prevent cross contamination.

9.2 Sampling Equipment Decontamination

Sampling equipment and heavy equipment may be decontaminated in accordance with procedures as defined in the work plan or as follows:

- Sampling equipment will be rinsed using water and a 5% tri-sodium phosphate solution (or an acceptable substitute).
- Sampling equipment will be decontaminated between sample collections to prevent cross contamination.

Disposable sampling equipment shall be utilized wherever practical to minimize employee exposure and possible cross contamination between sampling events.

10. EMERGENCY RESPONSE/CONTINGENCY PLAN

This section describes contingencies and emergency planning procedures to be implemented at the Site. This plan is compatible with local, state, and federal disaster and emergency management plans as appropriate.

10.1 Pre-Emergency Planning

A field pre-construction / field activities meeting will be conducted at the project site prior to implementation of field services. The meeting will include personnel from MGA and selected contractors, if applicable. Each of the activities and procedures presented will be reviewed during this meeting.

In addition, tailgate site safety discussions will be held daily. All employees will be trained in and reminded of provisions of the emergency response plan, communication systems, and evacuation routes. The plan will be reviewed and revised if necessary, on a regular basis by the HSO. This will ensure that the plan is adequate and consistent with prevailing site conditions.

10.2 Emergency Recognition/Prevention

Section 3 provides a listing of chemical hazards onsite. Additional hazards as a direct result of site activities are listed in Section 3.2 as are prevention and control techniques/mechanisms. Personnel will be familiar with techniques of hazard recognition from pre-assignment training and site specific briefings. The HSO is responsible for ensuring that prevention devices or equipment is available to personnel.

10.3 Evacuation Routes/Procedures

Since all individuals sampling will be within shouting distance, no special alarm system is anticipated as necessary. Contact appropriate emergency authorities. No other situation calling for site evacuation is reasonably anticipated.

10.4 Emergency Contact/Notification System

The following list provides names and telephone numbers for emergency contact personnel. In the event of a medical emergency, personnel will take direction from the HSO and notify the appropriate emergency organization. In the event of a fire or spill, the site supervisor will notify the appropriate local, state, and federal agencies.

Organization	**Telephone**
Ambulance:	911
Police:	911
Fire:	911
Northeastern Nevada Regional Hospital	(775) 738-5151
NDEP	(775) 687-4670
Regional EPA:	(415) 744-1500
EPA Emergency Response Team:	(908) 321-6660
National Response Center:	(800) 424-8802
Center for Disease Control:	(404) 488-4100
Chemtrec:	(800) 424-9555

10.5 Nearest Medical Assistance

The nearest medical facility is the Northeastern Nevada Regional Hospital. The facility is located at 2001 Errecart Boulevard, Elko, Nevada. A map of the route to this facility which can provide emergency care for individuals who may experience an injury or exposure on site is included in Appendix C of this HASP. The route to the facility should be verified by the HSO prior to sampling activities, and should be familiar to all site personnel.

10.6 Emergency Medical Treatment Procedures

Any person who becomes ill or injured in the work area must be decontaminated to the maximum extent possible. If the injury or illness is minor, full decontamination should be completed and first aid administered prior to transport. If the patient's condition is serious, at least partial decontamination should be completed (i.e., complete disrobing of the victim and redressing in clean coveralls or wrapping in a blanket.) First aid should be administered while awaiting an ambulance or paramedics. All injuries and illnesses must immediately be reported to the project manager.

10.7 Fire or Explosion

In the event of a fire or explosion, the local fire department should be summoned immediately. Upon their arrival, the project manager or designated alternate will advise the fire commander of the location, nature, and identification of the hazardous materials on site. If it is safe to do so, site personnel may:

- Use fire-fighting equipment available on site to control or extinguish the fire; and
- Remove or isolate flammable or other hazardous materials which may sustain a fire.

10.8 Emergency Equipment/Facilities

All emergency equipment will be located in the command post and/or support zone and shall include:

- First aid kit;
- Fire extinguisher;
- Mobile telephone;
- Eye wash station.

11. HAZARD COMMUNICATION

In order to comply with 29 CFR 1910.1200, Hazard Communication, the following written Hazard Communication Program has been established. All employees will be briefed on this program and have a written copy for review.

11.1 Container Labeling

All containers received on site will be inspected to ensure the following:

- All containers will be clearly labeled as to the contents;
- The appropriate hazard warnings will be noted; and
- The name and address of the manufacturer will be listed.

All secondary containers will be labeled with either an extra copy of the original manufacturer’s label or with generic labels which have a block for identify and blocks for the hazard warning.

11.2 Employee Training and Information

Prior to starting work, each employee will attend a health and safety orientation and will receive information and training on the following:

- An overview of the requirements contained in the Hazard Communication Standard, 29 CFR 1910.1200;
- Chemicals present in their workplace operations;
- Location and availability of a written hazard program;
- Physical and health effects of the hazardous chemicals;
- Methods and observation techniques used to determine the presence or release of hazardous chemicals;
- How to lessen or prevent exposure to these hazardous chemicals through usage of control/work practices and personal protective equipment;
- Emergency procedures to follow if they are exposed to these chemicals;
- How to read labels and review MSDSs to obtain appropriate hazard information;
- Specialized hot work and tank processing techniques.

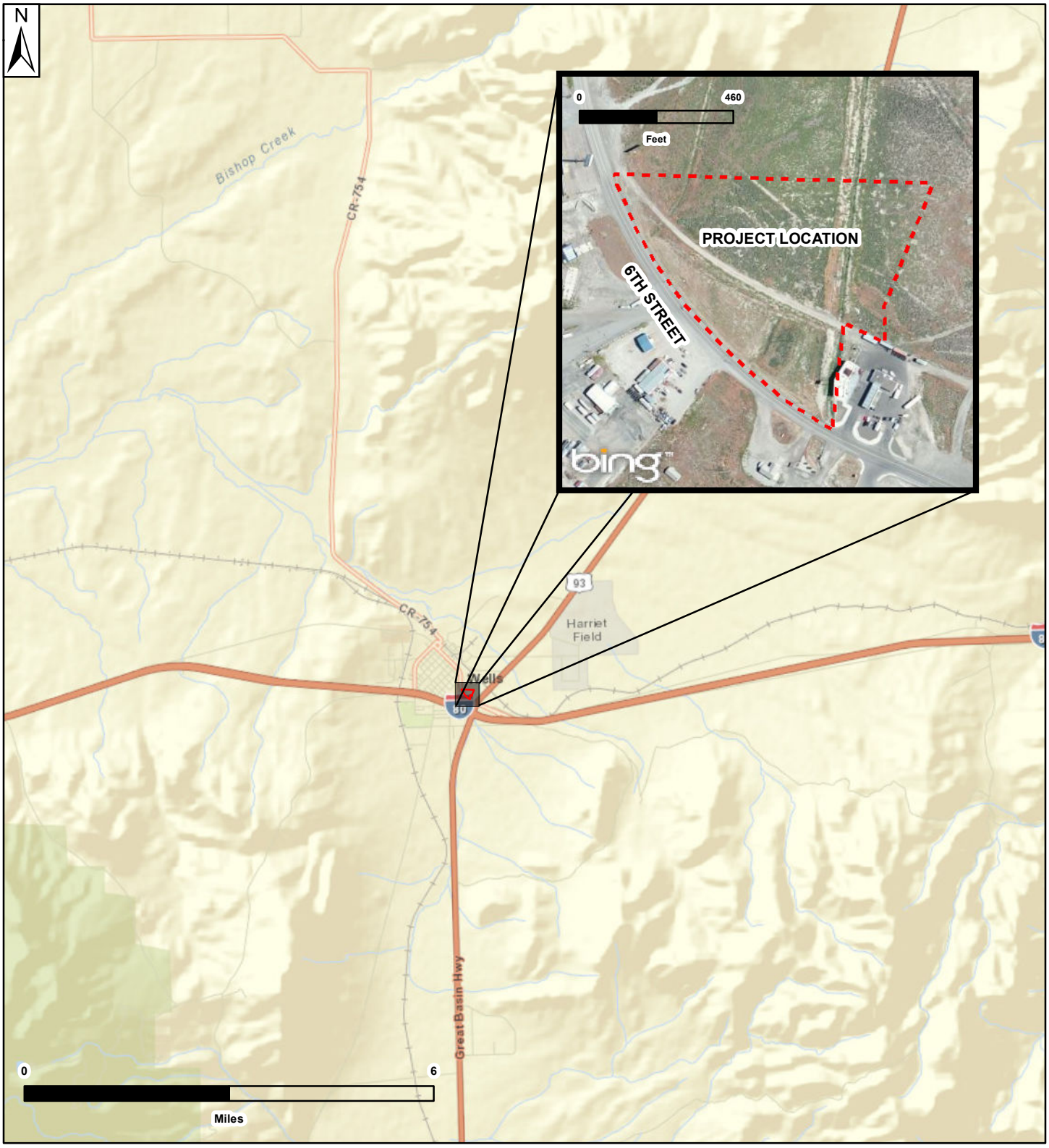


FIGURE 1

TITLE:

PROJECT LOCATION MAP

-SHOWING-

APN: 002-770-005

WELLS, NEVADA

JOB NO.: **LVBRN017**

DATE: **1/24/2013**

McGinley & Associates
Environmental Engineering and Science
RENO | LAS VEGAS | www.mcgin.com

FILE: **130124_LVBRN017_figure1_msp_85x11_portrait**

COORDINATE SYSTEM: **NAD 1983 UTM Zone 11N US Feet**

REF.					REVISION:
	DESIGNED	MSP	CHECKED	MSP	-
	DRAWN	MSP	APPROVED	BB	

APPENDIX A

Statement of Compliance

HASP

Statement of Compliance

I have read and understand the HASP for the site soils investigation at Elko County Assessor's Parcel Number (APN) 002-770-005, Nevada.

I agree to comply with the contents of the HASP and understand that not doing so may be reason for discharge from the site.

Signature: _____ Date: _____

Signature: _____ Date: _____

Signature: _____ Date: _____

Signature: _____ Date: _____

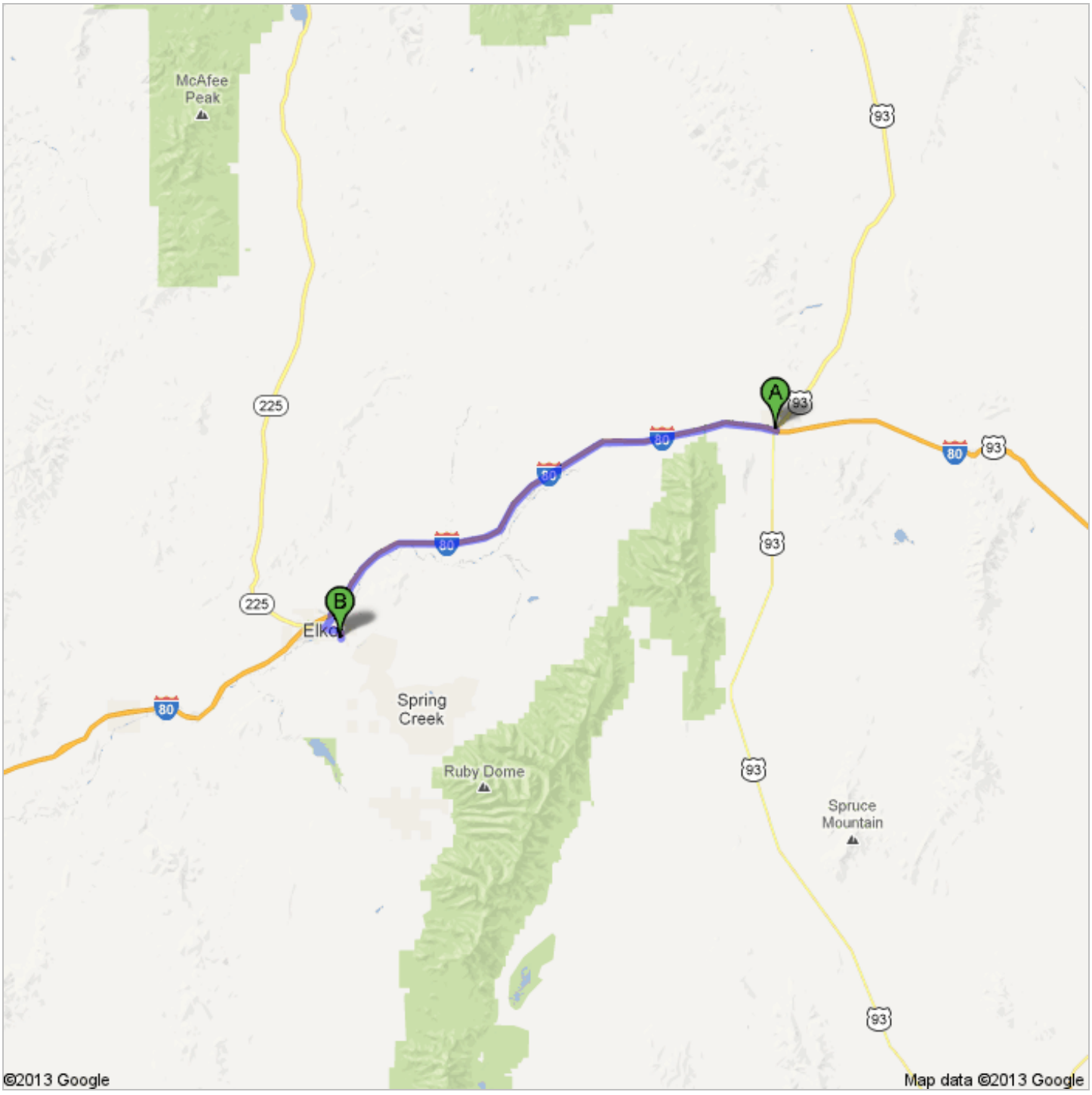
Signature: _____ Date: _____

Signature: _____ Date: _____

APPENDIX B

Route to Nearest Medial Assistance

Directions to 2001 Errecart Blvd, Elko, NV 89801
52.1 mi – about **54 mins**
Northeastern Nevada Regional Hospital



6th St/Interstate 80 Business

	1. Head **southeast** on **6th St/Interstate 80 Business** toward **US-93 N**	go 0.1 mi total 0.1 mi
93	2. Take the 1st right onto **US-93 S** About 46 secs	go 0.1 mi total 0.2 mi
80	3. Turn right to merge onto **I-80 W** About 42 mins	go 47.9 mi total 48.1 mi
	4. Take exit **303** toward **Elko E** About 55 secs	go 0.3 mi total 48.4 mi
	5. Turn left onto **E Jennings Way** About 50 secs	go 0.2 mi total 48.6 mi
	6. Turn right onto **Idaho St** About 3 mins	go 1.1 mi total 49.7 mi
	7. Turn left onto **12th St** About 3 mins	go 0.9 mi total 50.6 mi
227	8. Turn left onto **NV-227 E** About 2 mins	go 1.3 mi total 51.9 mi
	9. Turn right onto **Errecart Blvd** Destination will be on the right	go 0.2 mi total 52.1 mi

2001 Errecart Blvd, Elko, NV 89801