

Post-Closure Monitoring and Maintenance Plan

Winthrop Landfill



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POST-CLOSURE MONITORING AND MAINTENANCE PLAN

WINTHROP LANDFILL WINTHROP, MAINE

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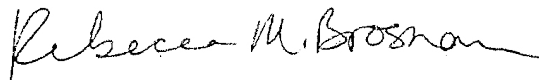
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LIST OF ACRONYMS AND ABBREVIATIONS

ACL	Alternate Concentration Limit
CD	Consent Decree
DQO	Data Quality Objective
EDD	Electronic Data Deliverable
ESD	Explanation of Significant Differences
GP	Gas Probe
GWETS	Groundwater Extraction and Treatment System
Landfill	Winthrop Landfill
MACTEC	MACTEC Engineering and Consulting, Inc.
MDL	Method Detection Limit
MEDEP	Maine Department of Environmental Protection
µg/L	micrograms per liter
mL	milliliters
MS/MSD	Matrix Spike/Matrix Spike Duplicate
MW	Monitoring Well
PARCC	Precision, Accuracy, Representativeness, Completeness and Comparability
PCL	Protective Concentration Limit
PCMP	Post-Closure Monitoring Plan
PCMMP	Post-Closure Monitoring and Maintenance Plan
POC	Points of Compliance
POE	Points of Exposure
ppmv	parts per million volume
QC	Quality Control
RAP	Remedial Action Plan
RL	Reporting Limit
RPD	Relative Percent Difference
Site	Winthrop Landfill
SW	Surface Water
USEPA	United States Environmental Protection Agency
UTC	United Technologies Corporation

VC	vinyl chloride
VCCP	Vinyl Chloride Contingency Plan
VES	Vapor Extraction System
VOC	Volatile Organic Compound
Wood	Wood Environment & Infrastructure Solutions, Inc.

1.0 INTRODUCTION

On behalf of United Technologies Corporation, Inc. (UTC), Wood Environment and Infrastructure Solutions, Inc. (Wood), (formerly Amec Foster Wheeler), has prepared this Post-Closure Monitoring and Maintenance Plan (PCMMP) for the Winthrop Landfill (the Landfill), located in the Town of Winthrop, Kennebec County, Maine (the Site) (**Figure 1.1**). This PCMMP Revision 7 updates and replaces the PCMMP Revision 6, (Wood, 2018) for the Site and incorporates revisions to achieve consistency with the Proposed Plan for the Site (United States Environmental Protection Agency ([USEPA], 2019).

1.1 Background

The Winthrop Landfill, located in the Town of Winthrop, Kennebec County, Maine, is an inactive municipal disposal site on the National Priorities List established by the Comprehensive Environmental Response, Compensation, and Liability Act of 1980. The Landfill consists of two contiguous parcels with surface areas of approximately 11 acres and 9.5 acres situated along the east side of Annabessacook Road (**Figure 1.1**). The landfill boundary and surrounding Site features are shown on **Figure 1.2**.

The Winthrop Landfill was operated as a former sand and gravel pit beginning in the early 1930s. The Landfill accepted both municipal and industrial wastes, and, until the mid-1970s, most of these wastes were burned periodically. Landfill operations, including disposal of hazardous substances, occurred in the northern portion of the Landfill from the early 1970s to the mid-1970s. Prior to that period, these wastes were also deposited on the western perimeter of the landfill. From the mid-1970s until 1982 the southern portion of the Site operated as a sanitary landfill. The wastes were covered with a silty sand. Beginning in 1982, the Town of Winthrop used the east central portion of the Landfill as a brush, building demolition, and stump dump until 1986 when these wastes were covered. By 1986, hazardous substances had been detected in groundwater northeast, east, and south of the Landfill and in residential drinking water wells located between the landfill and Annabessacook Lake.

A Consent Decree (CD) was issued March 23, 1986 by the United States District Court, District of Maine, between the USEPA and the MEDEP as Plaintiffs and the Inmont Corporation (now owned by BASF Corporation), Town of Winthrop, and Everett and Glenda Savage as Defendants. The CD contains a Remedial Action Work Plan (RAP) (E.C. Jordan 1986 and 1989) which defines the scope of investigation and remediation activities necessary to control contamination released from the Landfill. UTC, as the former owner of Inmont and on behalf of BASF Corporation, has taken primary responsibility for coordinating and fulfilling the requirements of the RAP. The RAP includes 10 elements that form a complete and comprehensive plan to protect public health and the environment from adverse effects of wastes and waste constituents deposited in the Landfill. The 10 elements are as follows:

Element

- II 1: Extension of Alternate Water Supply
- II 2: Fence and Landfill Use Control
- II 3: Groundwater Use Control

- II 4: Excavation Control
- II 5: Monitoring Program
- II 6: Landfill Cover System
- II 7: Engineering Studies
- II 8: Establishment of an Alternate Concentration Limit (ACL)
- II 9: Installation and Operation of a Groundwater Interceptor (Extraction) System (if ACL is exceeded)
- II 10: Installation and Operation of a Groundwater Treatment System (if ACL is exceeded)

Elements II 1 through II 4 and II 6 through II 10 have been completed. Element II 5 (Monitoring Program) has also been completed with regard to investigation and remedial implementation and is discussed in **Section 1.2**. The first Element (II 1) was completed by UTC in 1987. RAP Elements II 2, II 3, and II 4 were completed by the Town of Winthrop in the form of the Groundwater Protection ordinance and subsequently modified on April 1, 1991 as the Groundwater and Air Protection ordinance to control exposure to migrating landfill gas. Elements II 6 through II 10, as well as the fence installation of Element II 2, have been completed by UTC. In addition to these 10 elements, UTC voluntarily added a vapor extraction system (VES) that operated in the mid-1990s.

A Groundwater Extraction and Treatment System (GWETS) began operating in March 1995. Shortly after the GWETS was activated, the GWETS influent met clean up criteria (ACLs) for all target compounds with the exception of arsenic. The GWETS was not effective at reducing arsenic concentrations in groundwater. The GWETS was deactivated in November 2002 for a Rebound Evaluation. The Rebound Evaluation demonstrated that the GWETS was no longer an effective treatment technology and was not required for protection of public health and the environment.

In February 2007, the USEPA issued an Explanation of Significant Differences (ESD) to the Consent Decree which officially terminated the GWETS and shifted the focus of the remedial measures to controlling exposures to contaminants, primarily arsenic, at the Points of Exposure (POEs), or seeps (USEPA, 2007).

Element II 5 of the RAP, the monitoring program, is discussed below in **Section 1.2**.

1.2 History of Monitoring Program

Element II 5 of the RAP, the monitoring program, has been in effect since November 1985, before the Consent Decree was issued. The Monitoring Plan was provided as Attachment III to the RAP. Now that the remediation program at the Winthrop Landfill has progressed from the investigation and remedial implementation phase to the post-GWETS operations and maintenance, the monitoring activities conducted under Element II 5 are now focused on compliance monitoring at the points of compliance (POCs) and assessment of exposure conditions in the flow paths and at the POEs. The monitoring program has evolved as the remedial measures have changed at the Site.

The RAP provided two mechanisms for revising the Monitoring Plan. First, Section II 5B of the RAP provides for periodic Monitoring Plan revisions based upon the mutual consent of USEPA, MEDEP, and Inmont (UTC). Second, subsequent to setting the ACLs, Section II 5C also required USEPA and MEDEP

to conduct a review of the adequacy of the monitoring program at least every five years, or more often if requested by UTC, and to consider proposals from UTC to decrease the frequency of sampling rounds (RAP II 5Ci) and the number of sampling locations (RAP II 5Cii).

In February of 2007, the USEPA issued an ESD for the Site that places a focus on the minimization of exposure risks due to the ongoing discharge of arsenic from groundwater at the POEs (USEPA, 2007). Revision 5 of the PCMMP (MACTEC, 2010), approved in 2010, provided an updated environmental monitoring program and addressed the monitoring requirements outlined in the February 2007 ESD. It also reflected the decommissioning of the GWETS and VES systems and included:

- compliance groundwater monitoring at specified POCs identified in the Consent Decree
- groundwater monitoring in the groundwater flow paths for evaluation of ACL compliance and exposure conditions
- surface water and sediment monitoring at the POEs for evaluation of Protective Concentration Limit (PCL) compliance and exposure conditions
- soil gas monitoring for evaluation of explosive hazards and human exposure
- systematic monitoring and maintenance of the landfill cover system, fence, and compliance with the groundwater and air protection ordinance
- PCMMP reporting format and schedule.

PCMMP Rev6 included reductions to the sampling frequency and monitoring network in accordance with the December 6, 2016 Basis of Change letter (Amec Foster Wheeler, 2016) and subsequent discussions and agreement between UTC, MEDEP, and USEPA, as discussed below.

1.2.1 PCMMP Revision 6 Updates to Monitoring Program

Recommendations to revise the monitoring program were proposed by Amec Foster Wheeler in the 2016 Basis of Change letter (Amec Foster Wheeler, 2016), which provided justification for proposed updates to the monitoring plan for the Site. In accordance with discussion, comments, and agreements with MEDEP and USEPA in 2017, the PCMMP Revision 6 was prepared by Wood and approved by USEPA on December 6, 2017 and approved by MEDEP on September 26, 2018. A summary of revisions to the monitoring plan as implemented in PCMMP Revision 6 is presented below.

Groundwater, Surface Water, and Sediment Monitoring:

- Monitoring has been reduced to a 2.5-year sampling frequency, with the first monitoring event at this frequency conducted in fall 2018, and the next scheduled for spring 2021.
- The monitoring well (MW) locations along each flow path, at Annabessacook Lake and in Hoyt Brook have been retained for sampling on a 2.5 year frequency to provide data for tracking conditions (primarily arsenic and field parameters) at these important POC/flow path and POE monitoring locations. The first 2.5-year monitoring event was conducted in fall 2018.
- Five-year monitoring frequency will be implemented for locations in Sphagnum Bog and Cattail Marsh where fencing restricts public access. Five-year events will also include the

locations sampled at a 2.5-year frequency. The next five-year event is planned for spring 2021.

- Select monitoring locations have been discontinued: MW-8C, MW-9B, MW-103R, MW-108, SED-108, SED-108B, SED-108D, SP-108, SED-114N, SED-120, SED-121, SP-114N, SW-120, and SW-121. RAP Table 2 parameters have been discontinued.
- Remedial Action Plan Table 1 (RAP Table 1) volatile organic compound (VOC) sampling will be completed at Point of Compliance wells every five years.
- RAP Table 1 VOC sampling will be completed at two monitoring wells in the southern flow path every 2.5 years.
- Vapor intrusion contingency monitoring and evaluation will be completed at the southern flow path monitoring well where currently performed (MW-5A) every five years.
- Field parameters will be monitored at all locations and frequencies where samples are being collected for laboratory analyses.
- A synoptic round of water levels will be completed every 2.5 years (twice per five years, one spring event and one fall event).

Landfill Monitoring:

- Gas probe monitoring will be discontinued at all locations except at GP-11, which will be monitored once every five years.
- Landfill mowing will continue at a frequency of two times per year (no change from current PCMP).
- Inspection of Landfill Cap will be performed once per year after the first mowing.
- Hoyt Brook Seep cover inspections will be performed at least once per year.

Annual Desktop Review: An annual desk top review and Site area reconnaissance will be performed to confirm that there have been no land use changes that could impact groundwater flow or exposure conditions at the Site. The annual review will include:

- Ordinance Compliance – check in with Winthrop Code Enforcement Officer to confirm no new development in Site Area (no change).
- Field Verification of Site area conditions – windshield reconnaissance of Mallard Lane, Hubbard Lane, Annabessacook Road adjacent to Landfill to verify ordinance compliance (completed during annual Landfill Cap inspection).

1.2.2 Current Updates to Monitoring Program

The revisions listed in **Section 1.2.1** will remain in effect in this current version of the PCMMP (Revision 7). In addition, the following changes will be made to the monitoring program to accord with the USEPA's Proposed Plan for the Winthrop Landfill.

- Two additional sediment monitoring locations (SED-401 and SED-402) will be included in 2.5-year monitoring events beginning in spring 2021 (these locations were added after the

fall 2018 event). These locations are directly upstream and downstream of the Hoyt Brook Cover, as shown on **Figure 2.1**.

1.3 Objectives of Monitoring Program

The objectives of the revised PCMMP are to collect appropriate and sufficient data to:

1. Support USEPA's preparation of the 5-year reviews and communication of the current understanding of Site conditions to the public.
2. Be protective of human health and the environment.
3. Periodically confirm the concentrations of parameters that have been chosen for tracking.
4. Manage risks at the POEs and track compliance at the points of compliance POCs.

1.4 Organization

This PCMMP is organized as follows:

- Section 1.0 – Introduction
- Section 2.0 – Environmental Monitoring Plan
- Section 3.0 – Analytical Program
- Section 4.0 – Reporting Format and Schedule
- Section 5.0 – References

1.5 Monitoring Program Contacts

A summary of Wood, UTC, MEDEP, and USEPA project personnel, responsibilities, and contact information for the PCMMP is provided in Table 1.1.

Table 1.1: Personnel Responsibilities and Qualifications

Name	Title	Responsibilities	Contact
Nathan Hagelin, C.G.	Wood Project Manager	Meeting Project Schedule and Technical Requirements	nathan.hagelin@woodplc.com 207-828-3508
John Rand, C.G.	Wood Technical Lead	Planning/Executing PCMMP	john.rand@woodplc.com 207-828-2655
Madison Dinsmore	Wood Project Chemist	Chemistry Support	madison.dinsmore@woodplc.com 207-828-2661
Rebecca Brosnan	Wood Quality Assurance Lead	Quality Assurance/Quality Control	rebecca.brosnan@woodplc.com 207-828-2635
Julie Ricardi	Wood Senior Chemist	Senior Chemist / Data Validation Review	julie.ricardi@woodplc.com 207-828-3608
Michael Lounsbury	Wood Field Operations Lead	Implementation of field sampling and maintenance activities	michael.lounsbury@woodplc.com 207-828-3537
Binks Colby-George	Wood Data Manager	Data Management Coordination	binks.colby-george@woodplc.com 207-828-3650



Name	Title	Responsibilities	Contact
William Elwell	UTC Remediation Group	UTC Project Manager	william.elwell@utc.com 309-842-9049
Rebecca Hewett	MEDEP	MEDEP Project Manager	rebecca.l.hewett@maine.gov 207-287-8554
Almerinda Silva	USEPA	USEPA Project Manager	silva.almerinda@epa.gov 617-918-1246

2.0 ENVIRONMENTAL MONITORING PLAN

The environmental monitoring program described below will provide the information needed to evaluate:

- groundwater conditions at the POCs and flow path configuration wells
- surface water and sediment conditions at the groundwater discharge zone POEs
- potential for vapor intrusion in the vicinity of the landfill.

The analytical program is summarized in the following sections. ACLs and PCLs are provided in **Appendix A**, and monitoring activities will be conducted in accordance with the Sampling and Field Documentation Procedures provided in **Appendix B**. Collected samples will be transported under standard chain-of-custody procedures to a Maine-certified, UTC-approved laboratory, for analysis of the parameters outlined in **Table 2.1**.

2.1 Sampling Locations

Sampling locations are presented in **Table 2.1** and shown on **Figure 2.1**. Sampling locations are grouped into groundwater POCs, groundwater flow paths, and surface water/seep/sediment POEs. Additionally, one landfill location is included for landfill gas monitoring.

2.1.1 Surface Water, Seep, and Sediment Points of Exposure

Surface water, seep, and sediment monitoring locations will be sampled to assess conditions at the groundwater discharge zone POEs and evaluate PCL exceedances, if any exist. The POE areas include Annabessacook Lake, Hoyt Brook, Cattail Marsh, and Seep East. The POE monitoring locations for each area are shown in **Figure 2.1** and listed in **Table 2.1**.

Surface water and sediment sampling procedures are provided in **Appendix B**. Surface water samples will be collected near the surface water /sediment interface, and sediment samples will be collected within the top 0.5 foot of sediment. This procedure is designed to collect worst-case samples to most conservatively monitor the exposure conditions. It is likely that sediment will be entrained and collected as part of the surface water sample, and therefore, an unfiltered and a filtered sample will be collected for metals analyses to quantify the amount of constituents contributed by suspended solids.

Annabessacook Lake

The southern groundwater flow path discharges into Annabessacook Lake to an area that extends from the shoreline approximately 100 feet and is approximately 50 feet wide. The location and extent of the discharge area varies with lake level and groundwater elevation.

Wood has determined from visual observations and elevation survey that the seep is exposed to the atmosphere when the lake elevation is below approximately 167 feet above mean sea level.

As part of the environmental monitoring at this POE, the Annabessacook Lake elevation will be monitored through the Jug Stream outlet measurement point maintained by the Cobbossee Water District. Surface water sampling will be conducted at seep location SP-8 once every 2.5 years, alternating spring and fall. Sediment sampling from locations SED-108A and SED-8 is planned for the same schedule. A map of the area identifying sediment sample collection points will be included with

the reported results. Annual visual monitoring will also occur at SP/SED-8 as described below in **Section 4.6**.

Hoyt Brook

The northern groundwater flow path discharges into Hoyt Brook. In 2015, Amec Foster Wheeler constructed a Pilot Study on a portion of Hoyt Brook to address USEPA and MEDEP concerns about human contact with arsenic-impacted sediment and surface water in the Hoyt Brook seep area. Hoyt Brook monitoring has taken place as part of the Pilot Study to determine whether the Pilot Study construction has eliminated direct human contact with unacceptable concentrations of arsenic in the seep area without causing new contamination or seepage conditions, unacceptable erosion, or degradation of surface water.

As part of this PCMMP, surface water and sediment Hoyt Brook samples will be collected from the following locations (from upstream to downstream): SED-119, SW-119, SED-401, SED-402, SED-11, and SW-11, SW-15, at a frequency of once per 2.5 years.

Cattail Marsh

Cattail Marsh is located above a unit of clay-silt soils at the north end of the Landfill. Beneath Cattail Marsh, groundwater from the northern flow path migrates through the sand and gravel unit. Groundwater discharge into Cattail Marsh occurs from the perched leachate condition at the north end of the Landfill and from the northern flow path seeping upward through the 20-to-40-foot-thick clay-silt layer. There have been no PCL exceedances in this area since 2015. Sampling frequency has been reduced to once every five years. Monitoring locations at Cattail Marsh are sediment at location SED-SPMRSH and surface water at locations SP-MRSH, SP-MRSHA, and SW-18.

Seep East

The groundwater discharge along the east fence line is referred to as Seep East. Seep East drains to and is contiguous with Sphagnum Bog. There have been no PCL exceedances in this area since 2015. Sampling frequency has been reduced to once every five years. Seep East monitoring locations include sediment at SED-304 and surface water at SP-304, SP-304A, SW-3, SW-305, and SW-4.

2.1.2 Groundwater Monitoring Locations

To assess groundwater conditions at the landfill, POC wells and monitoring wells located in the groundwater flow paths are included in the environmental monitoring program. Monitoring well construction details are provided in **Table 2.2**. **Appendix B** includes monitoring well construction logs.

Flow Path Configuration

Flow path configuration at the Landfill is well-defined by piezometric conditions and landfill constituent concentrations. Flow path configuration monitoring will consist of water level measurements and sampling and analysis. Water levels will be measured every 2.5 years at the locations listed in **Table 2.1** and shown in **Figure 2.1**.

Southern Flow Path Sampling. The southern flow path is monitored by MW-212A, MW-212B, MW-212C, MW-8A, and MW-8B, located in the southern flow path downgradient from the POC. These wells lie along the axis of the southern flow path and together with MW-5A, the POC well, track the

migration of constituents in groundwater from the perimeter of the Landfill to the lake. Southern Flow Path monitoring well locations are shown in **Figure 2.1**.

Northern Flow Path Sampling. The northern flow path is monitored by MW-15A, MW-15B, MW-208A, MW-208B, MW-208C, MW-210A, MW-210B, MW-210C located in the northern flow path downgradient from the POC. These wells lie along the axis of the northern flow path and together with MW-10B and MW-10C, the POC wells, track the migration of constituents in groundwater from beneath the Landfill to the Hoyt Brook POE. Northern Flow Path monitoring well locations are shown in **Figure 2.1**.

Points of Compliance

The POC wells (MW-5A, MW-10B, MW-10C) will be sampled every 5 years for the RAP **Table 1** VOCs and every 2.5 years for total metals (arsenic, nickel, and zinc). MW-5A monitors groundwater for the southern flow path, MW-10B for the northern flow path, and MW-10C for the perched water at the northern end of the landfill. Groundwater results from the POC wells will be used to evaluate compliance with ACLs.

Vapor Intrusion Contingency Monitoring

The Vinyl Chloride Contingency Plan (VCCP) (MACTEC, 2003) was developed during the Rebound Evaluation in response to detections of vinyl chloride at MW-5A exceeding the ACL of 2 micrograms per liter ($\mu\text{g/L}$). The monitoring protocols established in the VCCP are incorporated herein as described in this subsection. These protocols are specific to vinyl chloride in the southern flow path. Should any other ACL exceedance occur at a POC, a similar monitoring protocol will be developed in consultation with USEPA and MEDEP.

The analytical method used in the past for the vinyl chloride vapor contingency monitoring program was USEPA method 524.2. This drinking water method was used because it resulted in a lower detection limit and a higher degree of confidence in the analytical precision and accuracy at low levels. However, the current detection limit for vinyl chloride analyzed by Method 8260 ($1 \mu\text{g/L}$) is adequate to meet the requirements of the contingency monitoring program. For simplicity and consistency with the rest of the VOC monitoring program, Method 8260 will be used for vinyl chloride vapor contingency monitoring as well.

The baseline monitoring program is the collection of samples once every five years at MW-5A for vinyl chloride analysis by Method 8260. Based upon the results of the baseline program the following protocols will be followed:

- Exceedance of $4 \mu\text{g/L}$ at MW-5A: if vinyl chloride (VC) $> 4.0 \mu\text{g/L}$, initiate monthly monitoring for VC at MW-5A and begin bi-monthly (every second month) monitoring of VC at MW-8C and initiate monthly monitoring of the existing gas probes GP-2B, C, D & E for each month that the groundwater concentration at MW-5A exceeds $4.0 \mu\text{g/L}$. Air monitoring will be conducted with SUMMA® canisters and analyzed by USEPA Method TO-15.
- Exceedance of VC Target Soil Gas Concentrations for Indoor air at GP-2 (0.110 parts per million volume (ppmv) for GP-2C, D, and E and 0.0110 ppmv at GP-2B): if VC > 0.0110

ppmv, initiate monthly monitoring of the skirt/crawl space under the existing mobile home located between MW-5A and MW-8B.

- Exceedance of the VC Target Indoor Air Concentration (0.0011 ppmv) in the crawl space beneath the residence between MW-5A and MW-8: if VC > 0.0011 ppmv, initiate remedial action such as soil vapor extraction to cut off the potential exposure route.

Should the contingency program described above be activated, results will be reported monthly to the agencies and summarized in the post-closure monitoring reports.

2.2 Sampling Frequency and Schedule

The frequency of water level measurements and groundwater, surface water, seep, and sediment sampling will be once per 2.5 years, alternating spring and fall. Certain monitoring locations and parameters will be sampled only once every 5 years. **Table 2.1** shows sampling frequency for the various monitoring locations and parameters. The Post Closure Monitoring and Maintenance schedule is presented in **Table 2.3**.

2.3 Target Analytes, Contaminants of Concern

The target analytes, PCLs, and ACLs are listed in **Appendix A**. VOC analysis by Method 8260C will be performed for POC and select southern flow path groundwater monitoring wells. Vinyl chloride concentrations at one POC well (MW-5A) will be evaluated for vapor intrusion potential, as discussed in **Section 2.1.2**. Groundwater monitoring wells in the northern and southern flow paths will be sampled for total arsenic. POE surface water, seep, and sediment sampling locations will be sampled for total/dissolved metals (arsenic, zinc, and/or nickel) as specified in **Table 2.1**. Landfill gas monitoring will be conducted with a gas probe for percent carbon dioxide, percent methane, percent oxygen, and pressure.

2.4 Alternate Concentration Limits and Protective Concentration Limits

ACLs for groundwater and PCLs for surface water, seeps, and sediment were established as part of the RAP process. These are presented along with Method Detection Limits and Laboratory Reporting Limits for each of the monitored parameters in **Appendix A**.

2.5 Monitoring Locations Inspection and Maintenance

Routine inspection of the groundwater, surface water, seep, and sediment monitoring location network will be performed during each monitoring event. Sampling locations will be inspected to verify each is clearly marked, capped, locked, sealed, and grouted. Maintenance and repairs will be conducted as needed to maintain proper function and integrity. For example, excessive accumulation of sediment in wells will be eliminated by redeveloping the well. Damaged wells, including damaged protective aprons and protective casings, will be repaired on an as-needed basis depending on severity and importance to the monitoring program. Surface water, seep, and sediment sampling locations will be evaluated for integrity during each sampling event.

3.0 ANALYTICAL PROGRAM

This section describes the off-Site laboratory analytical program and methods used to analyze aqueous samples collected during monitoring events. The majority of off-Site analytical data will be generated using USEPA analytical procedures. Analyses will be completed using current USEPA SW-846 methods (USEPA, 2007). Sample analyses will be completed by a UTC-approved laboratory that is certified in Maine. A summary of analytical methods that will be used for the PCMMP is provided in **Table 3.1**.

Table 3.1: Analytical Methods

Parameter	Analytical Method	Groundwater	Surface Water	Sediment
VOCs	USEPA 8260B/8270C	X		
Metals	USEPA 6010C/6020A	X	X	X

Notes:

VOCs – Volatile Organic Compounds

Metals (total and dissolved in accordance with Table 1.1): As, Ni, Zn

Laboratory location and contact information is provided below:

Chris McCord, Project Manager
Pace Analytical Nation
12065 Lebanon Road
Mt. Juliet, TN 37122
615.773.3281
cmccord@pacenational.com

3.1 Target Analytes and Detection Limits

A summary of target analytes, ACLs, PCLs, reporting limits, and method detection limits is provided in **Appendix A**.

3.2 Sample Container, Preservation, and Holding Times

Collected samples will be maintained in ice-filled coolers. Following each sampling event, Wood will coordinate transport of the sample coolers (under standard chain-of-custody procedures) to the laboratory. Sample container and preservation specifications and holding time requirements are presented in **Table 3.2**.

Table 3.2: Sample Container, Preservation, and Holding Time Criteria

MEDIUM	ANALYSIS	ANALYTICAL METHOD	SAMPLE CONTAINER	PRESERVATION	HOLD TIME	DATA EVALUATION
Groundwater	VOCs	USEPA 8260CB	3 – 40 mL VOC vial	HCL pH < 2 Ice to 4°C	14 days	See Note 1
Groundwater	SVOCs	USEPA 8270C	2 – 100 mL Amber Glass	Ice to 4°C	7 Days	See Note 1
Groundwater/ Surface water	Metals	USEPA 6020A	1 – 250 mL Plastic	HNO ₃ pH <2, Ice to 4°C	6 months	See Note 2
Sediment	Metals	USEPA 6010C	4 oz. glass jar	Ice to 4°C	6 months	See Note 2
Sediment	Total Solids	See above. Part of sediment metals analysis.				

Notes:

VOCs = Volatile Organic Compounds

HCL = Hydrochloric Acid

HNO₃ = Nitric Acid

ML = milliliters

1 = Data evaluation will include review of Sample Holding Times, Blank Contamination, Surrogate Recoveries, Matrix Spike Recoveries, Laboratory Control Sample Recoveries and Field Duplicate Precision.

2 = Data evaluation will include review of Sample Holding Times, Blank Contamination, Matrix Spike Recoveries, Laboratory Control Sample Recoveries, Field Duplicate Precision and Laboratory Duplicate Precision.

3.3 Analytical Quality Control Limits

Project quality control (QC) limits are established for each matrix and method. These QC limits will be used during data validation to assess the precision and accuracy of the laboratory results. A summary of QC limits is provided in **Table 3.3**.

Table 3.3: QC Limits

PARAMETER	QC TEST	FREQUENCY	ANALYTE	WATER (%R)	WATER (RPD)	SEDIMENT (%R)	SEDIMENT (RPD)
VOCs/SVOCs	Surrogate	5%	All Surrogates	Lab Limits	NA	Lab Limits	NA
	MS/MSD		All Target Compounds	Lab Limits	Lab Limits	Lab Limits	Lab Limits



Table 3.3: QC Limits

PARAMETER	QC TEST	FREQUENCY	ANALYTE	WATER (%R)	WATER (RPD)	SEDIMENT (%R)	SEDIMENT (RPD)
	LCS/LCSD		All Target Compounds	Lab Limits	Lab Limits	Lab Limits	Lab Limits
	Field Duplicates	5%	All Target Compounds	NA	30	NA	50
Metals	MS/MSD	5%	All Metals	Lab Limits	Lab Limits	Lab Limits	Lab Limits
	LCS/LCSD		All Metals	Lab Limits	Lab Limits	Lab Limits	Lab Limits
	Field Duplicates	5%	All Metals	NA	30	NA	50
	Laboratory Duplicates		All Metals	NA	Lab Limits	NA	Lab Limits

Notes:

QC = Quality Control

%R = Percent Recovery

RPD = Relative Percent Difference

LCS/LCSD = Laboratory Control Sample/Laboratory Control Sample Duplicate

VOCs = Volatile Organic Compounds

MS/MSD = Matrix Spike/ Matrix Spike Duplicate

Metals = As, Ni, Zn

NA = Not Applicable

3.4 Quantitation Limits

As shown in **Appendix A**, reporting limits (RLs) from the UTC-approved laboratory are expected to be less than the PCLs and ACLs for the parameters monitored at the Site. For constituents with reporting limits exceeding PCLs or ACLs, laboratory method detection limits (MDLs) may be used. The identity of the compound and value of the concentration will not be known with the same certainty and confidence as values above the reporting limit. In some instances, the RL and/or MDL are greater than the PCL or ACL. In cases where both the RL and MDL are greater than the PCL or ACL, PCL/ACL exceedances below the MDL will not be detected, and exceedances between the MDL and RL will be estimated.

As new analytical methods are developed and become commercially available for application to groundwater and surface water conditions at the Site, they will be evaluated. If deemed appropriate for use, a written correspondence will be submitted to the MEDEP requesting that the new analytical protocol be approved and formally adopted as part of the PCMMP.

3.5 Analytical Data Quality Objectives

Analytical data quality objectives (DQOs) include measuring the precision, accuracy, representativeness, completeness, and comparability (PARCC) parameters of the data set. PARCC parameters are indicators of the data quality and are described further in **Appendix C-1** (Data Management, Validation and Qualification Procedures). Laboratory standard operating procedures for the relevant analytical methods are provided in **Appendix C-2**.

3.6 Data Review and Validation

Wood will perform a Chemist Review on data generated for this Winthrop Landfill PCMMP to assess conformance to the DQOs. The project quality assurance officer will review field instrument calibration sheets, field data sheets, and log book notes for location-specific sampling conditions, completeness and accuracy.

Laboratory analytical data will be reviewed to establish that the data were generated by methods meeting the QC requirements for the methods. This is established during data validation which is a review of the quality control documentation provided by the laboratory. The validation, QC requirements, and data management requirements are described in **Appendix C**.

After the sampling and analytical data are established to be acceptable according to the validation guidelines, the data will be reviewed for their Site, environmental, and historical context. Data passing this review will be used for evaluating conditions at the Site.

4.0 LANDFILL MONITORING AND INSPECTION

Landfill monitoring will be conducted on a periodic and systematic basis to assess the condition of the Landfill cover system and its related structures. **Figure 4.1** shows features of the Landfill. Maintenance will be performed routinely and repairs are made on an as-needed basis to preserve the structural integrity and performance of the Landfill cover system.

4.1 Landfill Site

Visual Site monitoring of the Landfill and surrounding area will be performed once per year in mid-summer, after the first of two mowing events per year. Visual Site monitoring is performed to identify:

- evidence of erosion
- cap differential settlement resulting in soil cracking or ponded water
- seeps
- condition of the fence, gates, locks, and signs
- evidence of animal burrows into the cap
- condition of vegetative cover, presence of vegetation stress
- condition of gas probes
- condition of groundwater monitoring wells
- condition of drainage structures
- condition of roads
- new construction in area (e.g., residential)
- mining activity
- use of groundwater in Groundwater Protection Area

The Site observations will be performed by an engineer or geologist experienced in landfill Site monitoring and familiar with the RAP, landfill design, and corrective measures, maintenance and repair activities at the Site. Observations are recorded and photographs taken as a supplement if deemed appropriate by the observer. **Sections 4.2 through 4.4** describe the landfill monitoring in detail.

4.2 Landfill Gas Monitoring

An off-Site (outside the perimeter fence) Landfill Gas Monitoring Program was previously conducted beginning in 1996. To date, there has been no evidence of methane at levels that pose a threat to human health, cause an environmental effect (e.g., stressed vegetation), or pose an explosion danger to existing structures caused by the migration of landfill gas.

The gas probe (GP) analytical program has been discontinued with the exception of one on-Site landfill reference location, GP-11. This location will be monitored every 5 years using a hand-held detector. Measurements will be taken for gas pressure (measured to the nearest 0.1 inch of water) and percent methane, carbon dioxide, and oxygen (measured to the nearest 0.1 percent).

In the unlikely event the percent concentration of methane at GP-11 increases from one monitoring event to the next by 20 percent or more, the measurement will be checked one month later and if confirmed to be elevated relative to historic, an assessment of potential off-Site migration and explosion hazard will be considered by UTC in consultation with MEDEP.

4.3 Slope and Settlement Monitoring Program

To date, the settlement monitoring program implemented at the landfill indicates that no significant movement or settlement has been observed and that there is no indication of imminent slope or hydraulic barrier failure. Visual slope and settlement monitoring will be conducted annually to identify unusual changes in the slope morphology or signs of erosion or cracks in the landfill cover system. A settlement platform survey will be conducted every five years to confirm results are consistent with historical data. The next settlement platform survey will be conducted in Fall 2023.

4.4 Hoyt Brook Seep Cover Inspection

The seep cover at Hoyt Brook will be inspected once per year to identify evidence of erosion, settlement, seeps, animal burrows, or other impacts to the seep cover.

4.5 Maintenance Activities

Items identified during the Site inspections that require routine corrective maintenance will be repaired. Reports of all maintenance activity will be reported with the annual Landfill Inspection and Desktop Review Report.

4.5.1 Cover Maintenance

The vegetative cover will be mowed twice per year to prevent the growth of deep rooted, woody species and encourage the development of a good grass growth to control erosion. Reseeding of areas which are noted during inspection to have poor vegetative growth will be performed and maintained, in accordance with Technical Specification 02930 Seeding of the Landfill Cover System (E.C. Jordan Co., 1987).

Animal burrows into the cover identified during Site observations will be repaired as they are discovered during inspections. Shallow burrows into the vegetative soil, common borrow, or drainage (sand) layers will be filled and seeded in accordance with Technical Specifications 02221 and 02930, respectively. Deeper burrows, which penetrate the hydraulic barrier layer, require filling with hydrated bentonite chips placed from the bottom of the burrow to the top surface of the hydraulic barrier layer. The overlying layers are then reconstructed and revegetated in accordance with the Technical Specifications. Elimination of animal problems will be preferentially accomplished by trapping and relocating. If such attempts fail, the animals will be exterminated.

4.5.2 Erosion Control Maintenance

Erosion of the cover system will be repaired as needed in a manner which provides a long-term solution to such damage. Reconstruction of each cover component will be in accordance with Technical Specification 02221 - Excavation, Backfill, and Compaction and Section 6.0 of the Landfill Cover System Design Report (E.C. Jordan Co., 1986). The disturbed area shall be seeded in accordance with Technical Specification 02930 Seeding.

Erosion of the runoff control structures (drainage ditches) will be repaired as needed in a manner and schedule similar to that described above. Replacement of riprap will be in accordance with Technical Specification 02274. The final grades of all repaired areas will conform to the grades and slopes of the surrounding areas and comply with the limits of design grades and slopes.

4.5.3 Settlement and Subsidence Control Maintenance

The grades and slopes of the Landfill are expected to be sufficient to provide positive drainage slopes. If excessive settlement or damage is identified during Site inspections, repair of the cap will be implemented as necessary to avoid erosion and excessive differential settlement so that the cover system layers remain continuous, that a positive slope be maintained, and that ponding does not occur. The corrective action will be in conformance with maintaining positive slope drainage and Technical Specification 02221 - Excavation, Backfill, and Compaction. Following restoration of the hydraulic barrier grade, the overlying layers shall be reconstructed in accordance with the applicable technical specifications and Section 6.0 of the Landfill Cover System Design Report, (E.C. Jordan Co., 1986). Minor subsidence will be addressed by filling the low areas of the vegetative layer with more compacted silty material to eliminate the depression and reseeded.

4.5.4 Fence Maintenance

The landfill fence will be inspected annually for damage and structural integrity. Routine fence maintenance includes lubricating gate hinges and replacement of worn locks. Damage to the chain-link fence identified in the inspection program will be repaired. Minor erosion rills or animal burrows beneath the fence will be identified and repaired as necessary. Such repairs should occur within 60 days of observation provided weather or access does not restrict repair activities and that the scope of repairs can reasonably be accomplished within the 60-day period.

4.5.5 Roadway Maintenance

Periodic maintenance of the portion of Hubbard Lane that traverses the cap and underlying waste may be necessary. Exposure of the geotextile installed to indicate excessive loss of aggregate will require the addition of surface gravel as specified in Landfill Cap and Fence Technical Specification 02230.2.01(A)(1) - Aggregate for Base.

4.5.6 Sign Maintenance

The warning signs on the perimeter fence will be checked and confirmed to be present, secure, and legible along the fence. Warning signs should be present on each of the 10 gates, and on the fencing at increments of 100-200 feet between gates. Signs will be replaced as necessary within 30 days of inspection deficiency.

4.5.7 Settlement Platform Maintenance

The settlement platforms will be maintained in the event that quantification of settlement is required in the future.

4.6 Annabessacook Lake Seep Inspection

During each annual landfill inspection, location SP/SED-8 will be observed for changing conditions such as the seep being exposed to the atmosphere, and the presence of exposed, stained sediment. The Cobbossee Watershed District will be contacted on the day of the inspection to obtain Annabessacook

Lake water elevation. Based on historical observations and elevation survey, a lake elevation below approximately 167 feet above mean sea level corresponds to potential seep exposure conditions.

4.7 Annual Desktop Review

- An annual desk top review and Site area reconnaissance will be performed to confirm that there have been no land use changes that could impact groundwater flow or exposure conditions at the Site. The annual review will include:
- Ordinance Compliance – check in with Winthrop Code Enforcement Officer to confirm no new development in Site Area (no change). **Figure 4.2** shows the ordinance verification area.
- Field Verification of Site area conditions – windshield reconnaissance of Mallard Lane, Hubbard Lane, Annabessacook Road adjacent to Landfill to verify ordinance compliance (completed during annual Landfill Cap inspection).

5.0 REPORTING FORMAT AND SCHEDULE

5.1 Landfill Inspection and Desktop Review Report

A landfill inspection and desktop review report will be prepared after each annual inspection. This report will include results of the inspection, Site area reconnaissance, and desktop review to document changes to the landfill or surrounding land use that could impact groundwater flow or exposure conditions at the Site.

5.2 Post-Closure Monitoring and Maintenance Report

Following sampling events every 2.5 years, an electronic data deliverable (EDD) will be prepared in the current MEDEP format along with a report (including laboratory reports) to document monitoring and maintenance activities completed at the Site.

The PCMMP report will be formatted as follows:

Section 1: Introduction

Section 2: Sampling and Analysis Program

Section 3: Data Quality Review

Section 4: Data Evaluation

Section 5: Next Steps.

The PCMMP report will typically include discussion of the following:

- Field conditions and sampling locations – event-specific conditions and deviations from scope identified above; tables and figures depicting the sampling locations.
- Water levels and groundwater flow conditions – short narrative describing the groundwater flow conditions and deviations in flow path configuration; tables and figure presenting groundwater and surface water elevations for the reporting period and groundwater contours at appropriate intervals. Every 5 years, time series plots will be updated to evaluate if groundwater elevations are consistent with the historical record of groundwater elevation data. Flowpath groundwater elevations will be compared to elevations in the bounding wells to evaluate potential changes in groundwater flow direction.
- Sampling and analytical program – short narrative and tables describing analyses performed; appendix containing the field data records and instrument calibration records.
- Data quality assessment and analytical results – narrative presenting a summary of the data validation results, and figures and tables depicting the analytical detections. Appendix containing the data validation reports, cross-tabulation of analytical and field parameter results, laboratory reports, and an EDD in MEDEP Environmental and Geographic Analysis Database version 6 (or then current) format of analytical results.
- Evaluation of results – narrative assessment of results focusing on changes with respect to historical conditions, trends in constituent concentrations, and comparison to PCLs and

ACLs; figures presenting time-series by location of constituents that have exceeded an ACL or PCL in the last five monitoring events.

- Recommendations for modifications to the PCMMP.

5.3 Schedule

A PCMMP report will be submitted within 90 days following each sampling event. The EDD will be sent to MEDEP after delivery of each PCMMP report.

5.4 Environmental Monitoring Plan Revisions

As a result of changes in Site conditions and technology, future revisions to the PCMMP may be necessary to incorporate adjustments initiated by USEPA, MEDEP, or UTC. These revisions could consist of changes to the environmental monitoring program to reflect observations in the flow path configuration of the groundwater, or changes to POE monitoring program elements. Should revisions be deemed necessary, UTC will work in concert with the USEPA and MEDEP for review, comment, and approval. At a minimum, the scope of the PCMMP will be reviewed every five years during the 5-year review process.

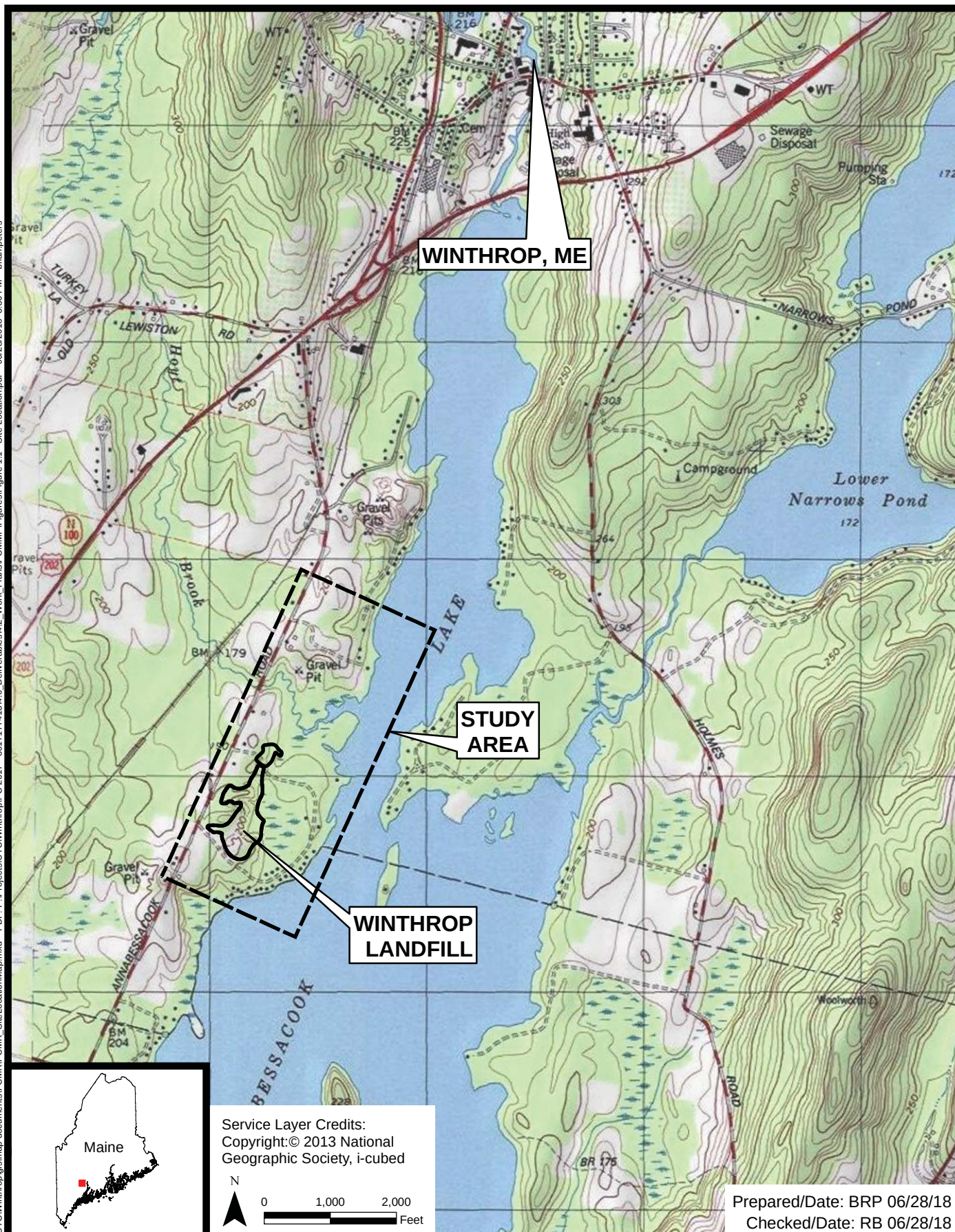
6.0 REFERENCES

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FIGURES

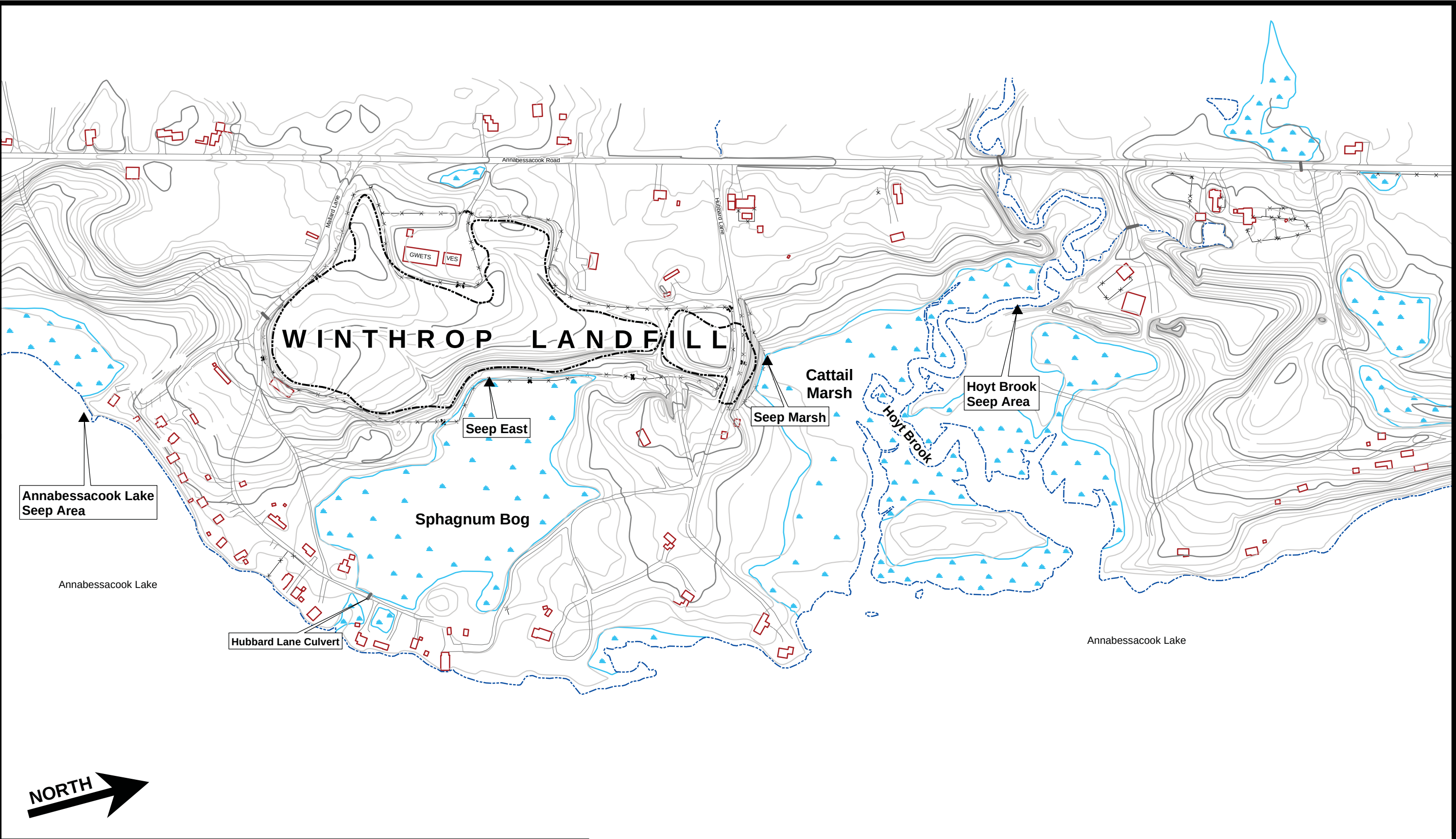
Document: P:\Projects\UTC\Winthrop\map documents\PCMR\PCMR_SiteLocationMap.mxd PDF: P:\Projects\UTC\Winthrop\PC 2017 - 3617174134_0_Deliverables\4.2_Work_Plans\PCMR\Figures\Figure 1.1 - Site Location.pdf 06/28/2018 3:36 PM brian.peters



United Technologies Corporation
Winthrop Landfill
Winthrop, Maine

wood.

Site Location
Project 3617197467 Figure 1.1



0 175 350
Feet

- Legend**
- | | | |
|--------------------------------|-----------------------------|-------------|
| — Culverts | --- Water Boundary | — Bog/Marsh |
| — Roads/Driveways | --- Landfill Limit Boundary | ××× Fence |
| — Index Contours (10 ft) | — Building Outlines | — Gate |
| — Intermediate Contours (2 ft) | | |

United Technologies Corporation
Winthrop Landfill
Winthrop, Maine

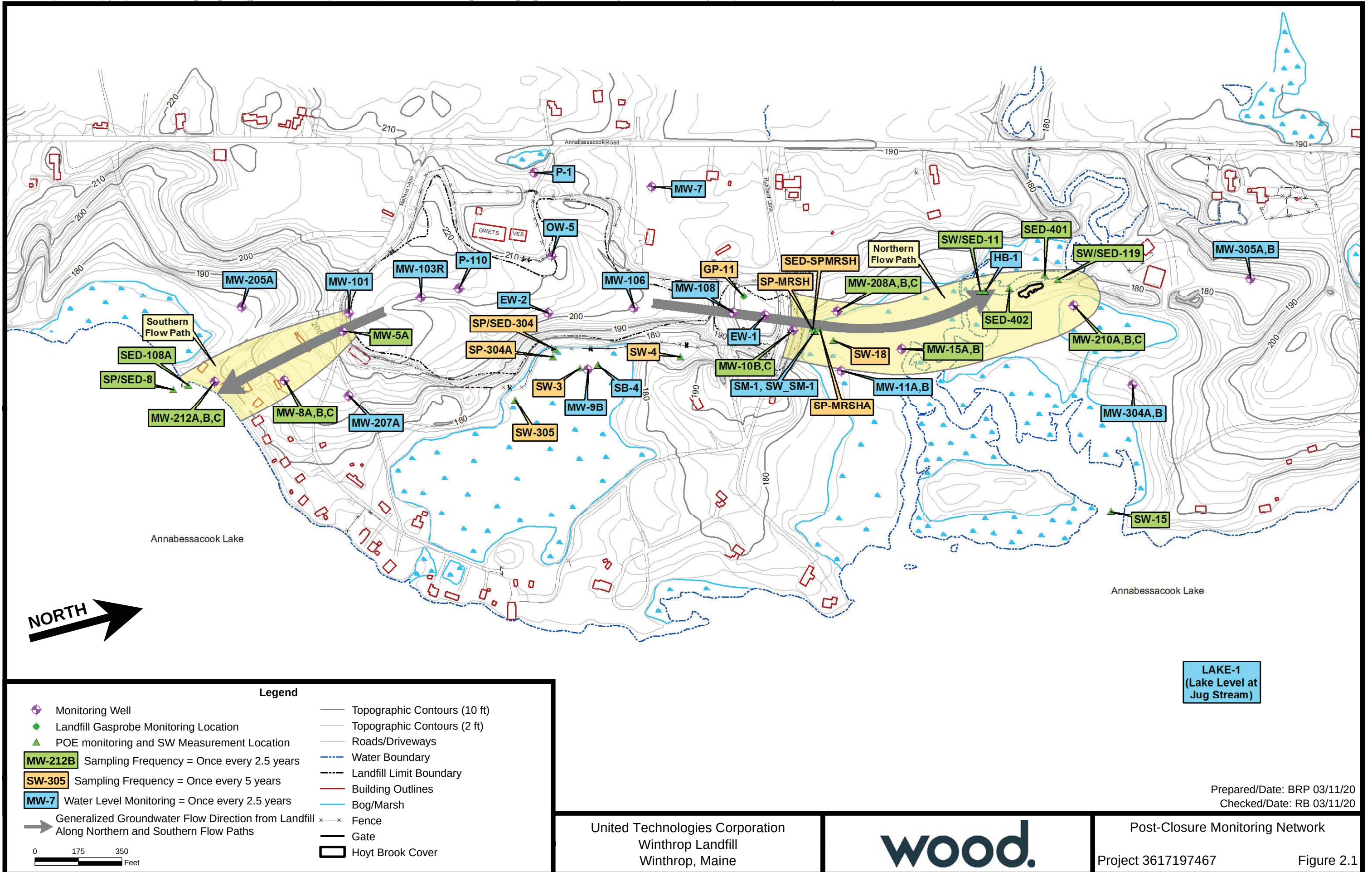
wood.

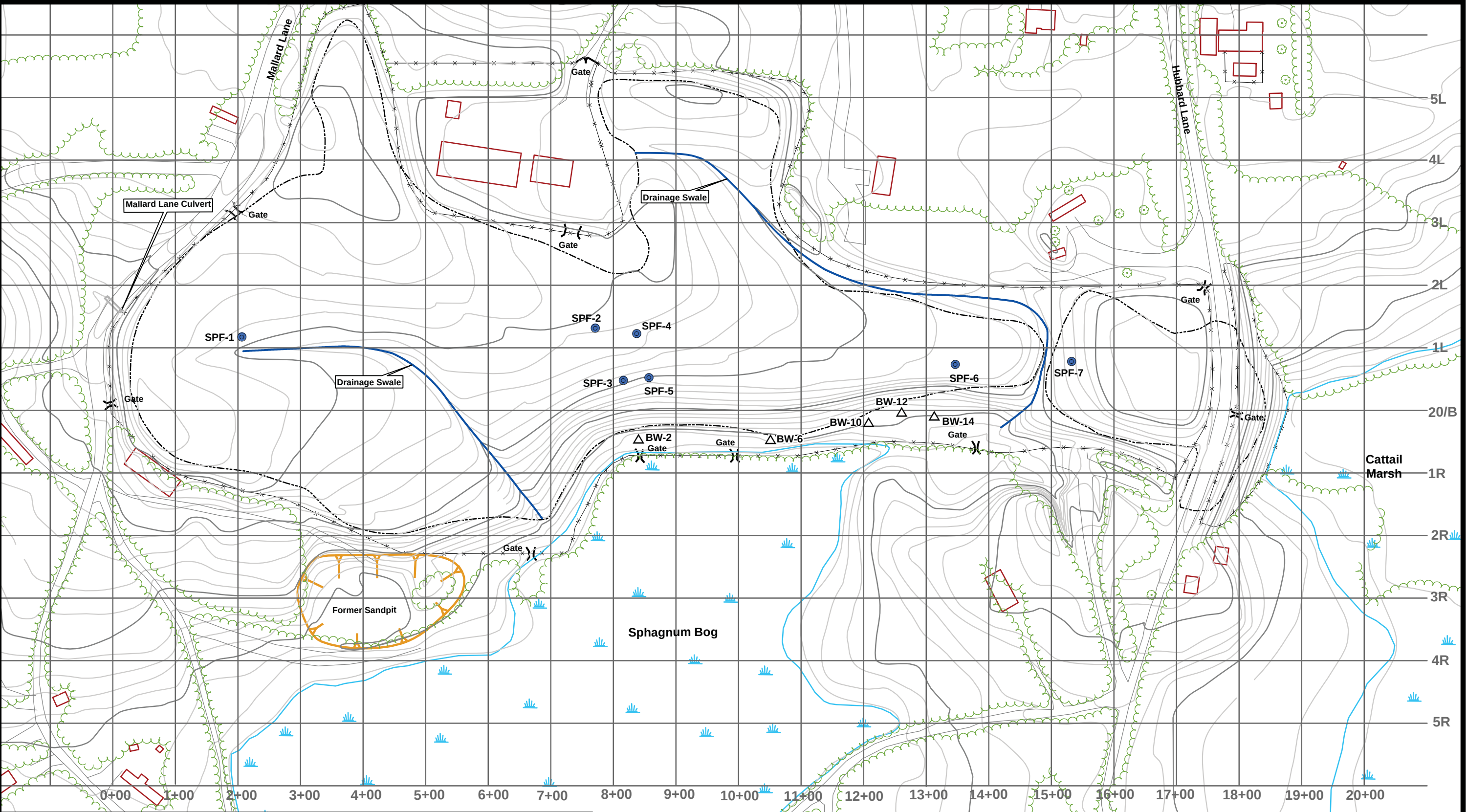
Prepared/Date: BRP 06/28/18
Checked/Date: RB 06/28/18

Site Features

Project 3617197467

Figure 1.2





0

75

150

Feet

Settlement Platforms

Slope Inclinerometer Location

Survey Grid

Tree Line

Drainage Swale

Culverts

Roads/Driveways

Index Contours (10ft)

Intermediate Contours (2ft)

Former Sandpit

Water Boundary

Landfill Limit Boundary

Building Outlines

Bog/Marsh

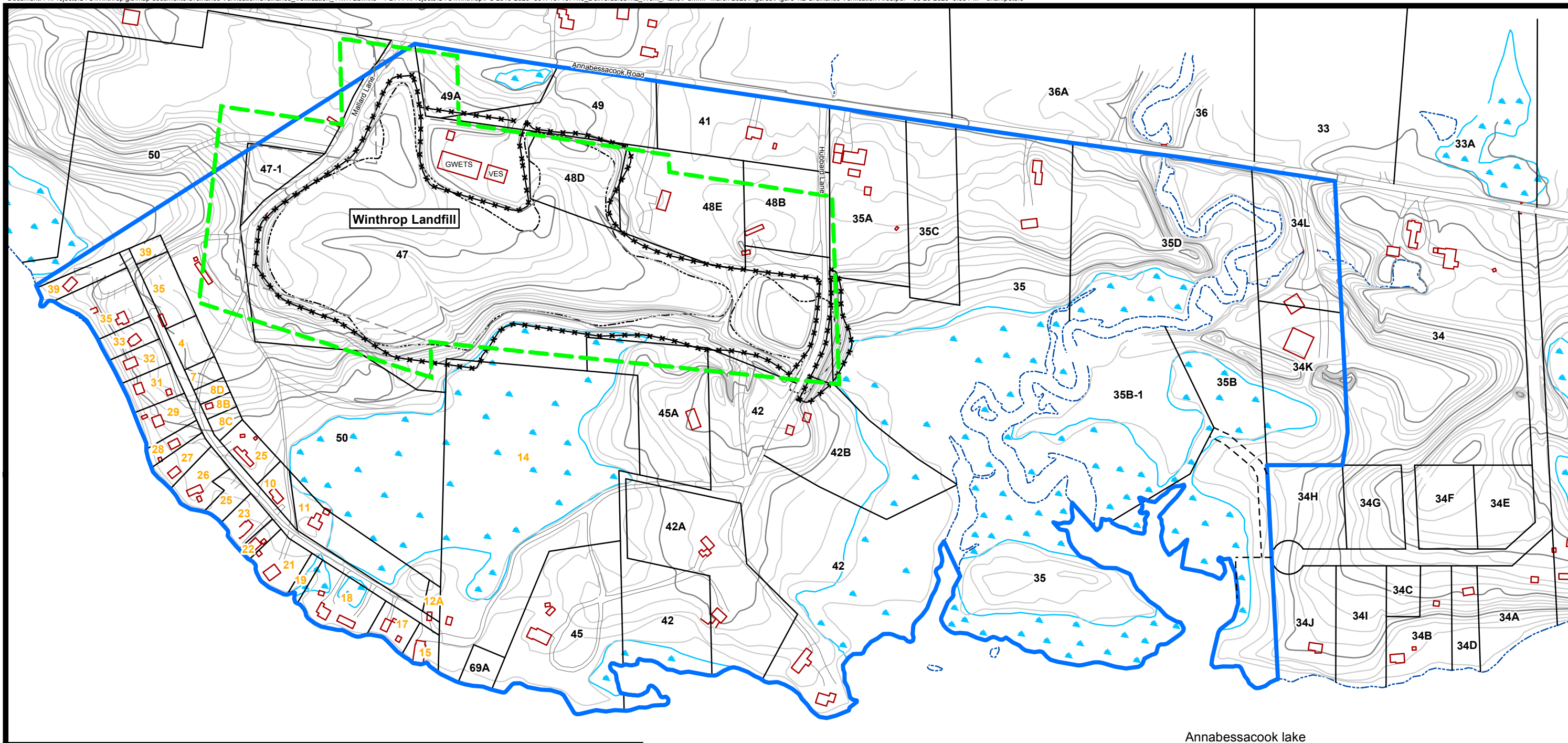
Fence

Gate

United Technologies Corporation
Winthrop Landfill
Winthrop, Maine

Winthrop Landfill Features
Project 3617197467
Figure 4.1

Prepared/Date: BRP 06/28/18
Checked/Date: RB 06/28/18



Annabessacook lake

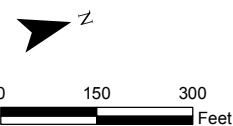
Legend

-  Groundwater Protection Ordinance Boundary  Index Contours (10 ft)
 Air Protection Ordinance Boundary  Intermediate Contours (2 ft)
 Winthrop Tax Parcel Property Boundaries  Water Boundary
 Parcel Line (Winthrop Tax Map 1985)  Landfill Limit Boundary
 Building Outlines  Bog/Marsh
 Roads/Driveways  Fence

Notes:

Notes:

1. The Air Protection Ordinance Boundary is based on the description in the "Town of Winthrop Ground Water and Air Protection Ordinance, dated April 1, 1991", utilizing Plans of Record, monumentation found and the 1985 Winthrop Tax Map.
2. Tax Map Parcels are digitized from the Town of Winthrop Tax Map # 2 and 13. These parcels are approximate in presentation and should not be interpreted as survey accurate.
49 - denotes lot # from Tax Map # 2 **19** - denotes lot # from Tax Map # 13
3. Property lines in the area of lots 35B and 35B-1 based on survey and deed review (Amec Foster Wheeler, July 2 and 3, 2015).



Prepared/Date: BRP 03-26-20
Checked/Date: RB 03-26-20

Winthrop Landfill
Winthrop, Maine

wood.

Ordinance Verification Area

Project 3617197467

Figure 4.2

TABLES

Table 2.1
Proposed Sampling and Analysis Program
Post Closure Monitoring and Maintenance Program
Winthrop Landfill

Key:

X	Sampling Frequency = Once every 2.5 years
\	Sampling Frequency = Once every 5 years

			Laboratory Parameters and Methods								Field Methods		
			RAP 1 VOCs	As Total	As Dissolved	As / Zn Total	As / Zn Dissolved	As / Ni / Zn Total	As / Ni / Zn Dissolved	Percent Solids / Moisture	Water Level Monitoring	Field Parameter Monitoring	Gas Probe*
Analysis Method			8260C/ 8270C	6010C/ 6020A	6010C/ 6020A	6010C/ 6020A	6010C/ 6020A	6010C/ 6020A	6010C/ 6020A	E245.1/SM2540G			
Location	Qc Code	Media	Points of Compliance (POC)										
MW-10B	FS	GW	\					X			X	X	
MW-10C	FS	GW	\					X			X	X	
MW-5A**	FS	GW	\					X			X	X	
Southern Flowpath Wells													
MW-212A	FS	GW	X	X							X	X	
MW-212B	FS	GW		X							X	X	
MW-212C	FS	GW		X							X	X	
MW-8A	FS	GW	X	X							X	X	
MW-8B	FS	GW		X							X	X	
Northern Flowpath Wells													
MW-15A	FS	GW		X							X	X	
MW-15B	FS	GW		X							X	X	
MW-208A	FS	GW		X							X	X	
MW-208B	FS	GW		X							X	X	
MW-208C	FS	GW		X							X	X	
MW-210A	FS	GW		X							X	X	
MW-210B	FS	GW		X							X	X	
MW-210C	FS	GW		X							X	X	
Points of Exposure (POE) - Annabessacook Lake													
SED-108A	FS	SED		X						X		X	
SED-8	FS	SED				X				X		X	
SP-8	FS	SW				X	X					X	
POE - Hoyt Brook													
SED-11	FS	SED		X						X		X	
SW-11	FS	SW		X	X							X	
SED-119	FS	SED		X						X		X	
SW-119	FS	SW		X	X							X	
SW-15	FS	SW		X	X							X	
SED-401	FS	SED		X								X	
SED-402	FS	SED		X								X	
POE - Seep East/Sphagnum Bog													
SED-304	FS	SED						\		\		\	
SP-304	FS	SW						\	\			\	
SP-304A	FS	SW						\	\			\	
SW-3	FS	SW				\	\					\	
SW-305	FS	SW				\	\					\	
SW-4	FS	SW				\	\					\	
POE - Cattail Marsh													
SED-SPMRSH	FS	SED				\				\		\	
SP-MRSH	FS	SW				\	\					\	
SP-MRSHA	FS	SW				\	\					\	
SW-18	FS	SW		\	\							\	
Landfill													
GP-11	FS												\
Additional Site Wide Water Level Monitoring Locations													
EW-1											X		
EW-2											X		
MW-7A											X		
MW-8C											X		
MW-9B											X		
MW-101											X		
MW-103R											X		
MW-106											X		
MW-108											X		
MW-11A											X		
MW-11B											X		
MW-205A											X		
MW-207A											X		
MW-304A											X		
MW-304B											X		
MW-305A											X		
MW-305B											X		
OW-5											X		
P-1											X		
P-110											X		
SM-1 (a)											X		
SW SM-1											X		
LAKE-1											X		
HB-1 (b)											X		
SB-4 (c)											X		

Notes:

- RAP 1 = Remedial Action Work Plan Table 1
As = Arsenic; Ni = Nickel; Zn = Zinc
FS = Field Sample
GW = Groundwater; SW = Surface Water; SED = Sediment
Field Parameters to include dissolved oxygen, oxidation/reduction potential, pH, specific conductance and temperature
* Gas probe monitoring to include % carbon dioxide, % methane, % oxygen, and pressure in gas probe
** Vapor intrusion evaluation to be completed every 5 years at MW-5A. Formerly analyzed with USEPA Method 524.4; however, data needs can now be met by Method 8260.
(a) A driven PVC piezometer in seep marsh north of landfill
SW_SM-1 = surface water elevation in marsh at SM-1
LAKE-1 = elevation of Annabessacook Lake at Jug Stream reported by Cobbosse Watershed District
(b) Hoyt Brook surface water elevation location HB-1 is a 0.5-inch rebar stake located near SW-11
(c) Measured from top of steel protective casing at MW-9A

Table 2.2
Well Construction Details
Post Closure Monitoring and Maintenance Program
Winthrop Landfill

Well ID	Well Type	Well Screen Interval (ft bgs)	Measuring Point Elevation (ft)	Ground Elevation (ft)	Borehole Bottom Depth (ft)
EW-1	overburden	45 - 120	204.81	NA	120
EW-2	overburden	38 - 67	201.87	201.3	69
MW-5A	overburden	~31 - ~41	203.81	202.9	~43
MW-7A	overburden	~14.5 - ~24.5	194.49	193.6	~25
MW-8A	bedrock	~106 - ~111	192.41	191.7	~111
MW-8B	overburden	~80 - ~95	192.43	191.7	~95
MW-8C	overburden	~20 - ~40	192.81	191.7	~40
MW-9B	bedrock	~114 - ~117	175.84	172.8	~119
MW-10B	overburden	~72.5 - ~82.5	193.91	191.6	~82.5
MW-10C	overburden	~10 - ~15	193.14	192.2	~15
MW-11A	overburden	~20 - ~30	174.14	171.7	~30
MW-11B	overburden	~50 - ~60	174.52	171.7	~60
MW-15A	overburden	~92 - ~102 ~122 - ~132	172.52	171.4	~140
MW-15B	overburden	~47 - ~57	173.24	171.7	~65
MW-101	overburden	25.5 - 35.5	218.32	213.5	37
MW-103R	overburden	25 - 35	210.88	207.4	35
MW-106	overburden	20 - 30	202.08	198.3	30
MW-108	overburden	27 - 37	204.4	200.8	37
MW-205A	overburden	26 - 36	180.11	177.6	41.5
MW-207A	overburden	16.5 - 26.5	196.65	195.3	27.5
MW-208A	bedrock	119 - 129	177.2	175.1	136
MW-208B	overburden	70 - 80	176.7	174.3	80
MW-208C	overburden	30 - 40	176.7	174.4	40
MW-210A	overburden	118 - 128	173.77	171.8	135
MW-210B	overburden	71.5 - 81.5	173.56	171.6	94
MW-210C	overburden	33 - 43	173.02	171.3	45
MW-212A	bedrock	116.2 - 126.2	177.05	174.6	134.5
MW-212B	overburden	64.5 - 74.5	177.11	175	74.5
MW-212C	overburden	25 - 35	177	175.4	35
MW-304A	bedrock	45.5 - 50.5	178.74	177.4	61.5
MW-304B	overburden	29 - 44	178.42	176.3	46.5
MW-305A	overburden	44 - 54	180.97	177.9	69.5
MW-305B	overburden	23 - 38	181.33	178.3	40
OW-5	overburden	42.5 - 47.5	212.17	209.7	52.5
P-1	overburden	14 - 24	206.73	204.3	24
P-110	overburden	43 - 45	216.52	NA	45

Notes:

~ = approximately
bgs = below ground surface
ft = feet
NA = Not available

Table 2.3
Post Closure Monitoring and Maintenance Schedule
Winthrop Landfill

	Landfill Inspection and Desktop Review			Environmental Sampling	
Year	Landfill Mowing	Landfill Inspection	Desktop Review	2.5-Year Sampling Event	5-Year Sampling Event
2020	X	X	X		
2021	X	X	X		X (Spring)
2022	X	X	X		
2023	X	X	X	X (Fall)	
2024	X	X	X		
2025	X	X	X		
2026	X	X	X		X (Spring)
2027	X	X	X		
2028	X	X	X	X (Fall)	
2029	X	X	X		
2030	X	X	X		
2031	X	X	X		X (Spring)

Notes:

1. Fall 2018 was the first 2.5 year PCMM event.
2. Refer to Table 2.1 for additional detail on the Environmental Sampling
3. Landfill Inspection Report is annual.
4. PCMM Report follows 90 days after sampling.

APPENDICES

APPENDIX A

PCLs and ACLs

Appendix A
Groundwater ACLs

Parameter	CAS Number	Groundwater ACL	Lab RL	Lab MDL
Volatile Organic Compounds (VOCs) via USEPA 8260B				
1,1,1-Trichloroethane	71-55-6	200	1	0.319
1,1-Dichloroethane	75-34-3	5	1	0.259
1,1-Dichloroethene	75-35-4	7	1	0.398
1,2-Dichloroethane	107-06-2	5	1	0.361
1,2-Dichloropropane	78-87-5	5	1	0.306
2-Butanone	78-93-3	170	10	3.93
2-Hexanone	591-78-6	1400	10	3.82
4-Methyl-2-pentanone	108-10-1	190	10	2.14
Acetone	67-64-1	390	50	10
Benzene	71-43-2	5	1	0.331
Chloroethane	75-00-3	1300	5	0.453
Ethylbenzene	100-41-4	440	1	0.384
Fluorotrichloromethane	75-69-4	2300	5	1.2
Methylene chloride	75-09-2	5	5	1
Styrene	100-42-5	100	1	0.307
Tetrachloroethene	127-18-4	5	1	0.372
Tetrahydrofuran	109-99-9	3300	5	1.82
Toluene	108-88-3	1000	1	0.412
trans-1,2-Dichloroethene	156-60-5	70	1	0.396
Trichloroethene	79-01-6	5	1	0.398
Vinyl chloride	75-01-4	2	1	0.259
Xylenes (total)	1330-20-7	590	3	1.06
Volatile Organic Compounds (VOCs) via USEPA 8270C				
2,4-Dinitrophenol	51-28-5	31	10	0.284
Metals - USEPA 6020A				
Arsenic	7440-38-2	30	2	0.25
Nickel	7440-02-0	88	2	0.35
Zinc	7440-66-6	59	25	2.56

Notes:

Concentrations shown in micrograms per liter (µg/L)

CAS = Chemical Abstracts Service

USEPA = United States Environmental Protection Agency

ACL = Alternate Concentration Limit

RL = Reporting Limit

MDL = Method Detection Limit

Appendix A
Surface Water ACLs

Parameter	CAS Number	Surface Water PCL	Lab RL	Lab MDL
Metals - USEPA 6020A				
Arsenic	7440-38-2	5	2	0.25
Nickel	7440-02-0	88	2	0.35
Zinc	7440-66-6	59	25	2.56

Notes:

Concentrations shown in micrograms per liter (µg/L)

CAS = Chemical Abstracts Service

USEPA = United States Environmental Protection Agency

ACL = Alternate Concentration Limit

RL = Reporting Limit

MDL = Method Detection Limit

Appendix A
Sediment ACLs

Parameter	CAS Number	Sediment PCL	Lab RL	Lab MDL
Metals - USEPA 6010C				
Arsenic	7440-38-2	31000	2000	460
Nickel	7440-02-0	50000	2000	490
Zinc	7440-66-6	270000	5000	590

Notes:

Concentrations shown in micrograms per kilogram (µg/kg)

CAS = Chemical Abstracts Service

USEPA = United States Environmental Protection Agency

PCL = Protective Concentration Limit

RL = Reporting Limit

MDL = Method Detection Limit

APPENDIX B

Sampling and Field Documentation Procedures

APPENDIX B-1

Sampling Standard Operating Procedures

WINTHROP LANDFILL SAMPLING AND FIELD DOCUMENTATION PROCEDURES

Appendix B-1 provides sampling procedures and requirements for conducting the Groundwater, Surface Water, and Sediment sample collection activities under the Post-Closure Monitoring and Maintenance (PCMM) Program. Procedures and requirements are included for sample collection (groundwater, surface water, and sediment), field documentation, and location and elevation survey.

Prior to conducting field investigation activities, field personnel must document that they have read and understand the appropriate sections of the Post-Closure Monitoring and Maintenance Plan (PCMMP) and these Standard Operating Procedures (SOPs) which are relative to the tasks to be completed. The field operations leader (FOL) will identify sections of the PCMMP that must be reviewed prior to conducting a specific field activity and will include at a minimum, applicable investigation techniques and procedures, decontamination procedures, field chain of custody (COC) procedures and investigation derived waste (IDW) handling. Work elements are described in detail below.

FIELD INVESTIGATION DOCUMENTATION PROCESS

Documentation of field activities will be completed using a combination of logbooks, field data records (FDRs), and sample custody records. Field logbooks will be completed to provide a general record of activities and events that occur during each field task. FDRs have been designed for each exploration type and sample collection task to provide a complete record of data obtained during the activity.

Deviations from the procedures specified in this PCMMP and/or SOPs will be clearly documented in the field logbooks and applicable field data records. Documentation shall include at a minimum: specific changes or modifications to procedures; reasons for the deviations; authorization for changes; and interpreted impacts to the data quality.

Field Logbooks

The field logbooks provide a daily handwritten account of field activities. Entries will be made in real-time and describe activities so that persons returning to the Site can reconstruct a particular situation without reliance on memory. Field notebooks will be bound, field survey books or notebooks. Notebooks will be assigned to field personnel, but will be stored in the project files when not in use. Each notebook will be identified with the project number and a unique logbook number. Notebook entries are made in indelible black or blue ink, and corrections are made with a single line strikeout with the entrant's initials and the date or the correction. The final page of each day's logbook entry will be dated and signed by the person completing the log. Partially completed pages will have a line drawn through the unused portion at the end of each day.

The following information is required for entry into the field logbooks:

- The date and time of each entry. The daily log should begin with weather conditions and the names and organization of personnel performing the documented task;
- A summary of important tasks or subtasks completed during the day;
- A description of field tests completed in association with the daily task;

- A description of samples collected including documentation of quality control samples that were prepared (rinse blanks, duplicates, matrix spikes, split samples);
- Documentation of equipment maintenance and decontamination activities;
- A summary of problems encountered during the day including a description of corrective actions and impacts on the daily task; and
- Signature of field representative completing the entry.

Field Data Records

In lieu of direct entry into the field logbook, FDRs will be completed to document sample collection and/or exploration details. Prior to use of FDRs, an entry must be placed in the field logbook to document relevant data and observations are recoded on the FDRs instead of the field logbook. An FDR is completed each time a field sample is collected. The goal of the FDR is to document exploration and sample collection methods, materials, dates and times, and sample locations and identifiers. Field measurements and observations associated with a given exploration or sample collection task are recorded on the FDR. FDRs are maintained throughout the field program in files that become a permanent record of field program activities. The FDRs that will be used for the PCMM program under this PCMMP include:

- Field Instrument Calibration Record
- Synoptic Groundwater and Surface Water Level Data Record
- Low-Flow Groundwater FDR
- Surface Water/Sediment FDR
- Landfill Gas Monitoring FDR
- Landfill Condition / Windshield Drive-by Visual Survey Form

FDRs, including the COC, are returned by the field operations leader directly to the project QA officer upon completion of field work. The QA officer reviews the FDRs upon receipt to resolve and correct discrepancies and deviations from procedures and the sampling analysis plan before the samples are analyzed by the laboratory.

Site Identification and Field Sample Number

To maintain consistency and comparability of sample location identification throughout the course of the current EMP, as well as previous sampling programs, samples will be labeled by sample type and exploration location (e.g., MW-10B; SED-8). The following sample types will be used:

<u>Sample Type</u>	<u>Sample Prefix Identification</u>
groundwater obtained from monitoring well	MW
surface water	SW
seep	SP
sediment	SED
gas probe	GP

Field duplicate samples will have "DUP" designated at the end of the sample ID.

Preparation of Sample Containers

Sample containers will be provided by the analytical laboratory. The analytical laboratory will obtain containers from a national supplier. Sample containers will be certified to meet USEPA standards. Certification documentation will be obtained for each lot of sample containers and filed by the laboratory for each task-specific project.

A summary of specific containers, required sample volumes, preservation and holding time requirements is provided in Table 3.2 of the PCMMP.

Decontamination and Investigation Derived Waste

Decontamination efforts will be implemented to prevent cross-contamination between sampling points and exploration locations. Sampling methods will use disposable or dedicated sampling tools and materials whenever possible to minimize the total amount of decontamination efforts. Sampling equipment will be decontaminated upon arrival, prior to leaving the Site, and between sampling activities at different locations.

Non-disposable sample equipment items will be decontaminated after each sample. The decontamination process will include a thorough scrubbing of the sampler and a double rinsing with deionized water. Decontamination of sampling equipment will be conducted at the sampling location.

Sample containers will be wiped clean after sampling before being moved to the sample handling and shipping area. Sample handling and shipping areas will be cleaned and wiped down daily.

Decontamination materials will be managed as investigation derived waste (IDW). IDW will be managed so that the potential for hazardous materials to be released to the environment is negated. IDW includes, but is not limited to: water from well purging and sampling, equipment decontamination; and personal protective equipment (PPE). The following procedures are described for the management of the different types of wastes that may be encountered during Post-Closure Monitoring and Maintenance activities:

- Sediment can be placed on the ground surface near the sampling station, and should not be intentionally returned directly to the water body.
- Groundwater derived liquids such as development water, purge water and excess samples can be returned to the ground in the vicinity of the well, taking care not to release liquid to nearby surface water bodies.
- Excess surface water samples should be discharged to the nearby ground surface.
- Decontamination fluids can be discharged back to the ground surface, avoiding discharge to surface water.
- Gases, including calibration gases and purged gases, can be discharged directly to the atmosphere.

- Decontaminated personal protective equipment and decontamination materials can be placed in a plastic bag and managed as municipal waste.
- Calibration liquids can be discharged to the ground surface since these materials are usually non-toxic and of small volume (usually less than 100 milliliters). Typical calibration liquids include pH buffer, specific conductivity standard solution, and oxidation-reduction potential solution.

FIELD INVESTIGATION TECHNIQUES AND PROCEDURES

The following subsections describe the field investigation techniques and procedures that will be performed for the characterization program. If there is any question about whether a sample should be collected, the Field Operations Leader (FOL) shall contact both the Project QA Officer and Project Manager to make a final decision on whether or not to collect a sample. Additionally, samples submitted to the laboratory will be held until the Project QA Officer has reviewed the COC and field forms and determined the samples are fit for analysis.

FIELD ANALYTICAL METHOD REQUIREMENTS

Field analytical methods planned as part of the characterization program will be limited to water quality measurements (pH, specific conductance, redox potential, turbidity, dissolved oxygen, and temperature), water level measurements, and landfill gas probe measurements.

Field equipment procedures, calibration and maintenance will be performed in accordance with the manufacturer's recommendations. Manufacturer usage guidelines will be kept with each instrument in the field. Each piece of equipment will be calibrated prior to each day's use following procedures described in the manufacturer's recommendation. Instruments requiring calibration include sampling meters for turbidity, pH, conductivity, DO, and redox potential; and hand-held monitoring equipment including gas probe. Field instrument calibration data will be recorded in the field logbook and on the Field Instrument Calibration Record (see Appendix B-2).

Field equipment and supplies include the following:

- Dedicated bladder pumps and/or tubing have been installed in monitoring wells at Winthrop Landfill for low flow application.
- Polyethylene tubing and silicone tubing may be used
- Water level meter
- 0.45µm cellulose-based membrane filter for dissolved sample fraction collection
- Power source (battery, generator, compressed gas)
- Flow-through cell with multi-probes
- Decontamination supplies
- Field Logbook
- Field Data Records
- Timer (watch)

- Sample bottles
- Labels
- Preservatives
- Well keys

Calibration procedures, frequency, acceptance criteria, and conditions that require re-calibration, as described by the manufacturer, will be followed. In addition, prior to use, and at the end of each day, the instrument will be cleaned, checked for damage, and repaired if needed in accordance with manufacturer's specifications. These activities will be documented in a field logbook.

Field monitoring equipment, including used by Wood Environment and Infrastructure Solutions, Inc. (Wood) is inspected, maintained, tracked, and staged in the Field Operations Support (FOS) department. Each piece of non-disposable equipment is affixed with a unique identification number. A computer database tracking program is used to keep track of the status and location of each piece of equipment. When equipment is returned to FOS, a technician inspects, cleans and tests the equipment to ensure that it is working properly. The technician then affixes a sticker to the equipment with the date the inspection was performed and the initials of the technician. The technician then updates the equipment database before making the equipment available for the next project. Hard copy records are maintained in FOS of major maintenance activities and parts replacements.

Groundwater Sampling Methodology

The groundwater sampling program during the course of the characterization program will be performed to further characterize the nature and distribution contaminants in groundwater at specific locations.

Non-dedicated groundwater sampling equipment will be decontaminated prior to use. Calibration of the monitoring equipment will be completed prior to each day's sampling activities and as required during the course of the day, and will be conducted in accordance with the 2017 USEPA Standard Operating Procedure for Field Instrument Calibration and the manufacturer's suggested procedures. Daily instrument calibration data will be recorded on the Field Instrumentation Calibration Record (Appendix B-2).

Groundwater samples will be collected from each monitoring location using the procedures described in the following sections. Data generated during groundwater sampling will be recorded on the Low Flow Groundwater Sample Data Sheet. To the extent practical, sampling of groundwater wells will proceed from the upgradient (background) wells to the more contaminated wells as best as can be determined, based on existing data. These details will be provided in each task-specific work plan.

Pre-purging Activities. The following activities will be performed immediately prior to purging each well:

1. Complete a synoptic round of water level measurements of wells and piezometers, if appropriate.
2. Check the well and piezometer for proper identification and location.
3. Measure and record the height of protective casing (not applicable for flush mount installations).

4. Measured and record the distance between the top of the well casing and the top of the protective casing.
5. Using the electronic water level meter, measure and record the static water level from the top of the well riser and the depth to the well bottom to the nearest 0.01 foot. Upon removing the water level wire, rinse the water level with deionized water.
6. Inspect the well for signs of poor integrity.
7. If stabilized drawdown cannot be achieved using low-flow sampling techniques, a no-purge technique will be used.
8. Place plastic sheeting on the ground surface around the well to protect sampling equipment from becoming contaminated by material located around the well.

Low-Flow Purging and Sample Collection

A variable speed peristaltic pump will be used to conduct the low-flow purging and sampling. Field parameter measurements will be made using instrumentation and a commercially manufactured flow through cell. Dedicated high density polyethylene (HDPE) tubing will be used. Further details on the low-flow purging and sampling procedure are presented in the USEPA Region I - New England Low Stress (low-flow) Purging and Sampling Procedures for the Collection of Ground Water Samples from Monitoring Wells, Revision 4, September 19, 2017. Sample collection information will be recorded on the Low Flow Groundwater Sampling Record. The following deviations from the USEPA guidance are listed below:

- The turbidity stabilization criteria of +/-10% for values greater than 10 NTU will take precedence over the turbidity stabilization criteria of +/- 10% for values greater than 1 NTU specified in the USEPA guidance.

The USEPA guidance will be used for purging and sampling procedures only. Procedures to be followed for decontamination, quality control sampling, logbook entries, etc., also presented in this guidance, are specified in other subsections within this EMP.

The following steps outline the purging and sample collection activities for low-flow sampling.

1. Open the well and measure and record the depth to water. Care should be taken to minimize disturbance of the water column within the well during pre-sample measurements. Leave the probe in the well just out of the water.
2. Decontaminate tubing prior to use (if sampling equipment is dedicated then this applies to the initial effort only).
3. Start the pump at a purge rate low enough to achieve 0.3 feet of drawdown or less based on historical data. If sampling the well for the first time, start the pump at the lowest possible setting (or approximately 100-milliliter [mL] per minute) and slowly increase the speed until discharge occurs. Check water level. Adjust pump speed until there is little or no drawdown (less than 0.3 feet) if possible. If minimal drawdown that can be achieved exceeds 0.3 feet (at a pump rate of approximately 100 mL per minute) but remains stable, continue purging until indicator field parameters stabilize. If drawdown cannot be stabilized at the lowest possible pump setting, follow the No Purge procedure at the end of this subsection.
4. Monitor and record pumping rate and water levels every 3 to 5 minutes (or as appropriate) during purging. Record adjustments to pumping rates.
5. During purging, monitor field parameters using a flow through cell (the flow through cell cannot be used for turbidity measurements and the sample for turbidity

measurement must be collected prior to entering the flow through cell using “T” valve or similar methodology). Purging is considered complete and sampling may begin when the field parameters have stabilized. Stabilization is considered to be achieved when three consecutive readings, taken at 3 to 5 minute intervals, are within the following limits:

- turbidity (+/- 10% for values >10 NTU)*
- DO (+/- 10%)
- specific conductance (+/- 3%)
- temperature (+/- 3%)
- pH (± 0.1 unit)
- redox potential (± 10 millivolts)

* If turbidity stabilizes at the greater than 25 NTU, collect both total and dissolved samples for metals and inorganics.

6. The final purge volume must be greater than the stabilized drawdown volume plus the tubing extraction volume.
7. During purging and sampling the tubing should remain filled with water.
8. Disconnect the tubing from the flow through cell to collect the analytical samples. Water samples for laboratory analyses must not be collected after water has passed through the flow through assembly. Fill sample containers directly from the tubing without alterations to the pumping rate.
9. The VOC fraction will be collected first. The VOC sample container will be completely filled without air space within the container. The remaining samples will be collected for other fractions specified in the SAP for the sample location.
10. For subsequent sampling efforts, duplicate the pump intake depth and final purge rate from the initial sampling event (use final pump dial setting information).
11. Obtain and record a depth to bottom of well measurement before closing the well.
12. After required samples are collected and put into their proper pre-preserved, laboratory-supplied containers, place in a cooler for transportation to the laboratory.
13. Document sample collection activities in the field log book and on the groundwater sampling Field Data Record.
14. Initiate sample COC.

No Purge Groundwater Sampling Procedure

1. If drawdown cannot be stabilized, adjust and stabilize the flow rate to the lowest sustainable yield, usually around 0.1 liter per minute, and discharge a minimum of one tubing volume.
2. Collect samples directly into sample vessels from the pump discharge. The order of sample collection is: VOCs, field-filtered parameters, total metals, and inorganics.
3. When vessels are filled, record the field parameters until stable or until the well runs dry. Shut off the pump and record the time and the total volume purged from the well. Close and secure the well.
4. After required samples are collected and put into their proper pre-preserved, laboratory-supplied containers, place in a cooler for transportation to the laboratory.
5. Document sample collection activities in the field log book and on the groundwater sampling Field Data Record.
6. Initiate sample COC.

Surface Water Sampling Methodology

The surface water sampling program will be performed to collect information representative of exposure conditions needed to evaluate risks that may result if landfill constituents are detected. Surface water samples will be collected no sooner than 24 hours after a precipitation event, unless the Project Manager and FOL determine that the precipitation will not adversely affect the technical objective of the sample.

The surface water column will be measured using a weighted tape, engineer's rule, or other device. The technique for surface water sampling is responsive to the following sampling location conditions and sampling and analysis objectives:

- Depth of water body
- Flow direction
- Analytical parameters of interest

Sample collection devices for surface water samples collected at the Winthrop Landfill site include:

- Wheaton Bomb Sampler
- Peristaltic Pump (with silicon and PVC tubing)
- Inline filter
- Glass Beaker

Surface water / seep samples will be collected in the following manner:

1. Collect the sample from the surface water body at a depth of approximately one foot above the bottom of the water column. Direct collection of surface water with the sample containers will be done in shallow water only (less than one foot). Avoid collecting floating debris and loss of volatiles from the water surface. Take precautions to avoid stirring up silt or sediment in the area where surface water samples are obtained. Chest waders or an electric or hand powered boat may be used to access a sample location. A boat may be used for surface water sample collection. If a boat is used it, maintain the boat's position to the extent practicable at the designated sample location taking care not to unnecessarily suspend sediments with the anchorage. Gasoline powered equipment shall not be used, to avoid sample contamination. Precautions shall be taken to avoid stirring up silt or sediment in the area where surface water samples are obtained. If wading is necessary, enter the sample area without disturbing the sediment. If a disturbance is observed, the wait until the water is visually clear before collecting the samples. If the water is flowing, stand downstream from the sample location.
2. In shallow waters less than 6 inches deep, unpreserved sampling bottles can be filled by dipping the bottles directly into the water. Pre-preserved bottles are not to be dipped directly into the water to avoid loss of preservatives to the water source. Instead, the sample is obtained using the glass beaker and then transferred to the pre-preserved bottle.

3. At dry locations or when there is an insufficient water column to sample, no surface water sample will be collected at that sampling location. The sampling location should not be excavated or otherwise altered to find water at dry locations. This applies to seep locations as well. If unsure whether a sample should be collected, contact both the Project QA Officer and Project Manager.
4. Measure the following parameters, if possible, directly in the water body lowering the multi meter wand to one foot above the bottom of the water column:
 - temperature
 - pH
 - DO
 - specific conductance
 - oxidation-reduction potential
 - turbidity.

For parameters measured in separate meters, use the Wheaton Bomb Sampler to obtain water from one foot above the bottom of the water columns.

5. After required samples are collected and put into their proper pre-preserved, laboratory-supplied containers, place in a cooler for transportation to the laboratory.
6. Document sample collection activities in the field log book and on the Surface Water Field Data Record.
7. Initiate sample COC.

Sediment Sampling Methodology

Sediment samples are usually taken in conjunction with surface water samples. The objective of sediment sampling, like surface water, is to collect information representative of exposure conditions needed to evaluate risks that may result if landfill constituents are detected.

Where sediments are to be obtained in the immediate vicinity of an associated surface water sample, discrete samples will be obtained from the surface (up to 0.5 foot deep) of the sediment. Surface water samples will be collected prior to the sediment sample to avoid stirring up the sediment. Field notes should be similar to those recorded for surface water, but focused on the sediment. Sample collection information will be recorded on the Surface water and Sediment Sampling Record presented in Appendix B-2.

Sediment will be collected regardless of whether surface water is present at the sampling location. Sediment will be collected from areas where sediment has been documented to be present. This would exclude, for example, collecting peat from Sphagnum Bog and will limit sample collection in the wetland areas, Cattail March and Sphagnum Bog, to the immediate perimeter of the landfill at the fence line. Sediment samples will be preferentially collected from stained areas, characteristically orange-colored, and observed areas of active seepage.

The sample collection devices used on a site specific basis are as follows:

- Aluminum pan
- Stainless Steel Spoon

- Stainless Steel Spatula
- Hand Core Sampler

Samples are collected in laboratory supplied containers (Table 3.2) by the following procedures depending on their location.

If a Hand Core Sampler is used to obtain sediments, the sampler would perform the following procedures. A stainless steel spoon may also be used to grab the sediment. The collection procedures using a stainless steel spoon would follow parts 2 through 5 as below:

1. Lower the bladed end to the sediment and turn the "T" end clockwise until it is about 6 inches deep. Once it reaches the desired depth, pull the core sampler out of the sampling location.
2. Empty the sediment from the Core Sampler into an aluminum pan. Additional sample is collected and emptied into the aluminum pan. This procedure is repeated until enough sediment is collected to fill the required containers for analysis. Once enough sample is collected, it is then gently mixed in the aluminum pan and spooned into the appropriate sample containers.
3. After required samples are collected and put into their proper pre-preserved, laboratory-supplied containers, place in a cooler for transportation to the laboratory.
4. Document sample collection activities in the field log book and on the Surface Water Field Data Record.
5. Initiate sample COC.

Landfill Gas Monitoring Methodology

Landfill gas monitoring is performed to evaluate on-Site landfill methane concentrations. Measurements are taken using a hand-held detector, typically a Landtec GEM 5000 or equivalent. The detector is calibrated with standard gases in the field prior to use. The detector is attached directly to the gas probe tubing. The highest measurement of methane on the detector is recorded. Measurements are recorded on the Landfill Gas Monitoring FDR for the following parameters:

- Gas pressure
- Percent methane
- Percent carbon dioxide
- Percent oxygen

Gas pressure will be recorded to the nearest 0.1 inch of water. Percentages will be recorded to the nearest 0.1 percent.

FIELD QUALITY CONTROL SAMPLES

Several types of field QC samples will be collected to provide additional data that will be used to evaluate whether the sample collection and handling procedures have affected the quality of the

samples. Where duplicate, MS/MSD, or split samples are to be collected, samplers should fill containers for a given analytical parameter before moving on to the next parameter. These samples include:

- **Field Duplicates** - Field duplicates are collected from the same sampling location, depth, or interval as the original field sample. Field duplicate samples are samples that have been divided into two or more portions at the same step in the sample collection process to measure sampling precision and analytical precision at the laboratory (see Table 3.3 for QC Limits). The field duplicate samples will be shipped along with field samples and analyzed by the same laboratory. Field duplicates will be collected at a rate of 5 percent for each media where total field sample counts are 6 or greater.
- **Matrix Spike/Matrix Spike Duplicate** – Matrix spikes are known amounts of specific chemical constituents added by the laboratory to selected samples to evaluate the effect of the sample matrix on the preparation and analytical procedures. Matrix spikes performed in duplicate and are referred to as MS/MSDs. Spike samples have the exact same ID as the original sample and are identified on the chain of custody. Additional volume may need to be provided to the lab. Matrix spikes will be collected at a rate of 5 percent for each media where total field sample counts are 9 or greater.
- **Equipment Blanks** - Equipment blanks are collected to evaluate the effectiveness of sampling equipment decontamination. After decontamination of the sampling device, the water used for final rinsing of the equipment is poured directly over or through the equipment, collected in the sample container, and shipped with the investigative samples for laboratory analyses. Rinse blanks will not be collected if dedicated equipment is used or if samples are collected without sampling equipment. Equipment blanks are collected at least once per sampling process for groundwater/storm water and soils.
- **Trip Blank** - Trip blanks assess potential contamination of the samples by VOCs during sample transport. The trip blank consists of VOC sample container filled at the laboratory with water (water samples) or a vial prepared with methanol or sodium bisulfate (soil). These containers are shipped to the Site with the sample containers, transported with the sample bottles to the sampling location, and an unopened trip blank is returned to the laboratory with each shipment of samples for analyses.

SAMPLE HANDLING REQUIREMENTS

This section provides sample handling procedures that will be followed as part of the Winthrop Landfill monitoring program. The handling procedures described below will be followed to assure that each sample is accounted for at all times. To maintain sample integrity, container labels and COC Records will be used to document sample collection.

The primary objective of sample custody procedures is to obtain an accurate written record that can trace the handling of samples during the sample collection process, through analysis, until final disposition. Logbooks will be used to record field collection activities: information pertaining to sample locations, numbers/types of samples, field measurements, and sampling conditions. Documentation will be done in indelible blue or black ink.

Field Sample ID Formats

Wood will uniquely identify every sample collected at the Site. The field sample identification is a flexible system derived to handle the different media to be sampled, and the variety of potential horizontal and vertical sample locations.

For pre-existing sampling locations, the current well ID will be used for identification (i.e., MW-5A or SW-3).

Trip blanks and rinse blanks will be labeled as “TB” or “RB” and followed by the date of collection. For example, a trip blank collected on December 11, 2013 will be labeled as TB121113.

For new sample locations, the following labeling system will be used:

Digits 1, 2 A two letter sample type designation will be used for the first two characters of the sample. The following list shows the different types of samples and letter designation that may be used:

MW	=	Monitoring Well Groundwater
SW	=	Surface Water
SP	=	Seep
SED	=	Sediment

Digits 3,4,5 Characters 3, 4, and 5 constitute the horizontal locator. Typically, the monitoring well or soil boring name is reported here. The locator must be three digits (right justified) so any code with less than three digits will include “0” for spacing (e.g., SB002).

Digits 6, 7 Characters 6 and 7 act as the vertical locator. The top of the depth interval from which the sample is collected is reported here. Ground surface will be the datum (or grade). For example, a soil sample, groundwater grab sample, or soil vapor sample obtained from 4 to 6-feet below grade would be designated “04”. For storm water and indoor air samples, the vertical designation would be “XX”.

Digit 8, 9 This two-character code will be used to identify samples, field duplicates and matrix spike samples. For regular samples, XX will be used; for a duplicate sample, a “XD” will be used; for a matrix spike, “MS” will be used and for a matrix spike duplicate “MD” will be used.

Field Sample Custody

Sample custody for samples collected during each sampling event will be maintained by the FOL or the personnel collecting the samples. Each sampler is responsible for documenting each sample transfer, maintaining sample custody until samples are shipped off-Site, and sample shipment. The sample custody protocol followed by the sampling crews involves:

- documenting procedures and amounts of reagents or supplies (e.g., filters) which become an integral part of the sample from sample preparation and preservation
- recording sampling locations, sample bottle identification, and specific sample acquisition measures on the appropriate forms
- using sample labels to document information necessary for effective sample tracking

- completing sample FDR forms to establish sample custody in the field before sample shipment

Prepared labels are normally developed for each sample prior to sample collection. At a minimum, each label will contain:

- sample location and depth (if applicable)
- date and time collected
- sampler identification
- analyses requested and applicable preservative

A COC Record generated from the Wood Sample Tracking Program or manually prepared will be initiated at the time of sample collection. The COC Record documents:

- project name, number, and location
- COC number
- location ID
- field sample ID
- date and time of collection
- sample matrix
- sample purpose (or QC code)
- analytical methods
- sample preservation information
- bottles type and number of sample containers
- sample handling procedures including sample location, sample number and number of containers corresponding to each sample number
- the names of the sampler(s) and the person shipping the samples
- signature of sampler and sample manager and time relinquished
- the date and time that the samples were delivered for shipping
- the names of those responsible for receiving the samples at the laboratory
- may note removals of various aliquots for analysis, as necessary
- comments or observations
- note for the laboratory to hold analyses pending approval by Wood.

To prevent nonconformance with SOPs, the COC will include a note for the laboratory to hold analyses pending approval by Wood. COC Records will be prepared and tracked by sample delivery group (SDG). SDGs will be identified by Wood during sampling activities and will contain up to twenty (20) field samples, inclusive of associated field QC samples.

Sample Container Packing

Sample containers will be packed in metal or plastic coolers for shipment. Bottles will be packed tightly so that no motion is possible. Styrofoam, vermiculite, and "bubble pack" are suitable as packing materials for most instances. Ice will be placed in the cooler along with paperwork in a separate "Ziploc" bag. A temperature blank provided by the laboratory will also be placed in each cooler prior to shipment to verify the cooler was maintained at 4oC (+/- 2oC) during sample shipment.

Sample Shipment

Samples will be shipped to the laboratory via laboratory courier service or delivered by Wood within 48-hours of sample collection. Prior to leaving for the field, the person responsible for sample collection must notify the analytical laboratory of the number, type and approximate collection and shipment dates for the samples. If the number, type or date of shipment changes due to Site constraints or program changes, the task leader must notify the analytical laboratory of the changes. This notification from the field also needs to occur if sample shipments are scheduled to arrive on Saturdays.

Laboratory Receipt and Custody

Once the samples are received at the analytical laboratory, the field COC is completed and signed by the laboratory's sample custodian. The sample custodian will then initiate laboratory COC protocols (comparing sample bottle labels against COC Record and noting discrepancies).

After sample receipt information is checked and recorded, the sample analysis information is entered into the laboratory's information system. The laboratory provides a unique sample identification number to each environmental sample for internal laboratory sample custody. The internal COC will document the transfer of samples from the storage location to the analyst for analysis and subsequently through final disposition. The signed field COC and internal laboratory COC Records will be provided with the laboratory deliverables for the project.

The Project QA Officer will review the complete package of FDRs, the photo log, and the COC upon receipt and will instruct the laboratory to make necessary corrections to submitted samples and associated analyses within three days of receipt of the samples by the laboratory and prior to laboratory analysis. The Project QA Officer will instruct the laboratory to proceed with analysis as appropriate.

APPENDIX B-2

Field Documentation Records

FIELD INSTRUMENTATION CALIBRATION RECORD

PROJECT NAME: <u>Winthrop Landfill Post-Closure Monitoring Program</u>	TASK NO: _____	DATE: _____
PROJECT NUMBER: <u>3617177413.01.02</u>	WOOD CREW: _____	
PROJECT LOCATION: <u>Winthrop</u>	SAMPLER NAME: _____	
WEATHER CONDITIONS (AM): _____	SAMPLER SIGNATURE: _____	
WEATHER CONDITIONS (PM): _____	CHECKED BY: _____ DATE: _____	

MULTI-PARAMETER WATER QUALITY METER

METER TYPE _____	<u>AM CALIBRATION</u>				<u>POST CALIBRATION CHECK</u>		
MODEL NO. _____	Start Time _____	End Time _____			Start Time _____	End Time _____	
UNIT ID NO. _____	Units	Standard Value	Meter Value	*Acceptance Criteria (AM)	Standard Value	Meter Value	*Acceptance Criteria (PM)
pH (4)	SU	4.0	_____	+/- 0.1 pH Units			
pH (7)	SU	7.0	_____	+/- 0.1 pH Units	7.0	_____	+/- 10% of standard
pH (10)	SU	10.0	_____	+/- 0.1 pH Units			
Redox	+/- mV	240	_____	+/- 10 mV	240	_____	+/- 10% of standard
Conductivity	mS/cm	1.413	_____	+/- 0.5% of standard	1.413	_____	+/- 10% of standard
DO (saturated)	%	100	_____	+/- 2% of standard			
DO (saturated) mg/L ¹ (see Chart 1)			_____	+/- 0.2 mg/L	_____	_____	+/- 10% of standard
DO (<0.1)	mg/L	<0.1	_____	≤ 0.5 mg/L			
Temperature	°C		_____				
Baro. Press.	mmHg		_____				

TURBIDITY METER

METER TYPE _____	Units	Standard Value	Meter Value	Standard Value	Meter Value	*Acceptance Criteria (PM)
MODEL NO. _____						
UNIT ID NO. _____	<0.1 Standard	NTU	<0.1	<0.1	_____	+/- 0.3 NTU of stan.
	20 Standard	NTU	20	20	_____	+/- 10% of standard
	100 Standard	NTU	100	100	_____	+/- 10% of standard
	800 Standard	NTU	800	800	_____	+/- 10% of standard

PHOTOIONIZATION DETECTOR

METER TYPE _____	Background	ppmv	<0.1	_____	<0.1	_____	within 5 ppmv of BG
MODEL NO. _____							
UNIT ID NO. _____	Span Gas	ppmv	100	_____	100	_____	+/- 10% of standard

O₂-LEL 4 GAS METER

METER TYPE _____	Methane	%	50	_____	50	_____	+/- 10% of standard
MODEL NO. _____	O ₂	%	20.9	_____	20.9	_____	+/- 10% of standard
UNIT ID NO. _____	H ₂ S	ppmv	25	_____	25	_____	+/- 10% of standard
	CO	ppmv	50	_____	50	_____	+/- 10% of standard

OTHER METER

METER TYPE _____	_____	_____	_____	_____	_____	_____	See Notes Below for Additional Information
MODEL NO. _____	_____	_____	_____	_____	_____	_____	
UNIT ID NO. _____	_____	_____	_____	_____	_____	_____	

☐

Equipment calibrated within the Acceptance Criteria specified for each of the parameters listed above.

☐

Equipment (not) calibrated within the Acceptance Criteria specified for each of the parameters listed above**.

MATERIALS RECORD

Cal. Standard Lot Number

Exp. Date

Deionized Water Source: <u>Portland FOS</u> Lot#/Date Produced: _____ Trip Blank Source: _____ Sample Preservatives Source: _____ Disposable Filter Type: <u>0.45µm cellulose</u> Calibration Fluids / Standard Source: - DO Calibration Fluid (<0.1 mg/L) <u>Portland FOS</u> - Other _____ - Other _____ - Other _____	pH (4) _____ pH (7) _____ pH (10) _____ ORP _____ Conductivity _____ <0.1 Turb. Stan. _____ 20 Turb. Stan. _____ 100 Turb. Stan. _____ 800 Turb. Stan. _____ PID Span Gas _____ O ₂ -LEL Span Gas _____ Other _____

NOTES:

wood.

511 Congress Street,
Portland Maine 04101

* = Unless otherwise noted, calibration procedures and acceptance criteria are in general accordance with USEPA Region 1 SOPs for Field Instrument Calibration (EQASOP-FieldCalibrat) and Low Stress Purging and Sampling (EQASOP-GW001), each dated 1/19/2010. Additional acceptance criteria obtained from instrument specific manufacturer recommendations.

** = If meter reading is not within acceptance criteria, clean/replace probe and re-calibrate, or use calibrated back-up meter if available. Contact the PM/Technical lead if issues persist. If project requirements necessitate use of the instrument, clearly document any deviations from acceptance criteria on all data sheets and log book entries.

1 = DO Saturated standard value is calculated based on Oxygen Solubility at Indicated Pressure Chart from the USEPA Region 1 SOP for Field Instrument Calibration (EQASOP-FieldCalibrat), dated 1/19/2010.

SYNOPTIC GROUNDWATER AND SURFACE WATER ELEVATIONS								WINTHROP LANDFILL		
Monitoring Event: _____										
Date: _____ Personnel: _____ PAGE ____ OF ____										
Area Description	Location	Known Installed Depth from PVC Riser	HAS LOCK	HAS LABEL	CASING CAP	PVC CAP	Casing Stick-up Elevation (Tenths of Feet)	Top of Casing to PVC Riser (Hundredths of Feet)	Depth to Water (Hundredths of Feet)	Comments
			(Mark as Y or N or NA)							
Groundwater Locations										
Upgradient	P-1	26.9								
Upgradient	MW-7A	26.2								
Landfill Cap	OW-5	50.1								
Landfill Cap	EW-2	67.0								
Landfill Cap	P-110	54.0								
Landfill Cap	MW-103R	47.7								
Landfill Cap	MW-101	55.8								
Landfill Cap	MW-5A	42.5								
Landfill Cap	MW-106	37.2								
Landfill Cap	MW-108	46.9								
Landfill Cap	EW-1	122.8								
Landfill Cap	MW-10B	95.5								
Landfill Cap	MW-10C	26.9								
Northern Flowpath	MW-11A	32.7								
Northern Flowpath	MW-11B	63.1								
Northern Flowpath	MW-208A	131.2								
Northern Flowpath	MW-208B	82.5								
Northern Flowpath	MW-208C	42.6								
Northern Flowpath	MW-15A	133.5								
Northern Flowpath	MW-15B	57.0								
Northern Flowpath	MW-210A	130.7								
Northern Flowpath	MW-210B	84.1								
Northern Flowpath	MW-210C	44.7								
Northern Flowpath	MW-304A	53.0								
Northern Flowpath	MW-304B	46.6								
Northern Flowpath	MW-305A	57.1								
Northern Flowpath	MW-305B	41.2								
Southern Flowpath	MW-205A	38.8								
Southern Flowpath	MW-207A	29.9								
Southern Flowpath	MW-8A	112.5								
Southern Flowpath	MW-8B	96.7								
Southern Flowpath	MW-8C	41.3								
Southern Flowpath	MW-212A	128.9								
Southern Flowpath	MW-212B	76.7								
Southern Flowpath	MW-212C	36.9								
Sphagnum Bog	MW-9C	63.0								
Surface Water Locations										
Annabessacook Lake	LAKE-1	Lake elevation measured at Jug Stream by Cobbesees Watershed District								
Hoyt Brook	HB-1	Rebar staff guage located about 150ft downstream of SP-114N								
Sphagnum Bog	SB-4	Measured from Top of steel Casing at MW-9A								
(outside) Seep Marsh	SW_SM1	Measured from top of PVC at Marsh Piezometer								
(inside) Seep Marsh	SM-1	Measured from top of PVC at Marsh Piezometer								

wood.

PROJECT NAME		Winthrop
PROJECT NUMBER		3617177413.01.02
SAMPLE ID	SAMPLE TIME	

LOCATION ID	DATE
START TIME	END TIME
SITE NAME/NUMBER	PAGE 1 OF 1

MEASUREMENT POINT (MP) ☐ TOP OF RISER (TOR) ☐ TOP OF CASING (TOC) ☐ OTHER

YES	NO	N/A

CAP	_____	_____	_____
CASING	_____	_____	_____
LOCKED	_____	_____	_____
COLLAR	_____	_____	_____

CALCULATED GAL/VOL	GAL	TOTAL VOL. PURGED	GAL	DRAWDOWN/ TOTAL PURGED	PRESSURE TO PUMP	PSI

(water column X well diameter² X 0.041) (mL per minute X total minutes X 0.00026 gal/mL)

BEGIN PURGING

FINAL STABILIZED FIELD PARAMETERS (to appropriate significant figures[SF])

TEMP.: nearest degree (ex. 10.1 = 10)
COND.: 3 SF max (ex. 3333 = 3330, 0.696 = 0.696)
pH: nearest tenth (ex. 5.53 = 5.5)
DO: nearest tenth (ex. 3.51 = 3.5)
TURB: 3 SF max, nearest tenth (6.19 = 6.2, 101 = 101)
ORP: 2 SF (44.1 = 44, 191 = 190)

TYPE OF PUMP

☐	PERISTALTIC
☐	SUBMERSIBLE
☐	BLADDER
☐	WATTERA
☐	OTHER
☐	OTHER

DECON FLUIDS USED

	LIQUINOX	
	DEIONIZED WATER	
	POTABLE WATER	
	NITRIC ACID	
	HEXANE	
	METHANOL	
X	OTHER	Dedicated

TUBING/PUMP/BLADDER MATERIALS

X	SILICON TUBING		S. STEEL PUMP MATERIAL
	TEFLON TUBING		PVC PUMP MATERIAL
	TEFLON LINED TUBING		GEOPROBE SCREEN
	HDPE TUBING		TEFLON BLADDER
X	LDPE TUBING		OTHER _____
	OTHER _____		OTHER _____
	OTHER _____		OTHER _____

EQUIPMENT USED

X	WL METER	_____	_____
	PID	_____	_____
X	WQ METER	_____	_____
X	TURB. METER	_____	_____
	PUMP	_____	_____
	OTHER	_____	_____
	<u>FILTERS</u>	NO.	TYPE

PARAMETER

[illegible]

PURGE WATER	YES	NO
CONTAINERIZED	☐	☒
NO-PURGE METHOD	YES	NO
UTILIZED	☐	☐

NUMBER OF GALLONS
GENERATED _____

If yes, purged approximately 1 standing volume prior
to sampling or _____ mL for this sample location.

Sampler Signature: _____
Checked By: _____

Print Name: _____
Date: _____

LOCATION SKETCH

SURFACE WATER AND SEDIMENT SAMPLING RECORD



Wood E&IS
511 Congress Street
Portland Maine 04101

PROJECT NAME Winthrop Landfill Algae Sampling Program	
PROJECT NUMBER 3617177413.01.02	
SAMPLE ID	SAMPLE TIME

SAMPLE LOCATION	DATE
START TIME	END TIME
SITE NAME/NUMBER	PAGE OF

SURFACE WATER DATA

WATER DEPTH AT _____ DEPTH OF SAMPLE BELOW _____ FLOW RATE _____
SAMPLE LOCATION _____ FT. WATER SURFACE _____ FT. _____ ML/MIN

WATER QUALITY PARAMETERS:

TEMPERATURE _____ °C
SPEC. COND. _____ mS/cm
PH _____ pH Units
ORP _____ mV
TURBIDITY _____ NTUs
DO _____ mg/L

☐ WINKLER METHOD
☐ DO PROBE

EQUIPMENT USED:

☐ BEAKER
☐ BOTTLE
☐ PACS BOMB
☐ PUMP
☐ FILTER
No. _____ Type: _____

☐ FIELD DUPLICATE COLLECTED
DUP. ID _____

TYPE OF SURFACE WATER:

☐ STREAM
☐ RIVER
☐ LAKE
☐ POND
☐ SEEP

FIELD SKETCH SHOWN/ATTACHED

☐ YES ☐ NO

DECON FLUIDS USED

☐ ALL USED
☐ LIQUINOX/DI H₂O SOLUTION
☐ DEIONIZED WATER
☐ POTABLE WATER
☐ NITRIC ACID
☐ HEXANE
☐ 25% METHANOL/75% ASTM TYPE II H₂O
☐ ETHYL ALCOHOL

SAMPLING EQUIPMENT

WATER QUALITY METER MODEL NO. _____ UNIT ID NO. _____
TURBIDITY METER MODEL NO. _____ UNIT ID NO. _____

SEDIMENT SAMPLE INFORMATION

TYPE OF SAMPLE

☐ DISCRETE
☐ COMPOSITE

QC SAMPLES

☐ DUPLICATE _____
☐ EQ BLK _____

MS/MSD:

☐ YES
☐ NO

SAMPLE INTERVAL:

TOP _____
BOTTOM _____

TYPE OF MATERIAL:

☐ ORGANIC
☐ SAND
☐ GRAVEL
☐ CLAY
☐ FILL
☐ OTHER _____

COLLECTION EQUIPMENT

☐ HAND AUGER/CORER
☐ S.S. SPLIT BARREL
☐ ALUMINIUM PAN
☐ S.S. SHOVEL
☐ HAND SPOON/SPATULA
☐ S.S. BUCKET
☐ OTHER _____

SAMPLE OBSERVATIONS

ODOR _____
COLOR _____
OTHER _____
PID _____

DECON FLUIDS USED

☐ ALL USED
☐ LIQUINOX/DI H₂O SOLUTION
☐ DEIONIZED WATER
☐ POTABLE WATER
☐ NITRIC ACID
☐ HEXANE
☐ 25% METHANOL/75% ASTM TYPE II H₂O
☐ ETHYL ALCOHOL

FIELD SKETCH SHOWN/ATTACHED

☐ YES
☐ NO

ANALYTICAL PARAMETERS

	PARAMETER	METHOD NUMBER	PRESERVATION METHOD	VOLUME REQUIRED		SAMPLE COLLECTED	QC COLLECTED	BOTTLE ID NUMBERS
				SW	SED			
☐	RAP 1 VOCs	8260C	HCl, 4° C	3 X 40 ml	-			
☐	Total As	6010C/6020A	HNO ₃ , 4° C	1 X 500 ml P	1 X 4 oz. A			
☐	Dissolved As	6010C/6020A	HNO ₃ , 4° C	1 X 500 ml P	1 X 4 oz. A			
☐	Total As/Zn	6010C/6020A	HNO ₃ , 4° C	1 X 500 ml P	1 X 4 oz. A			
☐	Dissolved As/Zn	6010C/6020A	HNO ₃ , 4° C	1 X 500 ml P	1 X 4 oz. A			
☐	Total As/Ni/Zn	6010C/6020A	HNO ₃ , 4° C	1 X 500 ml P	1 X 4 oz. A			
☐	Dissolved As/Ni/Zn	6010C/6020A	HNO ₃ , 4° C	1 X 500 ml P	1 X 4 oz. A			
☐	Percent Moisture	E245.1/SM2540G	4° C	-	1 X 4 oz. A			
☐								
☐								
☐								
☐								
☐								
☐								
☐								

NOTES/SKETCH

Sampler Signature:

Print Name:

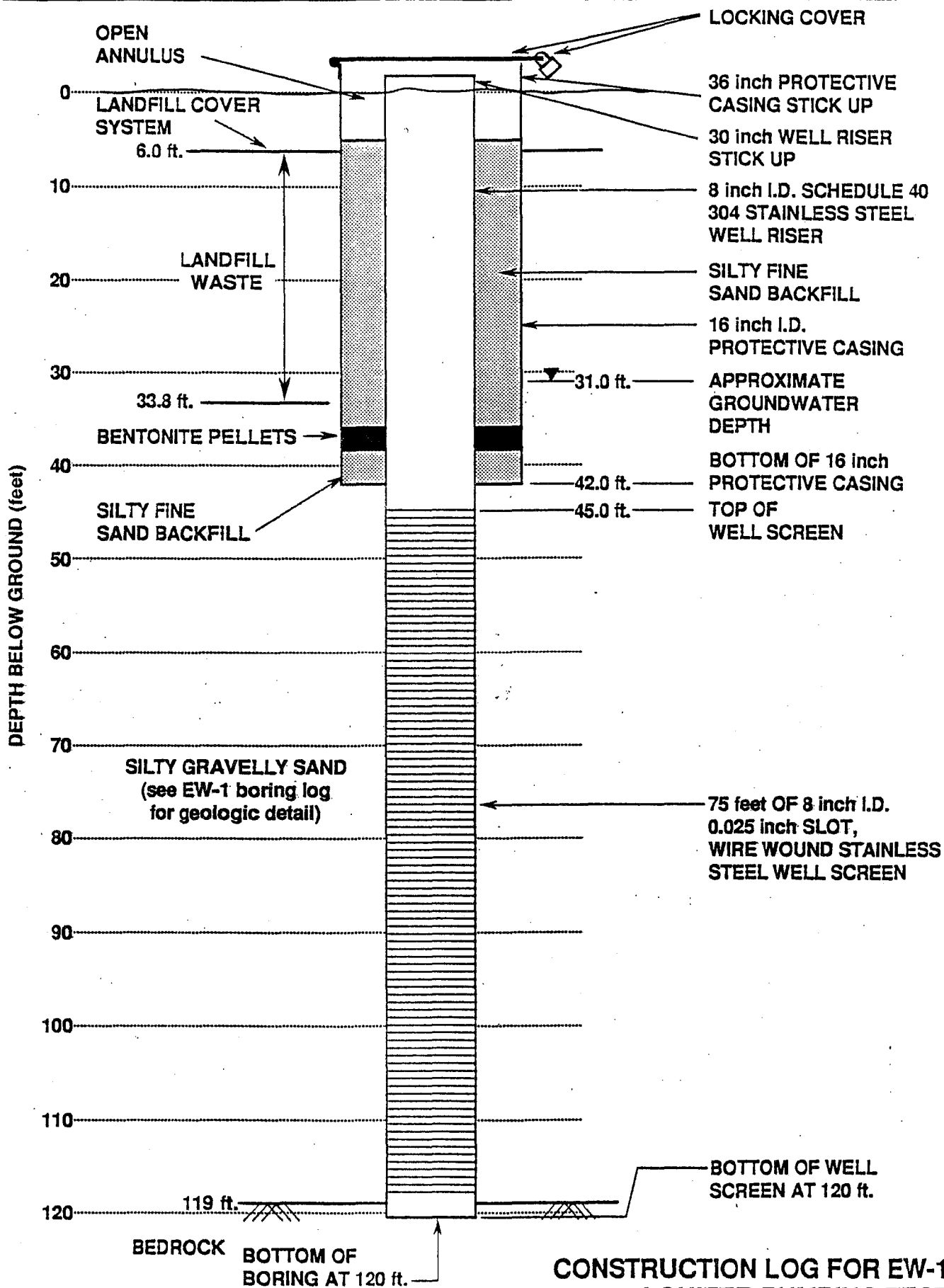
Checked By:

Date:

Winthrop Landfill Post-Closure Monitoring & Maintenance Program Landfill Gas Monitoring Field Data Sheet					Date:			
					Atmospheric Pressure			
Job Number:					Consultant:		Ambient Air Temperature	
Sampler(s):					Start		Finish	
Gas Probe Identifier	Probe Depth (feet)	Sample Time	Methane (% Volume)	Oxygen (% Volume)	CO2 (% Volume)	dP (inches H2O)	Comments	
GP-11	7							
Ambient Air	N/A							
Notes:								
Completed by/Date: _____								
Checked by/Date: _____								

LANDFILL CONDITION/WINDSHIELD DRIVE-BY VISUAL SURVEY FORM WINTHROP LANDFILL POST-CLOSURE MONITORING & MAINTENANCE PROGRAM		
DATE/TIME:	*Contact the Winthrop Code Enforcement Officer to verify if any new construction or	
OBSERVERS:	building permits were issued for tax lots located within the groundwater ordinance protection	
WEATHER:	boundary during the yearly monitoring period (207-377-7200 ext: 427).	
Hubbard Lane Observations:		
Guardrail		
Drainage Swale		
Aggregate Condition		
Roadway Geotextile		
Side Slopes		
Other		
Fence Observations:		
Gate Conditions:		
<u>Mallard Lane Road Gate</u>	Locked	<u>Hubbard Lane Road Gate</u>
	Hinges Oiled	
	Locks Oiled	
<u>MW-9 Gate</u>	Locked	<u>Other Misc.</u>
	Hinges Oiled	
	Locks Oiled	
Hazard Sign Condition and Placement:		
(1 per 50' length)		
Locations Missing		
Total Signs Missing		
Culverts:		
Mallard Lane Road Condition:		
Sphagnum Bog Outlet Condition:		
Landfill Cover and Surface Conditions:		
(Erosion Rills, Depressions, Stained Soil, Ponded Water, Animal Burrows, Settlement, Vegetative Stress, slope condition, etc. See Figure 1 from 2017 Landfill Inspection Report)		
Points of Exposure (POEs):		
(General observations, note any new construction*, staining, foreign debris, etc.)		
Hoyt Brook		
Annabessacook Lake		
Sphagnum Bog		
Cattail Marsh		
Form Completed By: _____		
Form Reviewed By: _____		
Date: _____		

APPENDIX B-3
Well Construction Logs

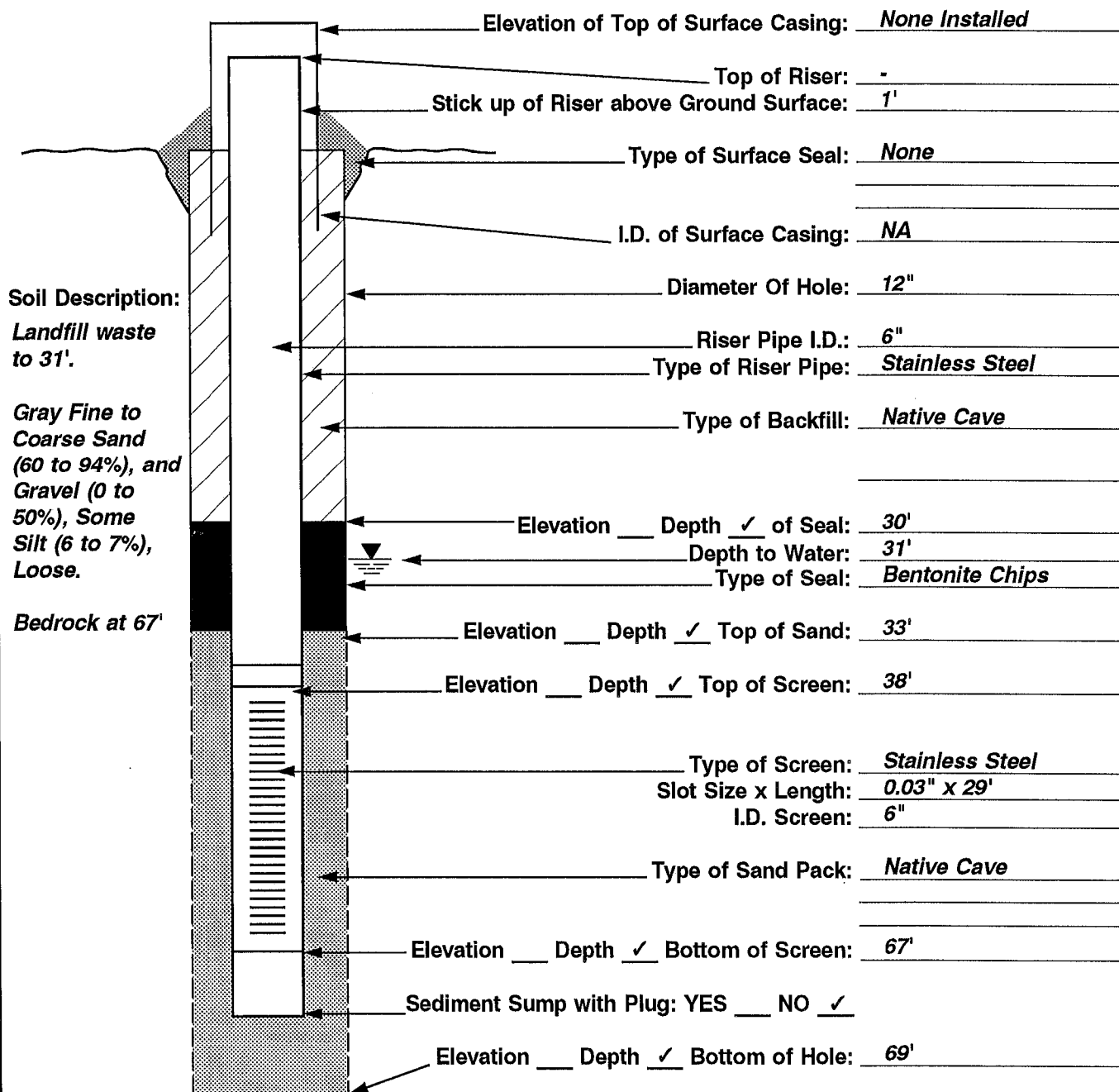


**CONSTRUCTION LOG FOR EW-1
AQUIFER PUMPING TEST
WINTHROP LANDFILL**

E.C.JORDAN CO.

MONITORING WELL CONSTRUCTION DIAGRAM

Project: Winthrop Landfill Location: EW-2 Driller: New Hampshire Boring
 Project No.: 03971-33 Boring: - Drilling Method: Hollow Stem Auger
 Elevation: - Date: 1/4/95 Development Method: Surge & Pump



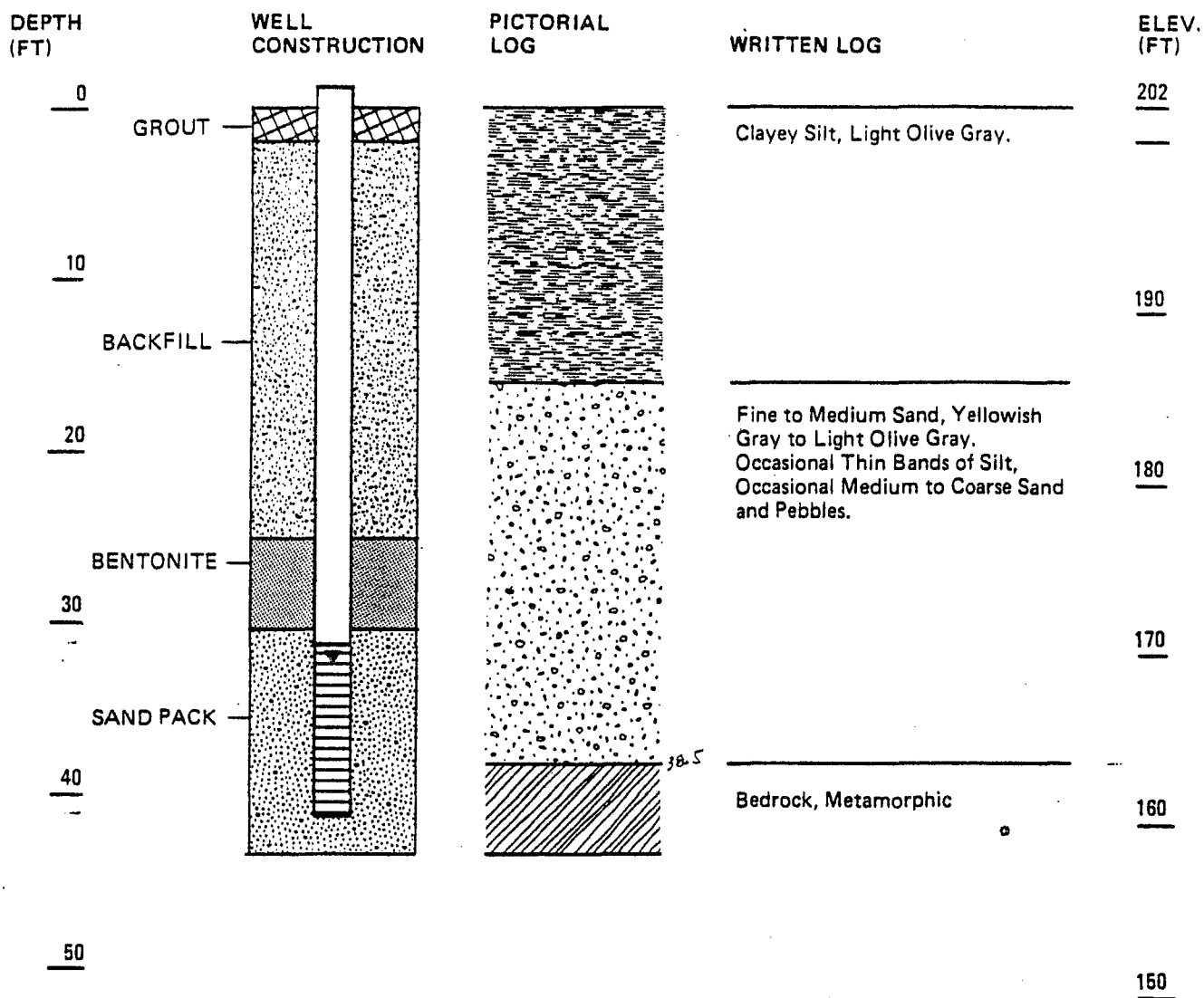


Figure C-8
Summary Pictorial and Written
Log of Well 5A



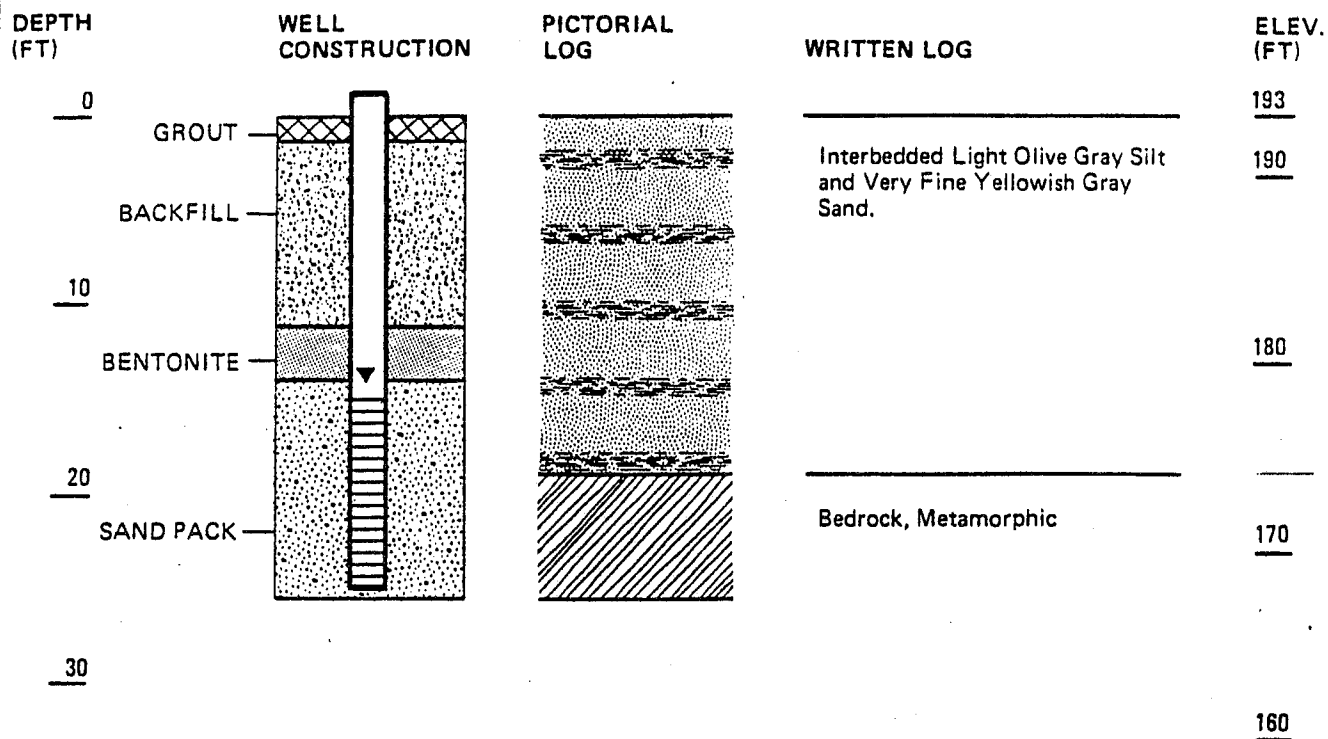


Figure C-10
Summary Pictorial and Written
Log of Well 7A



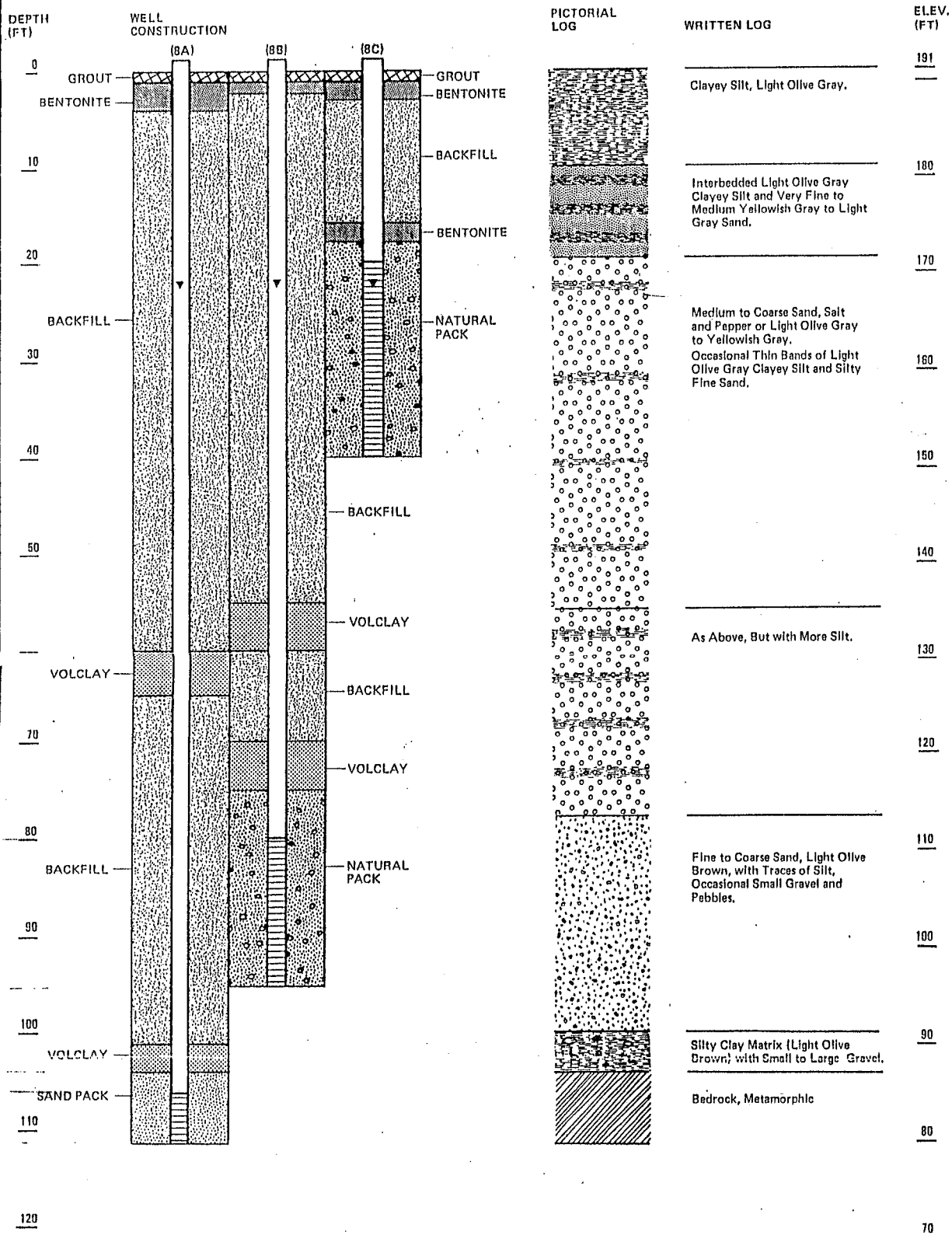
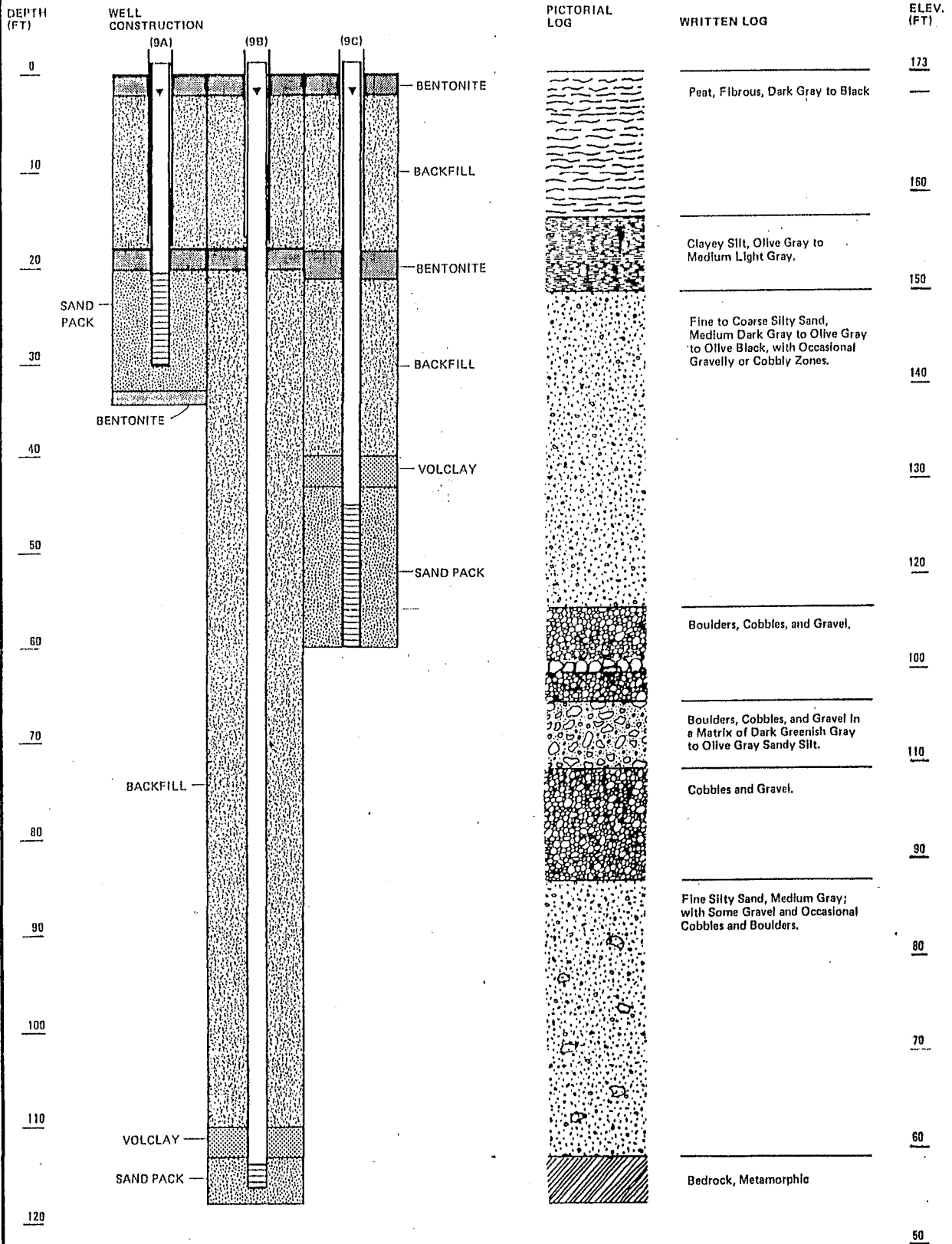


Figure C-11
Summary Pictorial and Written
Log of Wells 8A, B and C





NOTE: Protector pipe for all three wells consists of 20 feet (two 10-foot coupled sections) of galvanized 1/3-inch electrical conduit.

Figure C-12
Summary Pictorial and Written
Log of Wells 9A,B and C



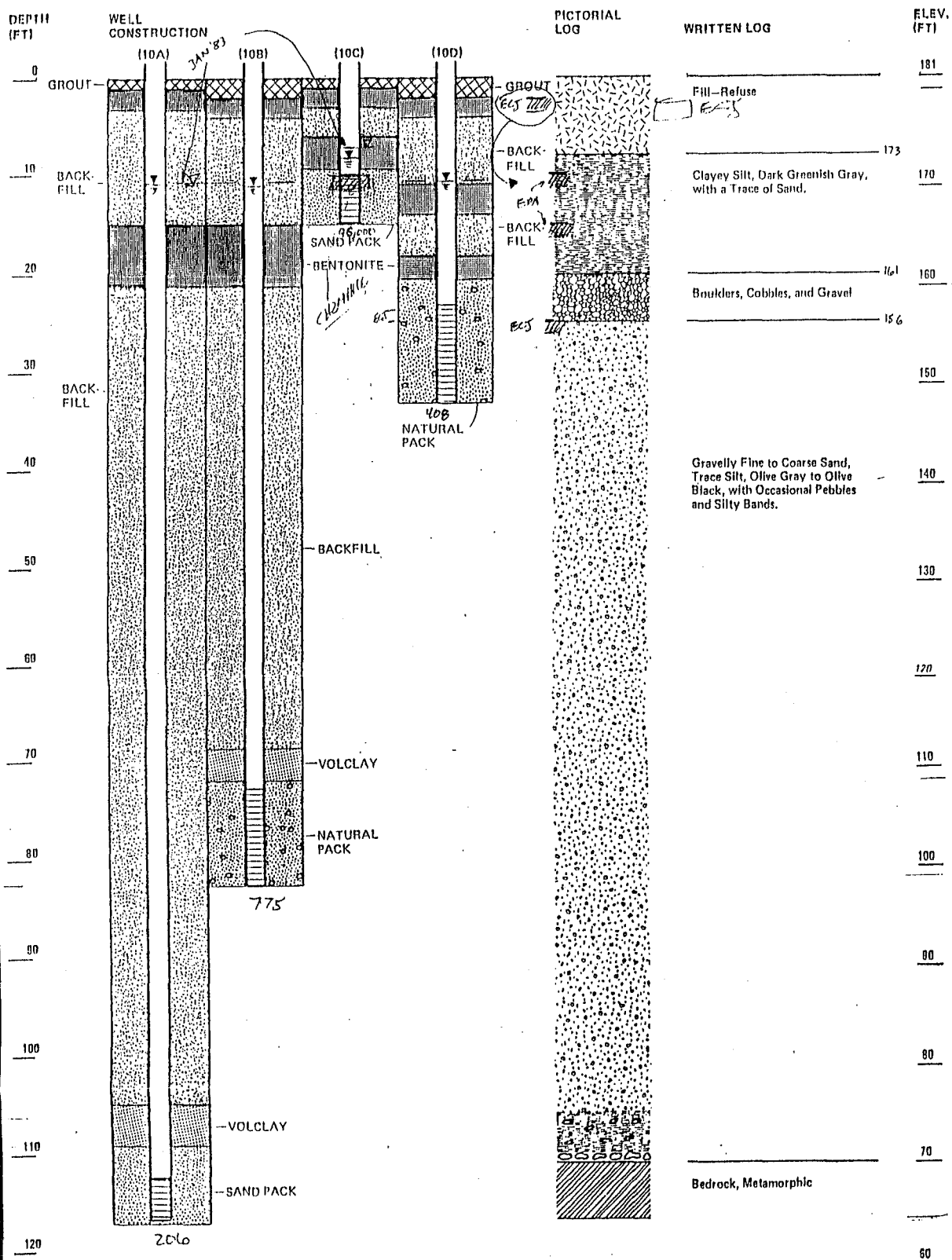


Figure C-13
Summary Pictorial and Written
Log of Wells 10A,B,C and D



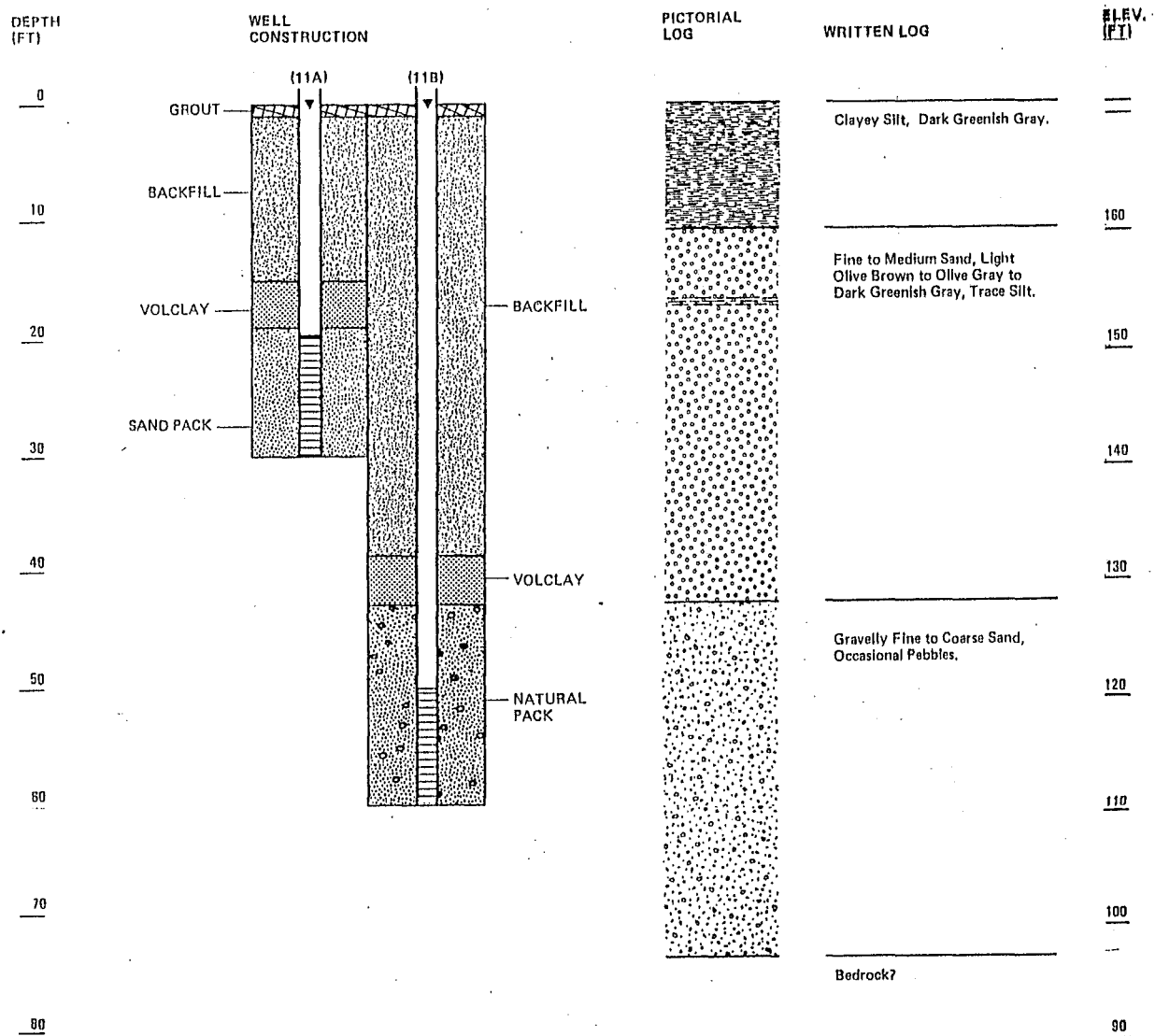


Figure C-14
Summary Pictorial and Written
Log of Wells 11A and B



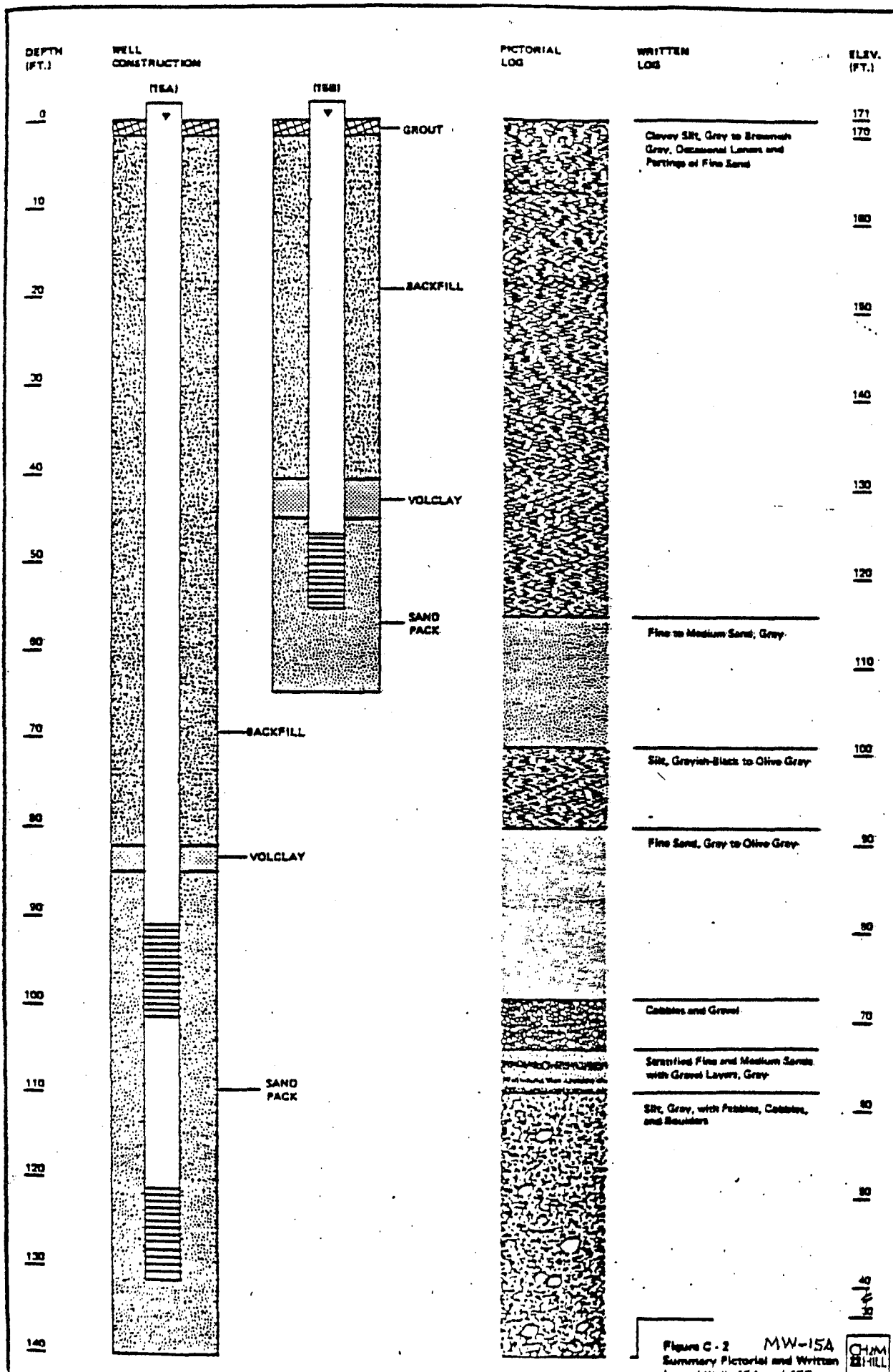


Figure C-2 MW-15A
Summary Pictorial and Written
Log of Wells 15A and 15B

CHM
BI-HILL

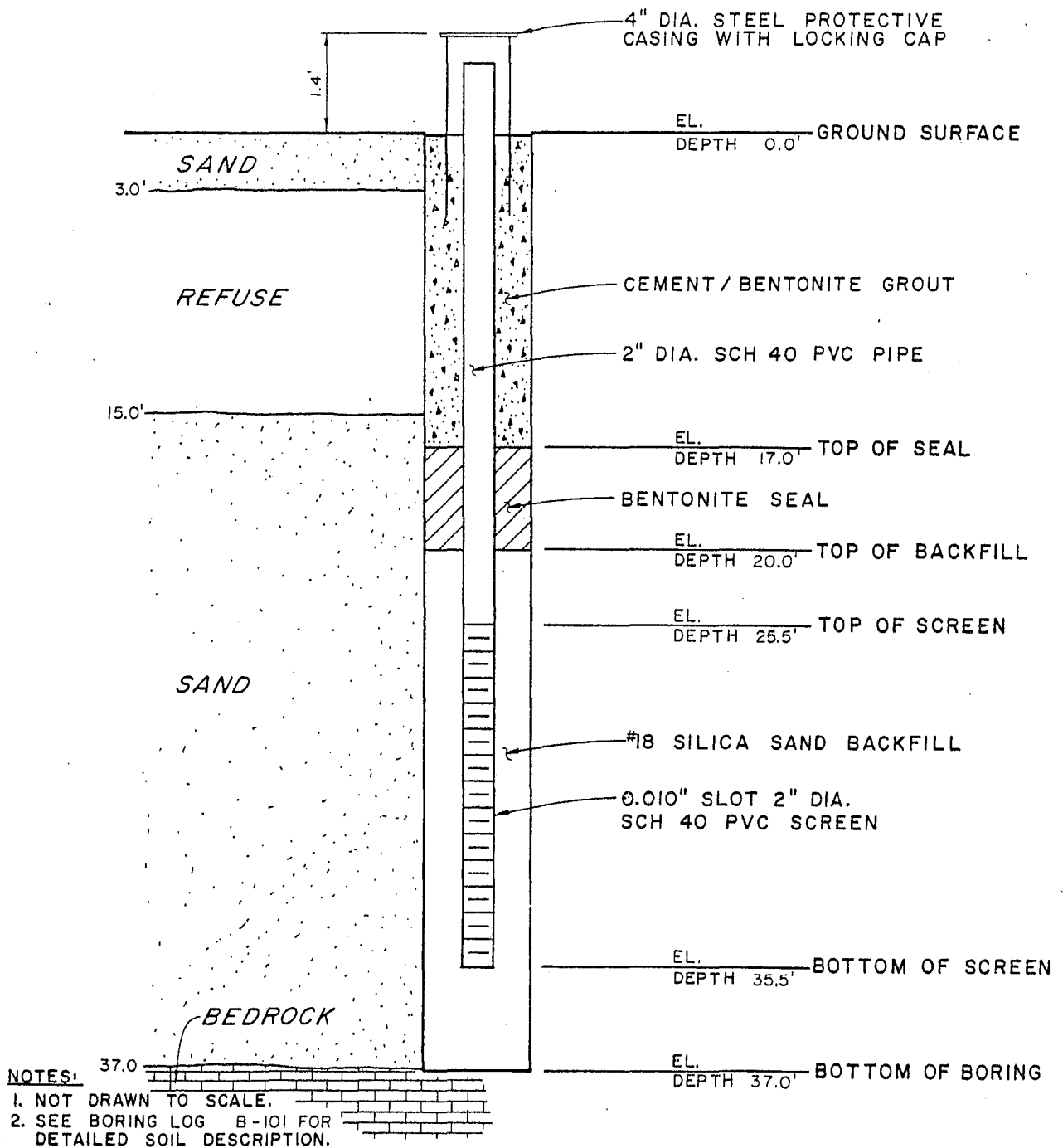
Observation Well Details

PROJECT No. CH 85-031

WELL No. MW-101

PROJECT NAME UNITED TECHNOLOGIES INMONT/WINTHROP SITE

BORING LOCATION _____ DATE 10-18-85 BY KMB



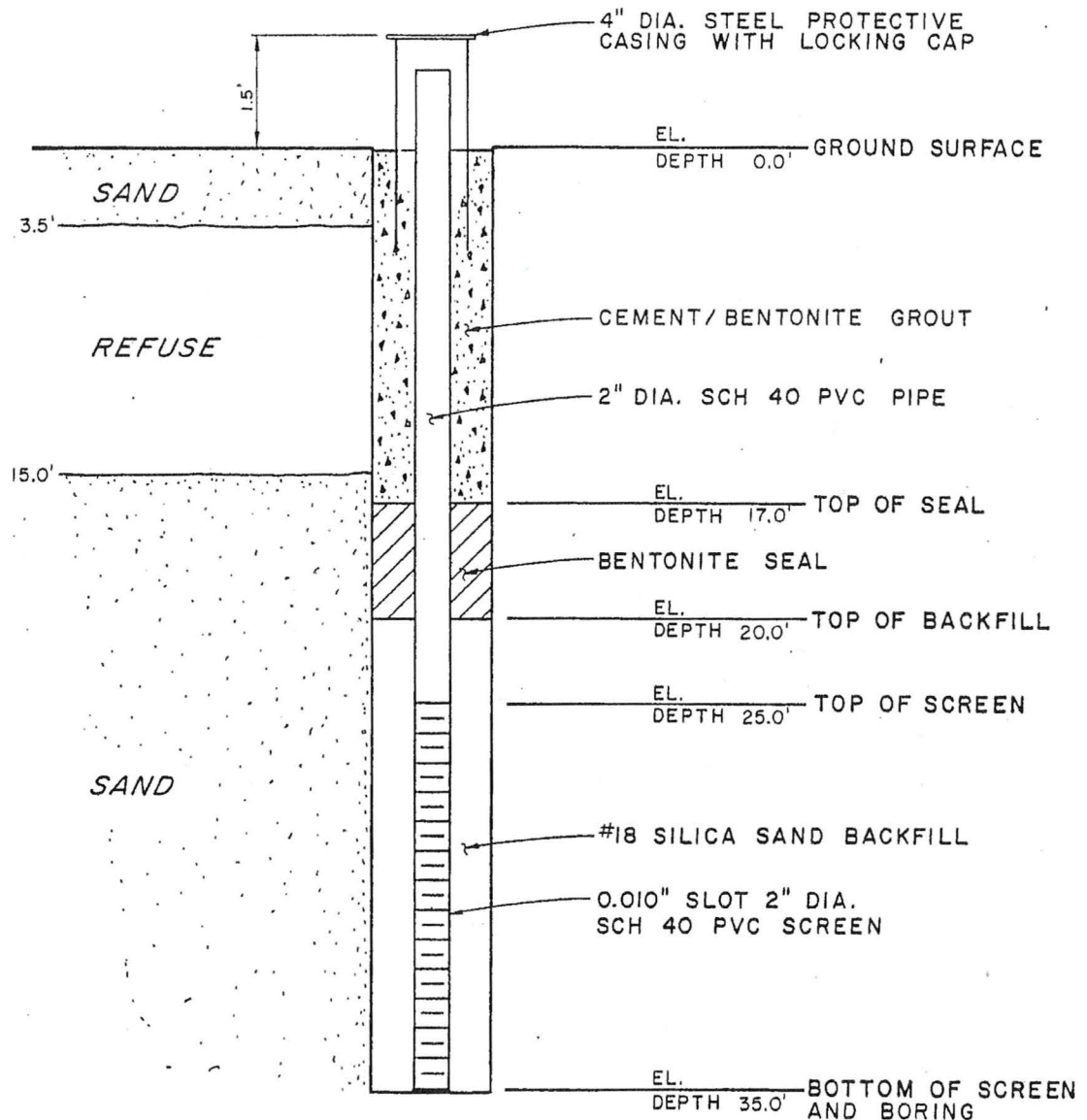
Observation Well Details

PROJECT No. CH 85-031

WELL No. MW-103

PROJECT NAME UNITED TECHNOLOGIES INMONT/WINTHROP SITE

BORING LOCATION _____ DATE 10-22-85 BY KMB



NOTES:

1. NOT DRAWN TO SCALE.
2. SEE BORING LOG B-103 FOR DETAILED SOIL DESCRIPTION.

*Note: construction log for
MW-103R could not be located.
This log provided for reference.
6/29/18 RUB*

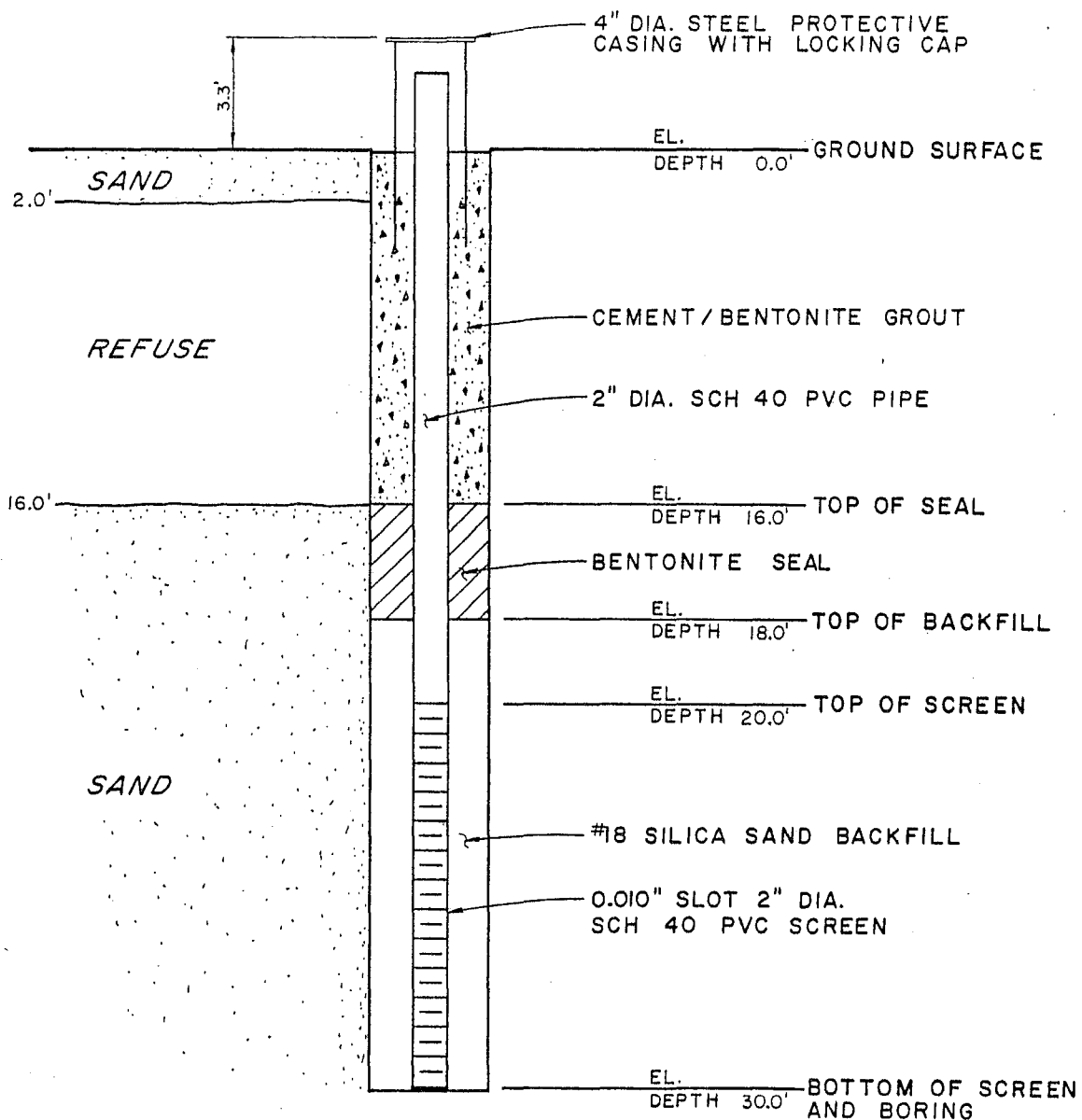
Observation Well Details

PROJECT No. CH 85-031

WELL No. MW-106

PROJECT NAME UNITED TECHNOLOGIES INMONT/WINTHROP SITE

BORING LOCATION _____ DATE 10-24-85 BY JPI



NOTES:

1. NOT DRAWN TO SCALE.
2. SEE BORING LOG B-106 FOR DETAILED SOIL DESCRIPTION.

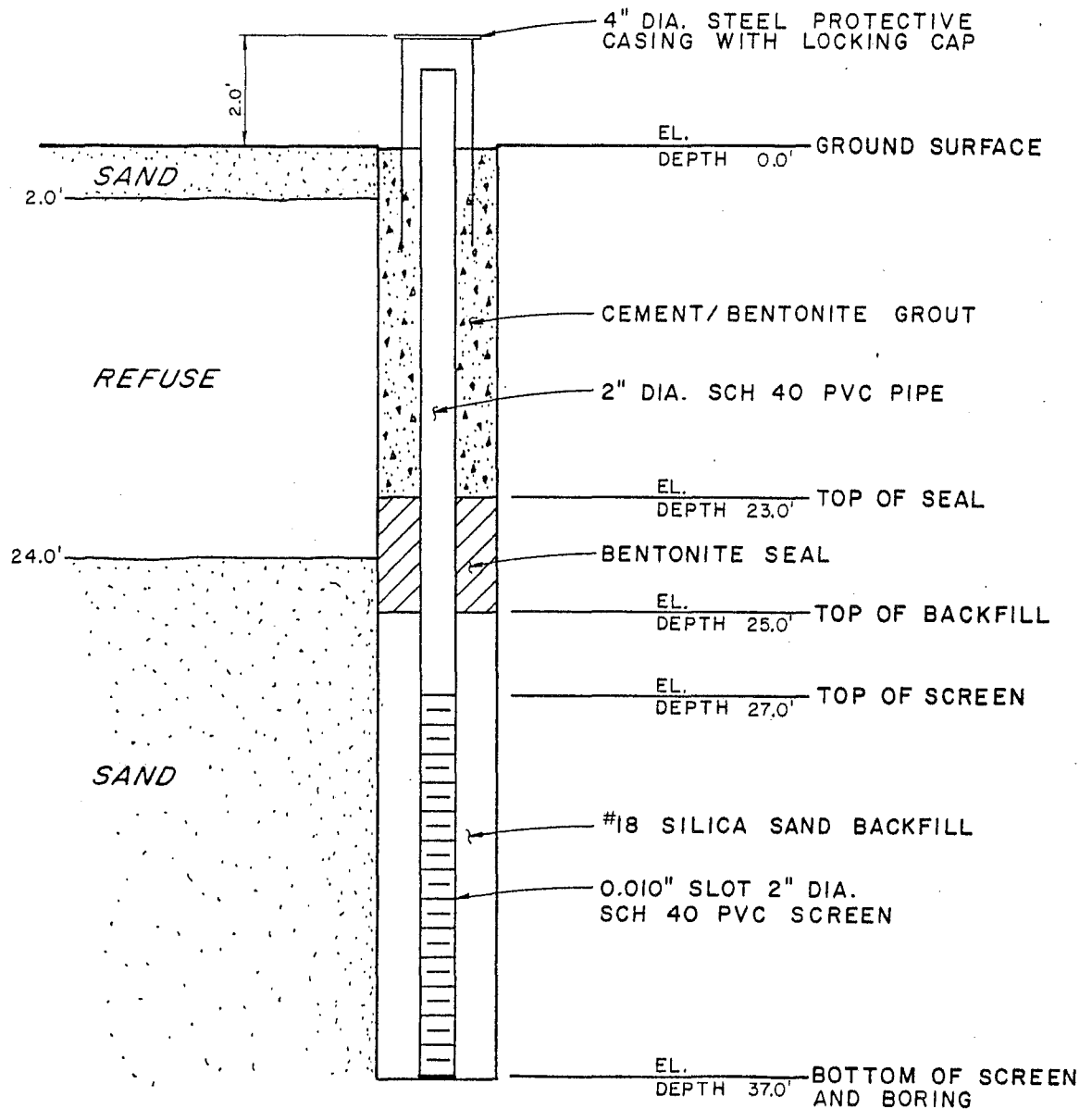
Observation Well Details

PROJECT No. CH 85-031

WELL No. MW-108

PROJECT NAME UNITED TECHNOLOGIES INMONT/WINTHROP SITE

BORING LOCATION _____ DATE 10-29-85 BY KMB



NOTES:

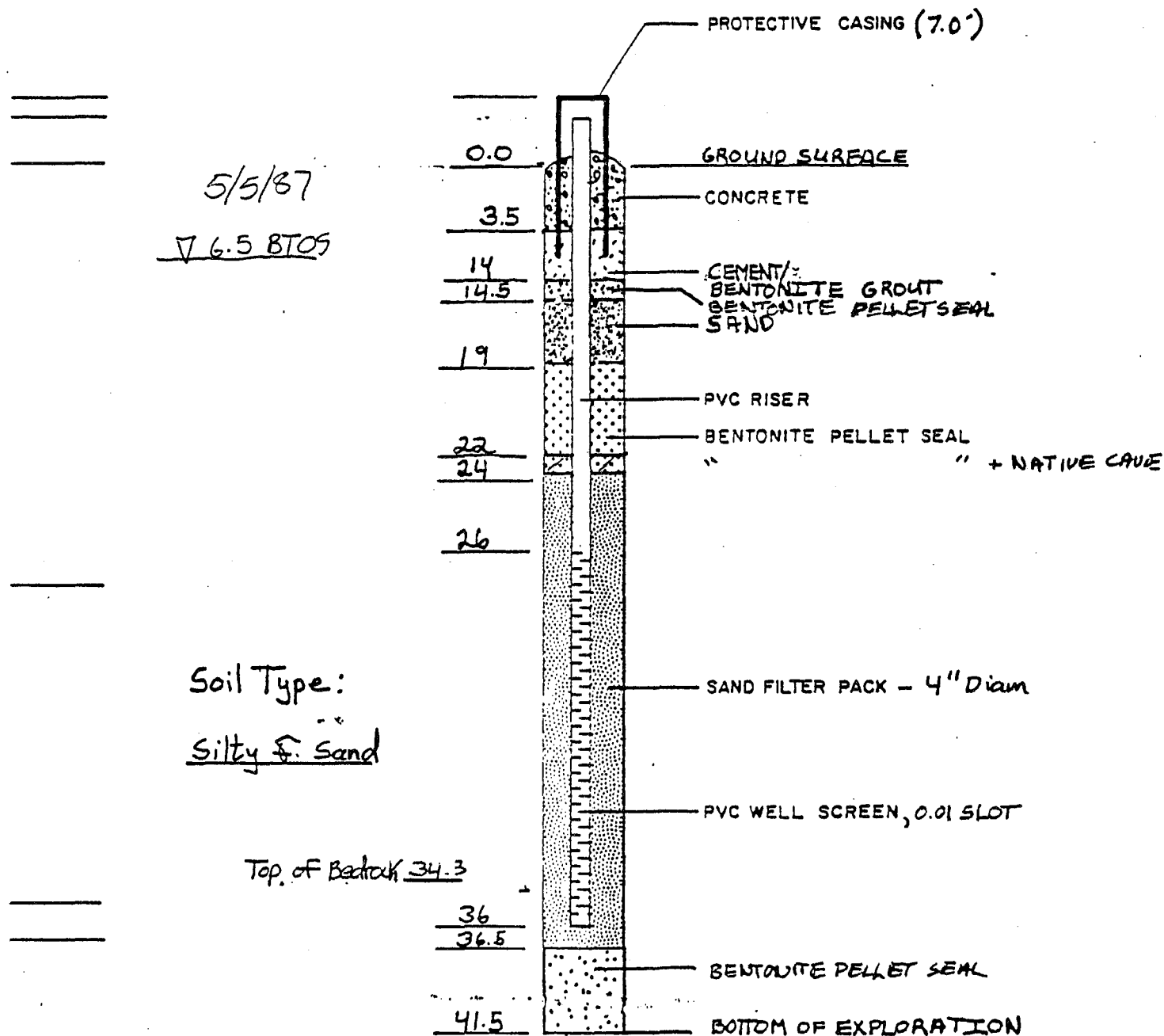
1. NOT DRAWN TO SCALE.
2. SEE BORING LOG B-108 FOR DETAILED SOIL DESCRIPTION.

MONITORING WELL INSTALLATION DIAGRAM

PROJECT NO. 3971-63	PROJECT NAME <i>Winthrop Landfill Hydrogeologic Investigation</i>	WELL NO. MW- 205A
INSTALLED BY: <i>Guild Drilling Co., Inc.</i> INSPECTED BY: L. FITZGERALD	DATE INSTALLED 4/4/88	BORING DIAMETER ☐ 6" ☐ 5" ☒ 4" ☐ 3"
WELL DIAMETER 2" I.D.	WELL MATERIAL Sch. 80 PVC	Drilling Method: Drive + Wash Casing

ELEV., FT.

DEPTH, FT.

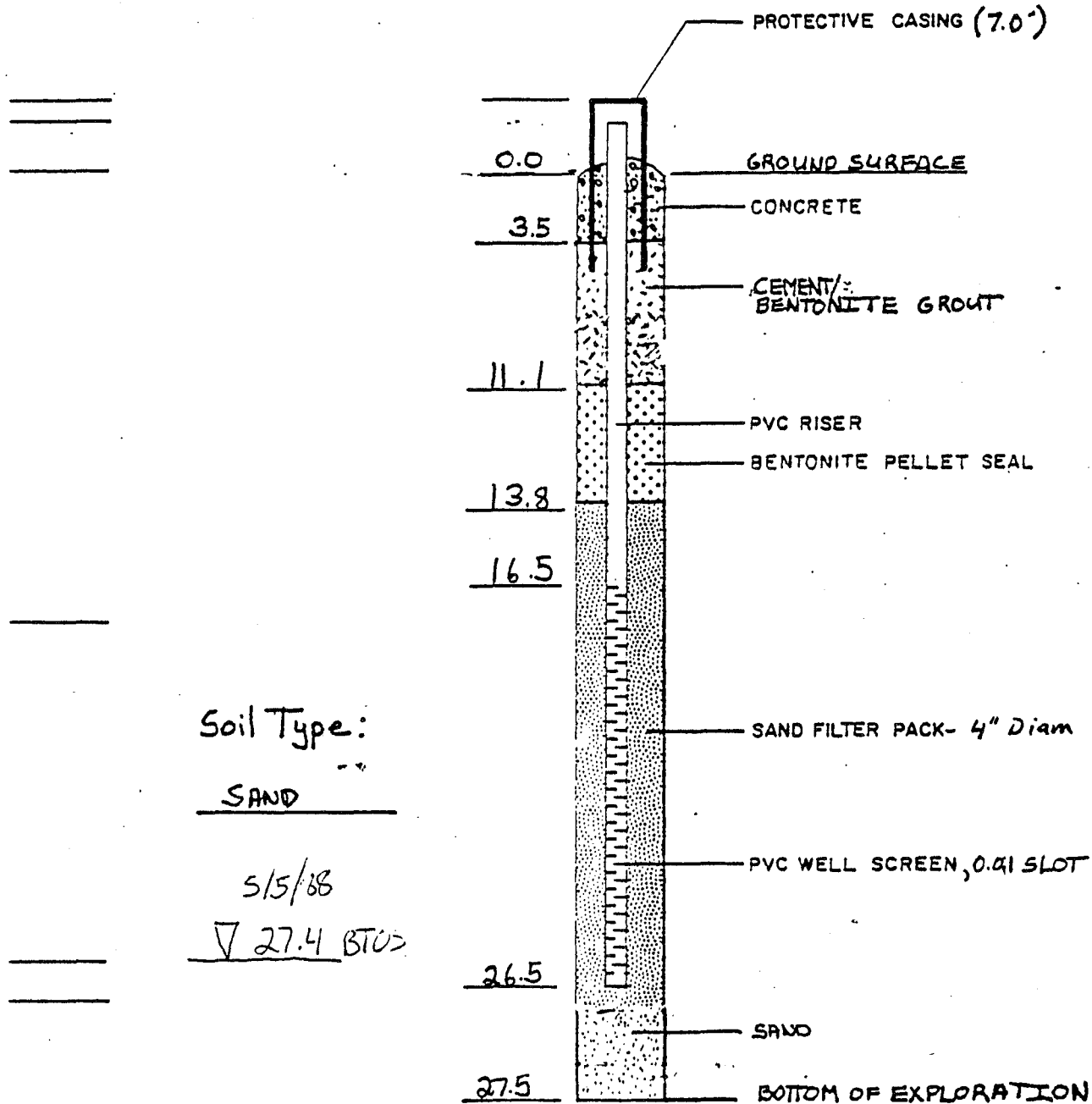


MONITORING WELL INSTALLATION DIAGRAM

PROJECT NO. 3971-63	PROJECT NAME <i>Winthrop Landfill</i> <i>Hydrogeologic Investigation</i>	WELL NO. MW- 207A
INSTALLED BY: <i>Guild Drilling Co., Inc.</i> INSPECTED BY: <i>J. Rand</i>	DATE INSTALLED 3/21/88	BORING DIAMETER ☐ 6" ☐ 5" ☒ 4" ☐ 3"
WELL DIAMETER 2" I.D.	WELL MATERIAL Sch. 80 PVC	Drilling Method: Drive + Wash Casing

ELEV., FT.

DEPTH, FT.

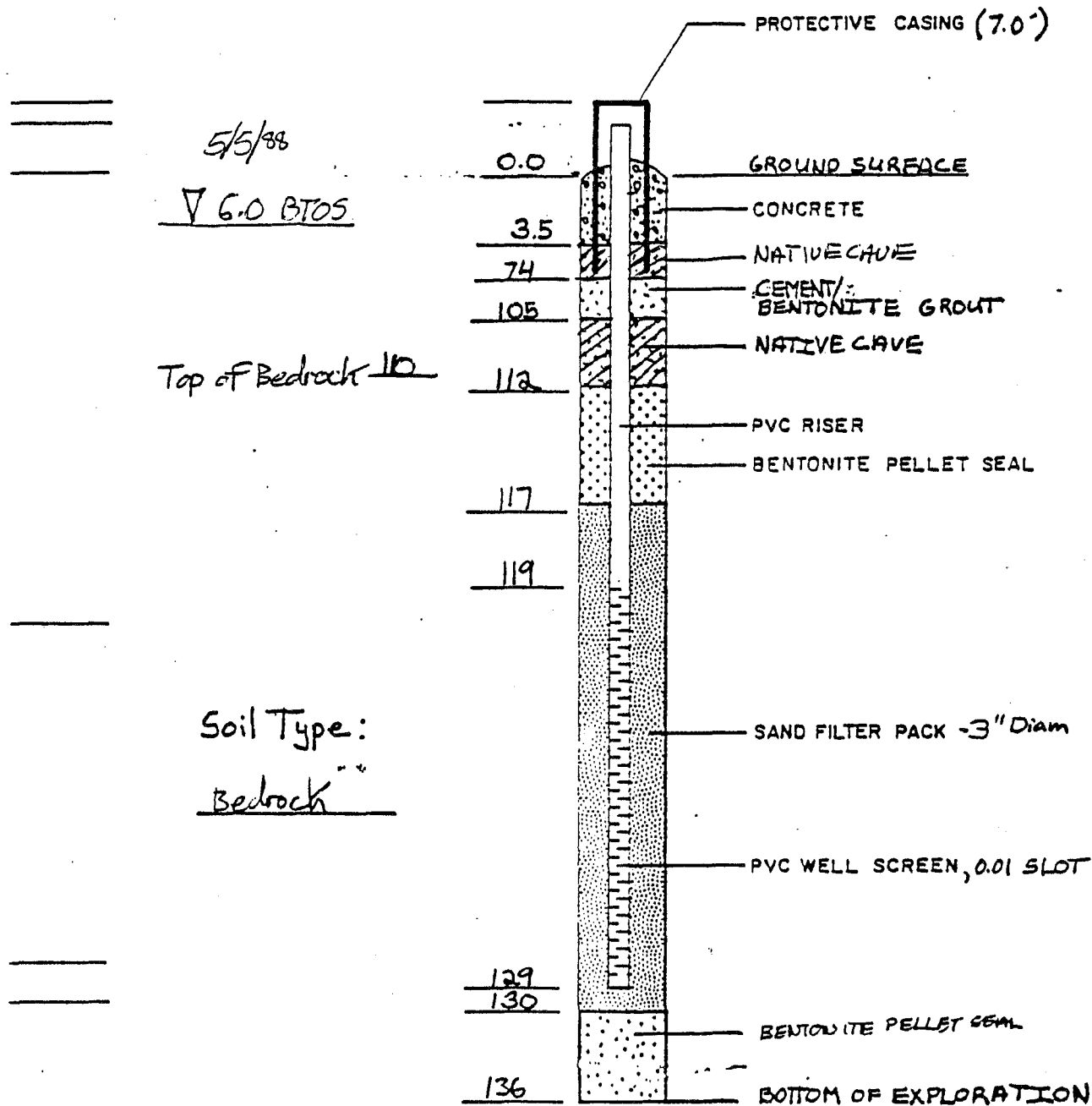


MONITORING WELL INSTALLATION DIAGRAM

PROJECT NO. 3971-63	PROJECT NAME <i>Winthrop Landfill Hydrogeologic Investigation</i>	WELL NO. MW-208A
INSTALLED BY: <i>Guild Drilling Co., Inc.</i> INSPECTED BY:	DATE INSTALLED 4/20/88	BORING DIAMETER 6" 5" 4" 3"
WELL DIAMETER 2" I.D.	WELL MATERIAL Sch. 80 PVC	Drilling Method: Drive + Wash Casing

ELEV., FT.

DEPTH, FT.

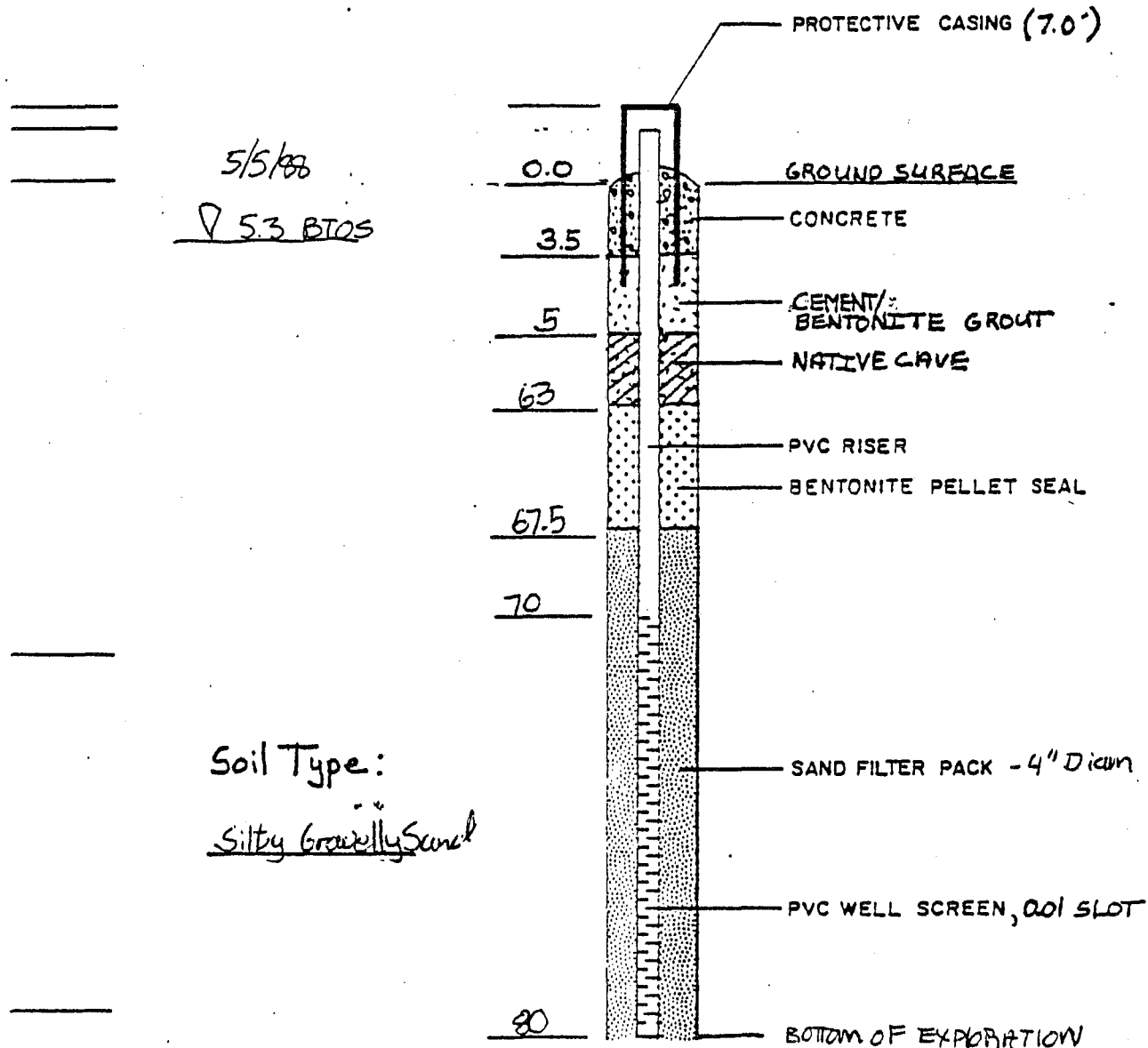


MONITORING WELL INSTALLATION DIAGRAM

PROJECT NO. 3971-63	PROJECT NAME <i>Winthrop Landfill Hydrogeologic Investigation</i>	WELL NO. MW- 208.B
INSTALLED BY: <i>Guild Drilling Co., Inc.</i> INSPECTED BY: <i>J. RAND</i>	DATE INSTALLED 4/25	BORING DIAMETER ☐ 6" ☒ 5" ☒ 4" ☐ 3"
WELL DIAMETER 2" I.D.	WELL MATERIAL Sch. 80 PVC	Drilling Method: Drive + Wash Casing

ELEV., FT.

DEPTH, FT.

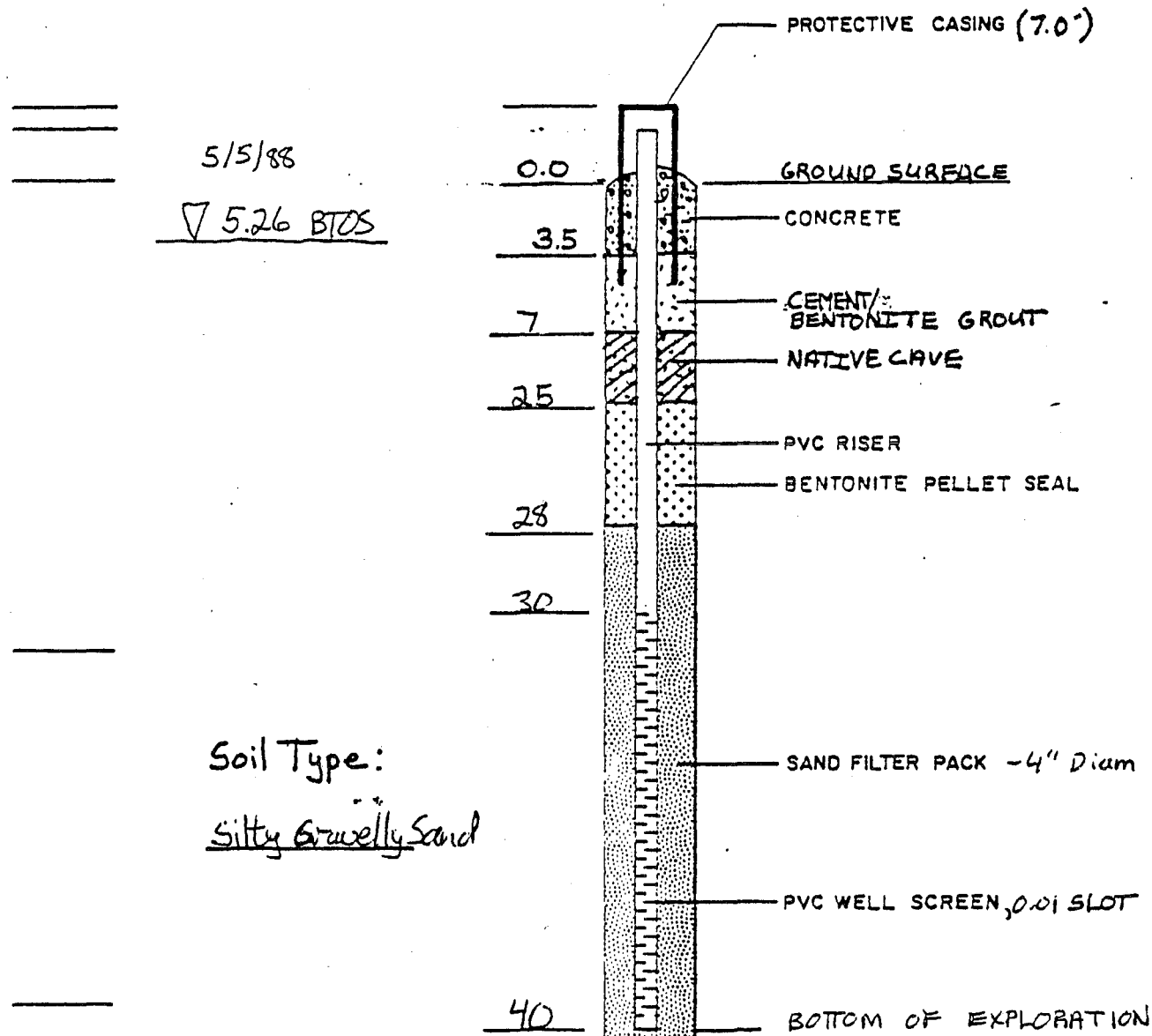


MONITORING WELL INSTALLATION DIAGRAM

PROJECT NO. 3971-63	PROJECT NAME <i>Winthrop Landfill Hydrogeologic Investigation</i>	WELL NO. MW- 208C
INSTALLED BY: <i>Guild Drilling Co., Inc.</i> INSPECTED BY: <i>J. Rand</i>	DATE INSTALLED 4/26/88	BORING DIAMETER ☐ 6" ☐ 5" ☒ 4" ☐ 3"
WELL DIAMETER 2" I.D.	WELL MATERIAL Sch. 80 PVC	Drilling Method: Drive + Wash Casing

ELEV., FT.

DEPTH, FT.

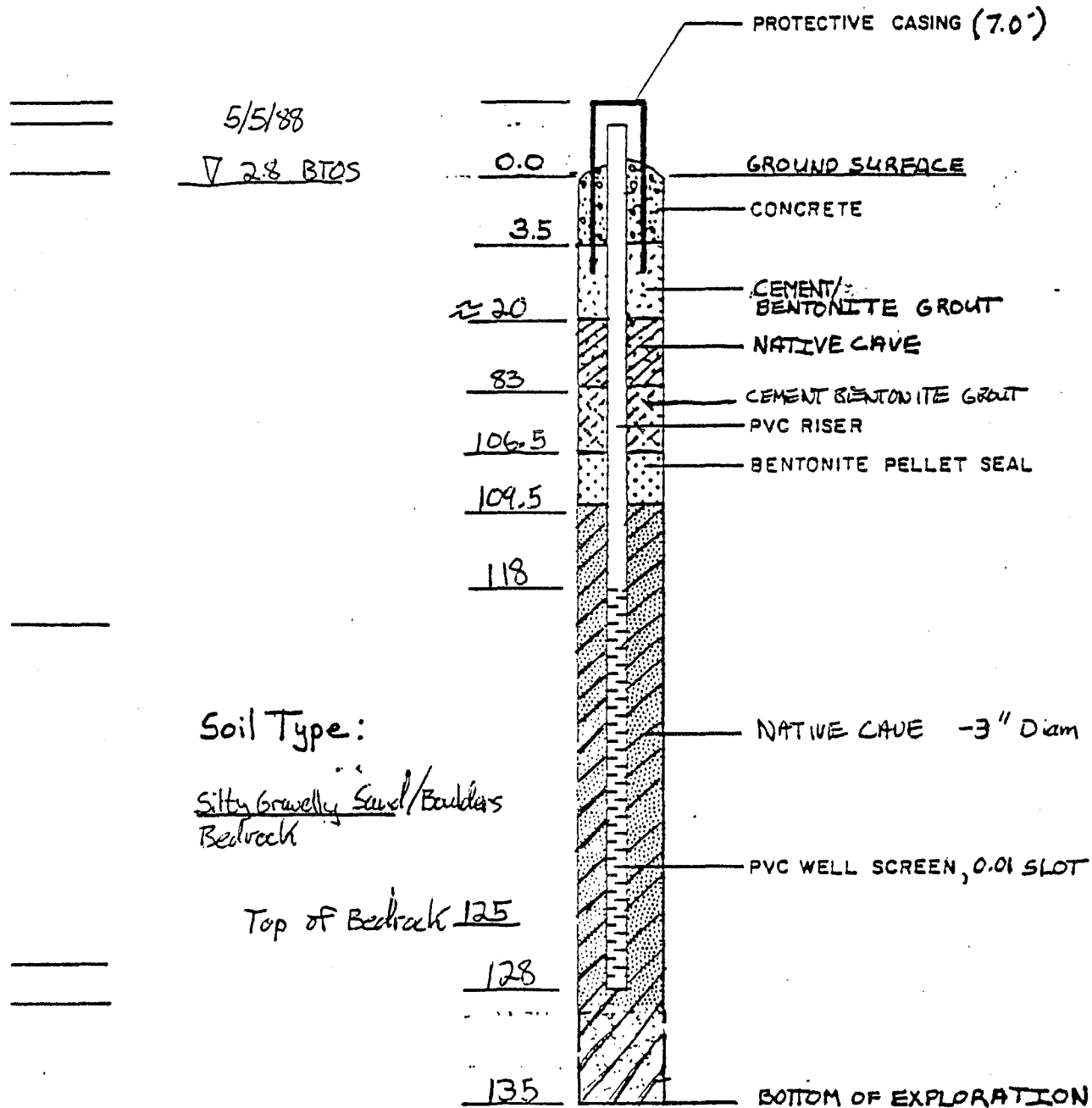


MONITORING WELL INSTALLATION DIAGRAM

PROJECT NO. 3971-63	PROJECT NAME <i>Winthrop Landfill Hydrogeologic Investigation</i>	WELL NO. MW- 210A
INSTALLED BY: <i>Guild Drilling Co., Inc.</i> INSPECTED BY: <i>J. Rand</i>	DATE INSTALLED 12/3/87	BORING DIAMETER ☐ 6" ☒ 5" ☒ 4" ☒ 3"
WELL DIAMETER 2" I.D.	WELL MATERIAL Sch. 80 PVC	Drilling Method: Drive + Wash Casing

ELEV., FT.

DEPTH, FT.

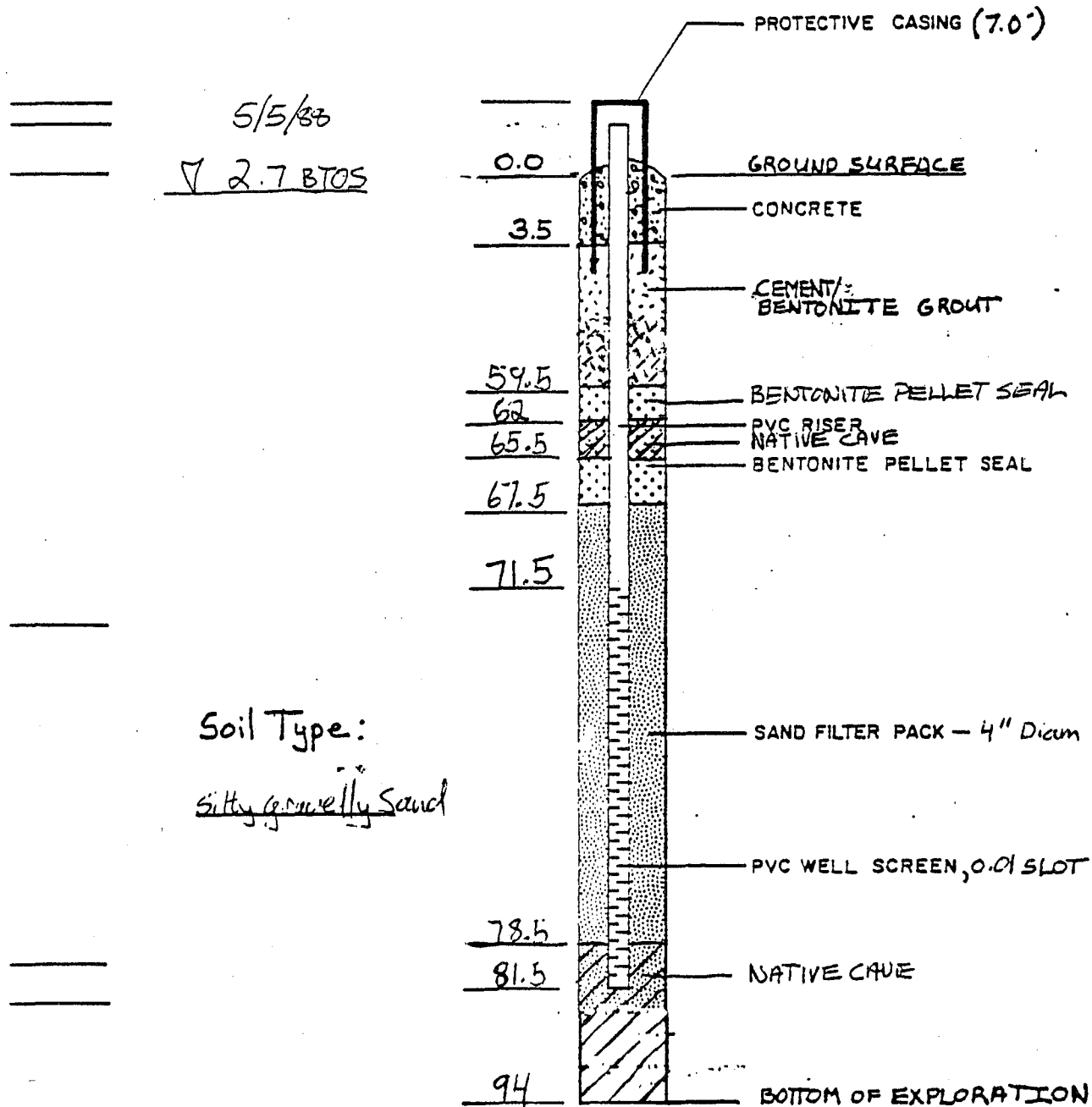


MONITORING WELL INSTALLATION DIAGRAM

PROJECT NO. 3971-63	PROJECT NAME <i>Winthrop Landfill Hydrogeologic Investigation</i>	WELL NO. MW-210B
INSTALLED BY: <i>Guild Drilling Co., Inc.</i> INSPECTED BY: <i>B. METZGER</i>	DATE INSTALLED 12/3/87	BORING DIAMETER ☐ 6" ☒ 5" ☒ 4" ☒ 3"
WELL DIAMETER 2" I.D.	WELL MATERIAL Sch. 80 PVC	Drilling Method: Drive + Wash Casing

ELEV., FT.

DEPTH, FT.

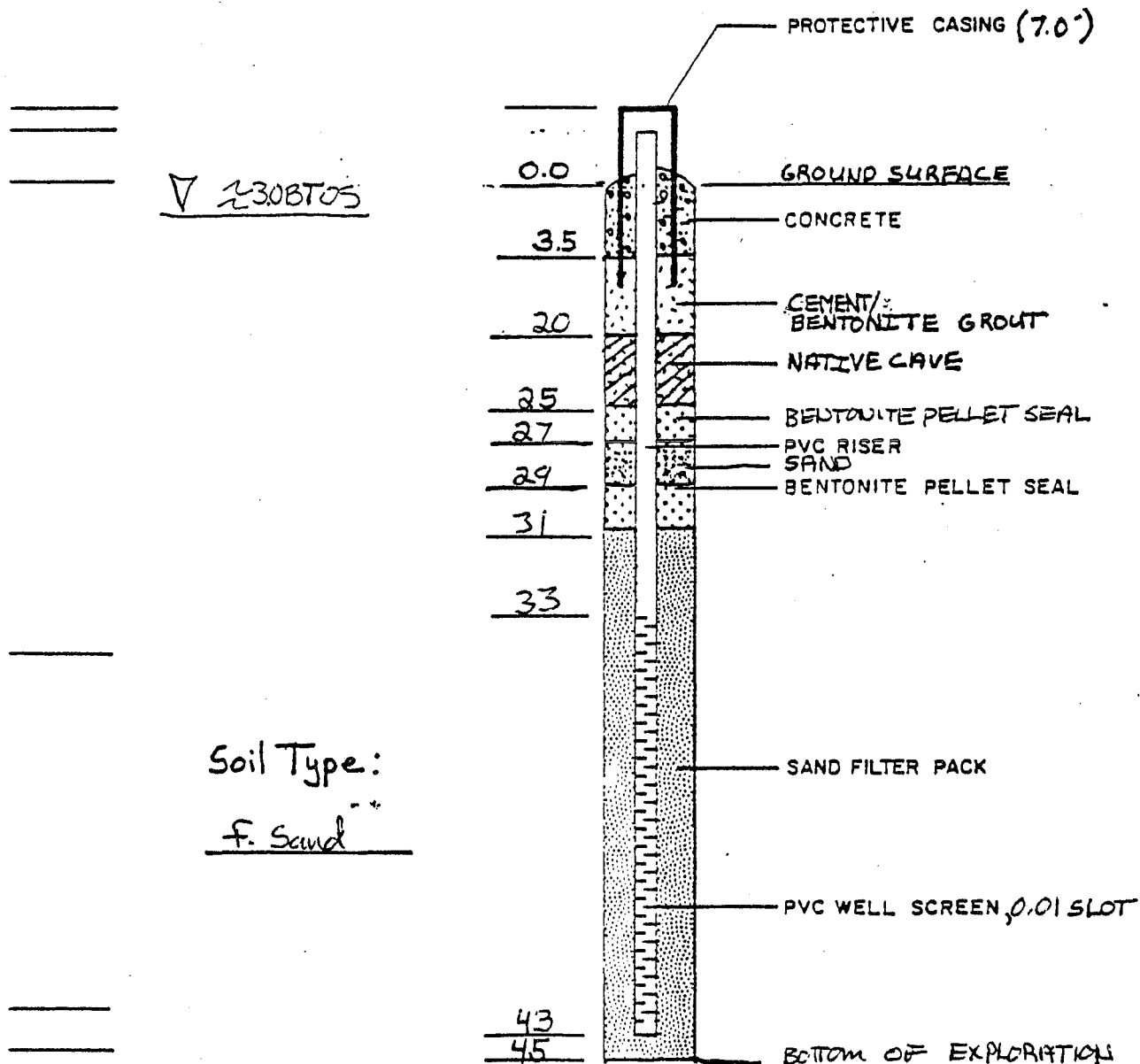


MONITORING WELL INSTALLATION DIAGRAM

PROJECT NO. 3971-63	PROJECT NAME <i>Winthrop Landfill</i> <i>Hydrogeologic Investigation</i>	WELL NO. MW- 210 C
INSTALLED BY: <i>Guild Drilling Co., Inc.</i> INSPECTED BY: <i>B. METZGER</i>	DATE INSTALLED 12/18/88	BORING DIAMETER ☐ 6" ☐ 5" ☒ 4" ☐ 3"
WELL DIAMETER 2" I.D.	WELL MATERIAL Sch. 80 PVC	Drilling Method: Drive + Wash Casing

ELEV., FT.

DEPTH, FT.

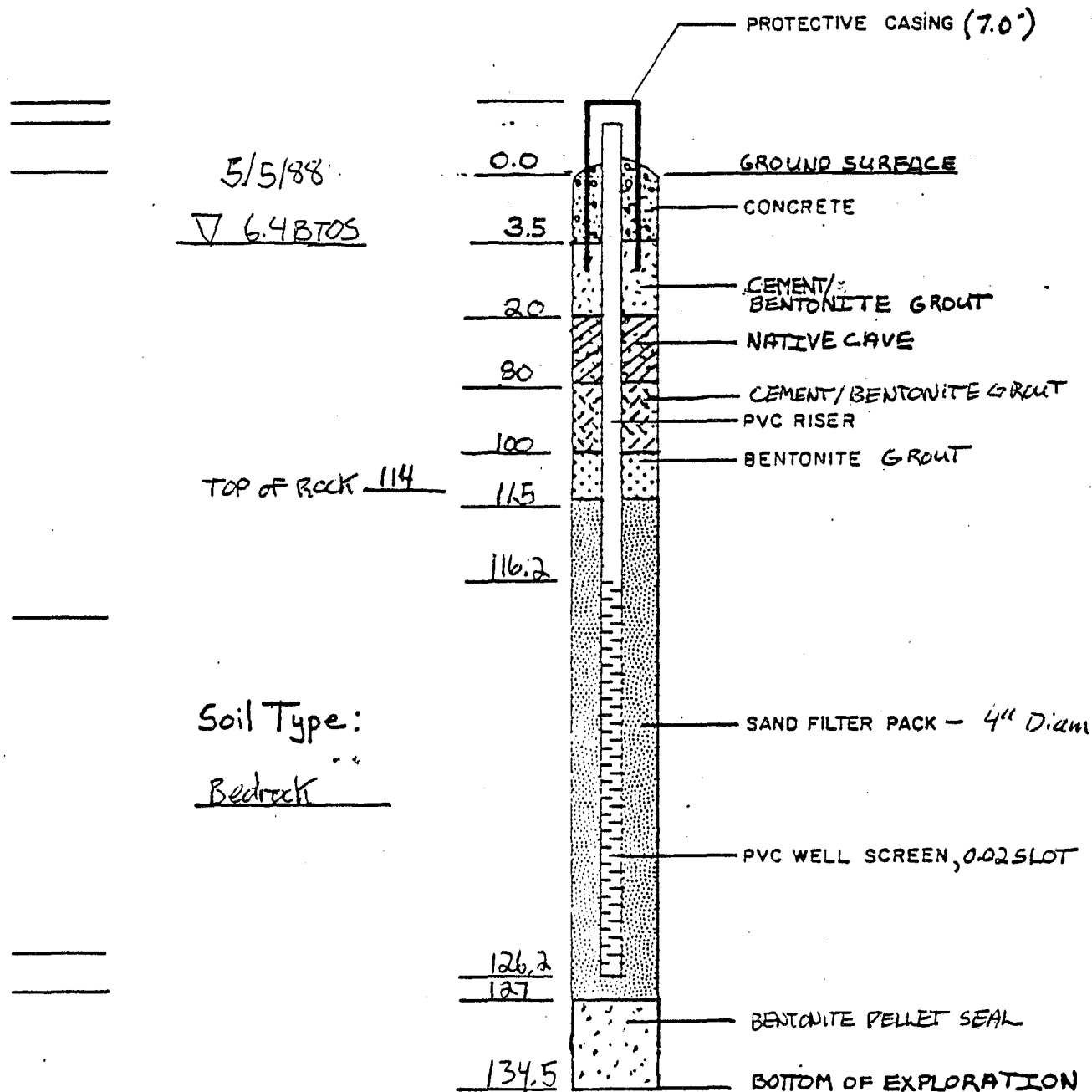


MONITORING WELL INSTALLATION DIAGRAM

PROJECT NO. 3971-63	PROJECT NAME <i>Winthrop Landfill Hydrogeologic Investigation</i>	WELL NO. MW- 212A
INSTALLED BY: <i>Guild Drilling Co., Inc.</i> INSPECTED BY: <i>L. FITZGERALD</i>	DATE INSTALLED 4/20/88	BORING DIAMETER 6" 5" 4" 3"
WELL DIAMETER 2" I.D.	WELL MATERIAL Sch. 80 PVC	Drilling Method: Drive + Wash Casing

ELEV., FT.

DEPTH, FT.

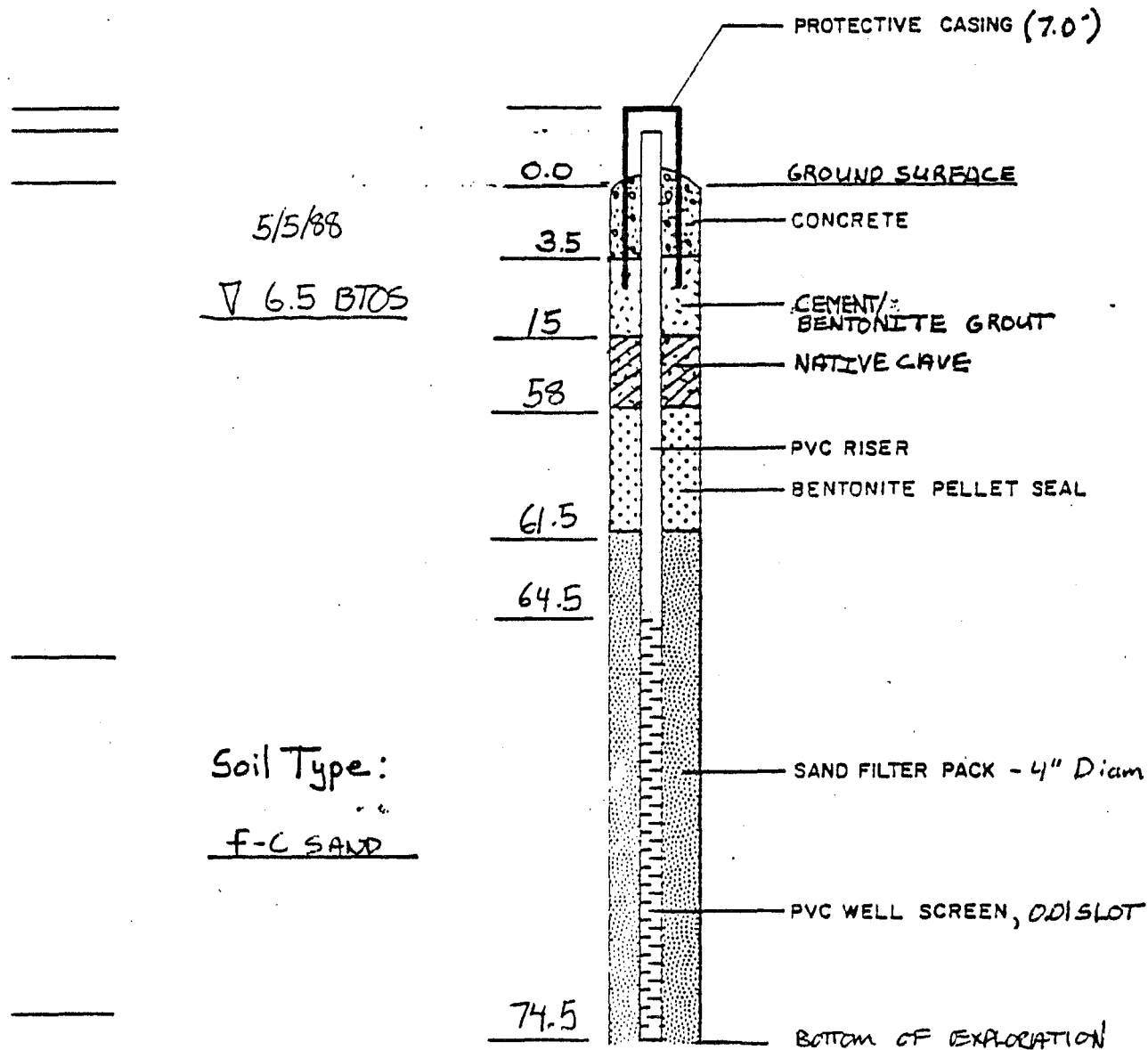


MONITORING WELL INSTALLATION DIAGRAM

PROJECT NO. 3971-63	PROJECT NAME <i>Winthrop Landfill</i> <i>Hydrogeologic Investigation</i>	WELL NO. MW- 212B
INSTALLED BY: <i>Guild Drilling Co., Inc.</i> INSPECTED BY: <i>B. METZGER</i>	DATE INSTALLED <i>4/26/88</i>	BORING DIAMETER ☐ 6" ☒ 5" ☒ 4" ☐ 3"
WELL DIAMETER 2" I.D.	WELL MATERIAL Sch. 80 PVC	Drilling Method: Drive + Wash Casing

ELEV., FT.

DEPTH, FT.

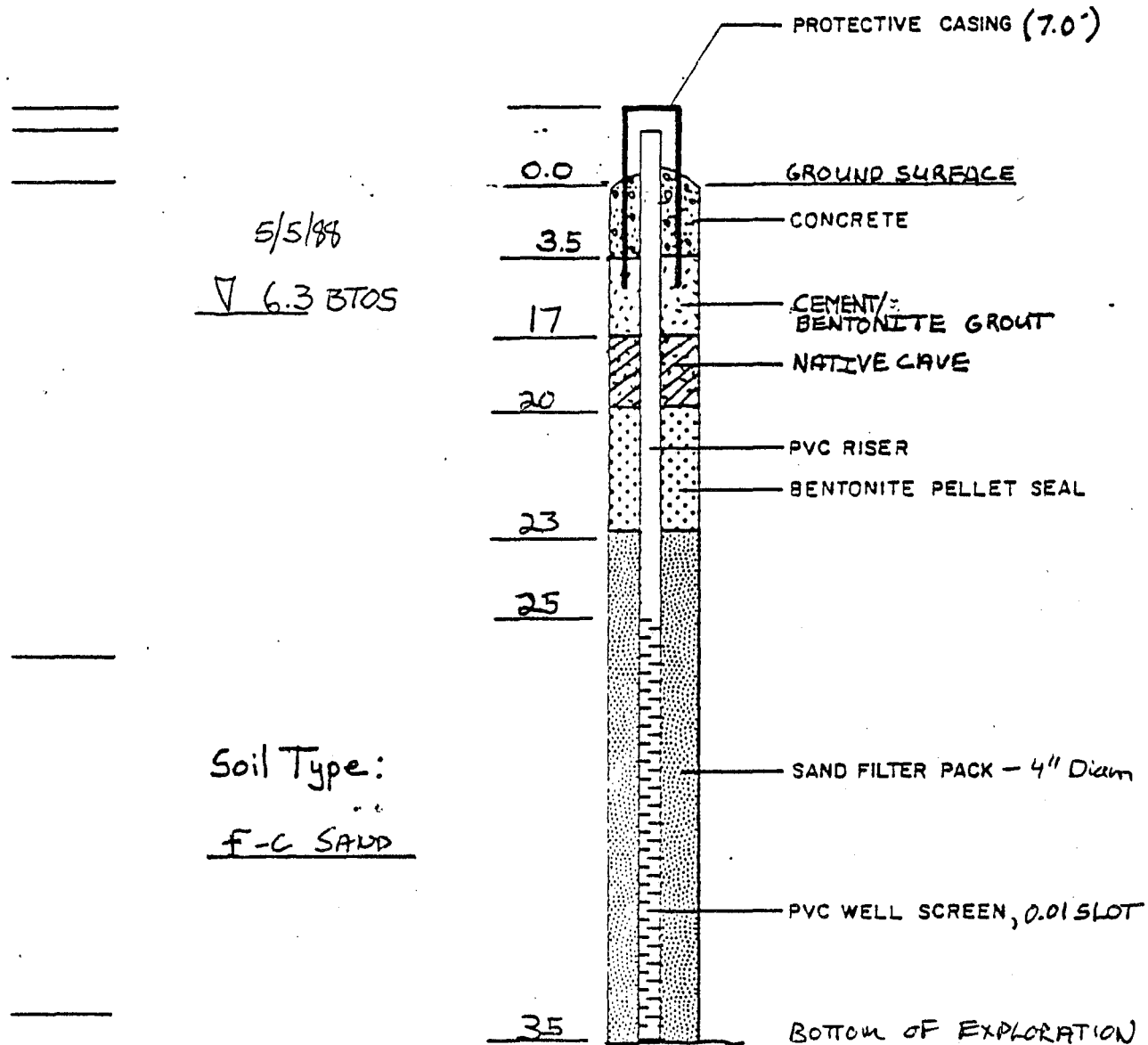


MONITORING WELL INSTALLATION DIAGRAM

PROJECT NO. 3971-63	PROJECT NAME <i>Winthrop Landfill Hydrogeologic Investigation</i>	WELL NO. MW- 212C
INSTALLED BY: <i>Guild Drilling Co., Inc.</i> INSPECTED BY: <i>B. METZGER</i>	DATE INSTALLED 4/27/88	BORING DIAMETER ☐ 6" ☐ 5" ☒ 4" ☐ 3"
WELL DIAMETER 2" I.D.	WELL MATERIAL Sch. 80 PVC	Drilling Method: Drive + Wash Casing

ELEV., FT.

DEPTH, FT.

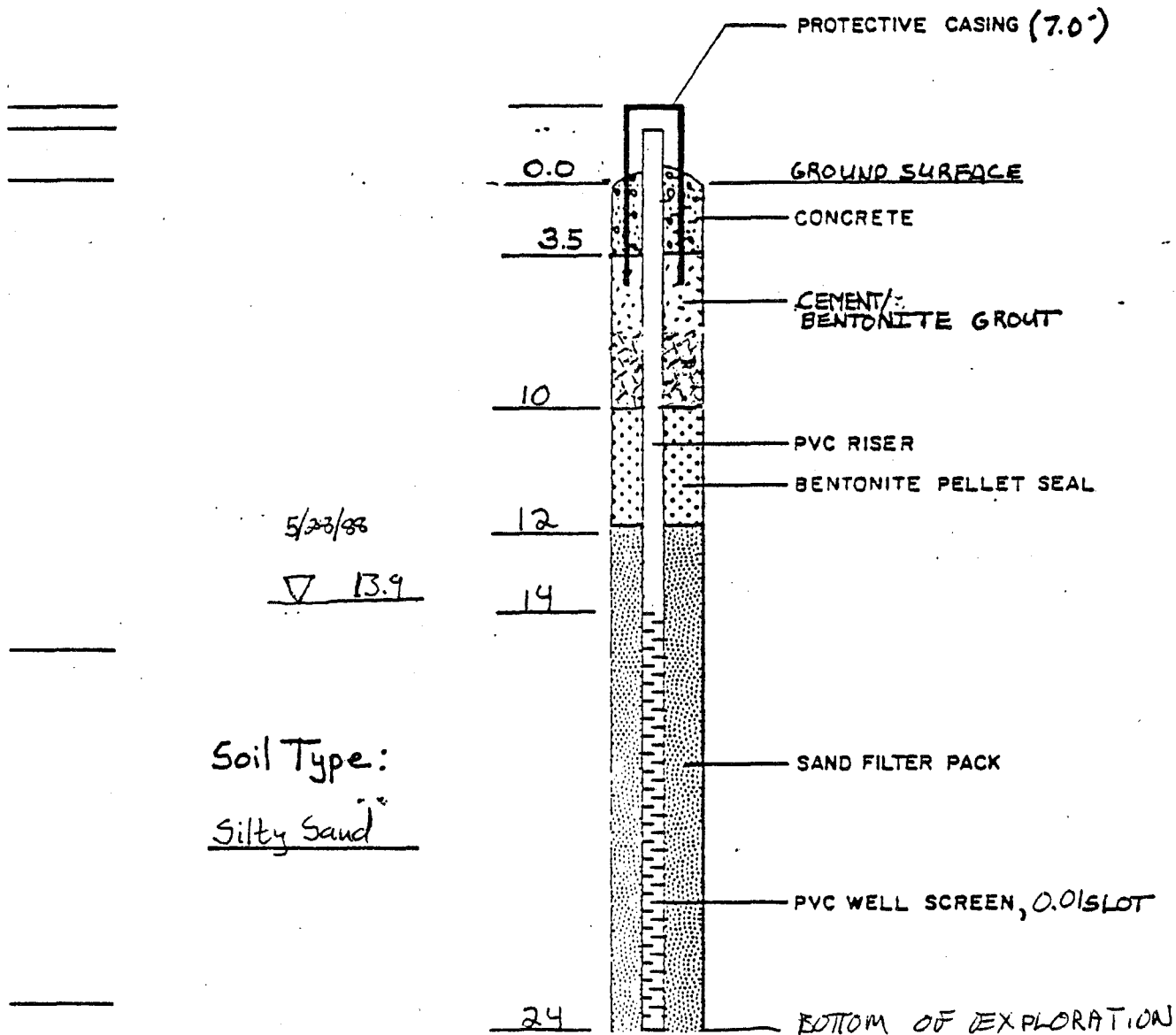


MONITORING WELL INSTALLATION DIAGRAM

PROJECT NO. 3971-63	PROJECT NAME <i>Winthrop Landfill Hydrogeologic Investigation</i>	WELL NO. P-1
INSTALLED BY: <i>Guild Drilling Co., Inc.</i> INSPECTED BY: <i>B. METZGER</i>	DATE INSTALLED 4/28/88	BORING DIAMETER ☐ 6" ☐ 5" ☒ 4" ☐ 3"
WELL DIAMETER 2" I.D.	WELL MATERIAL Sch. 80 PVC	Drilling Method: Drive + Wash Casing

ELEV., FT.

DEPTH, FT.

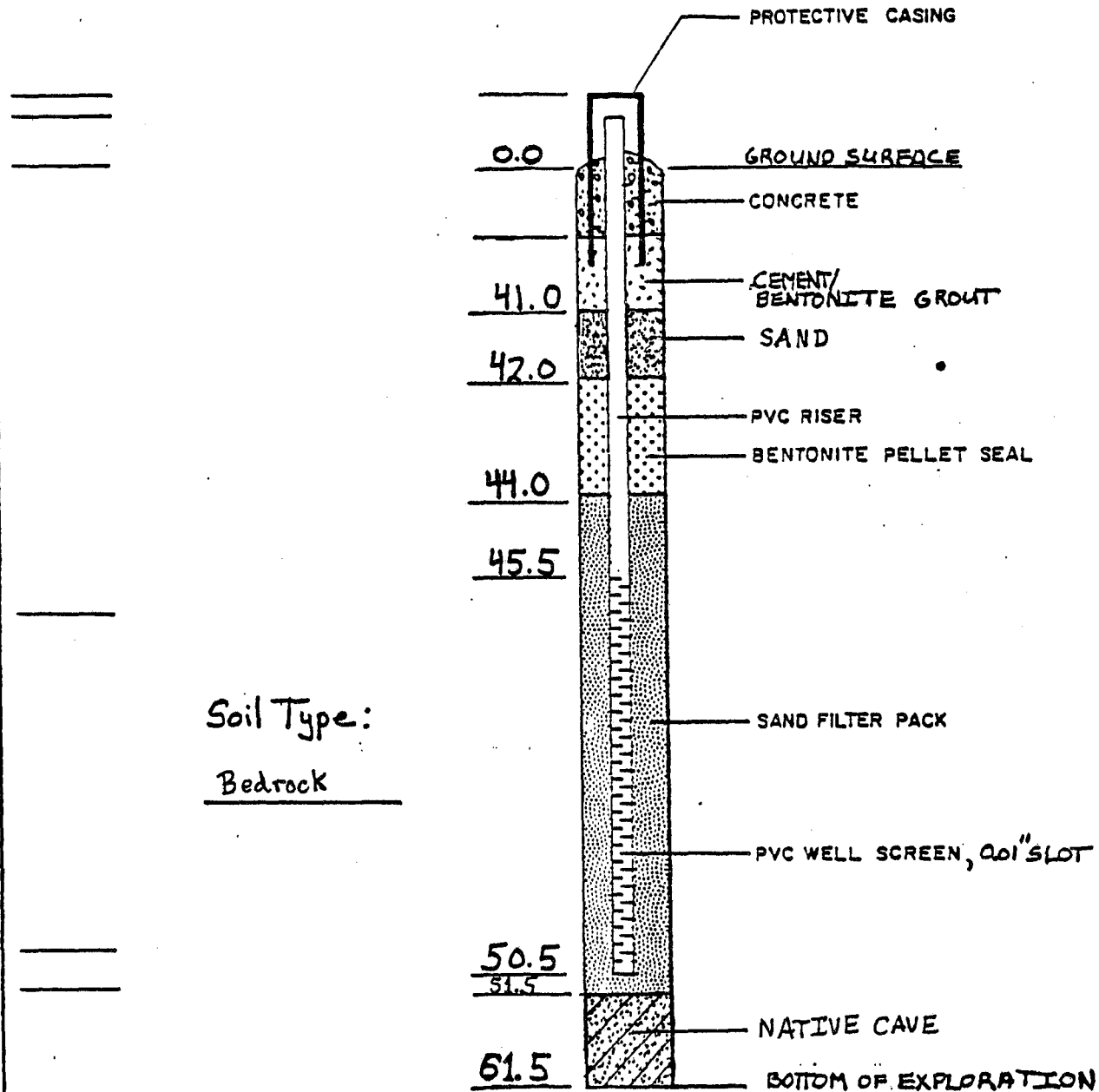


MONITORING WELL INSTALLATION DIAGRAM

PROJECT NO. 3971-63	PROJECT NAME <i>Winthrop Landfill Hydrogeologic Investigation</i>	WELL NO. MW-304A
INSTALLED BY: MATHES DRILLING INSPECTED BY:	DATE INSTALLED 11/19/88	BORING DIAMETER 6" 5" ~~4~~ 3"
WELL DIAMETER 2" I.D.	WELL MATERIAL Sch. 80 PVC	Drilling Method: Drive + Wash Casing

ELEV., FT.

DEPTH, FT.

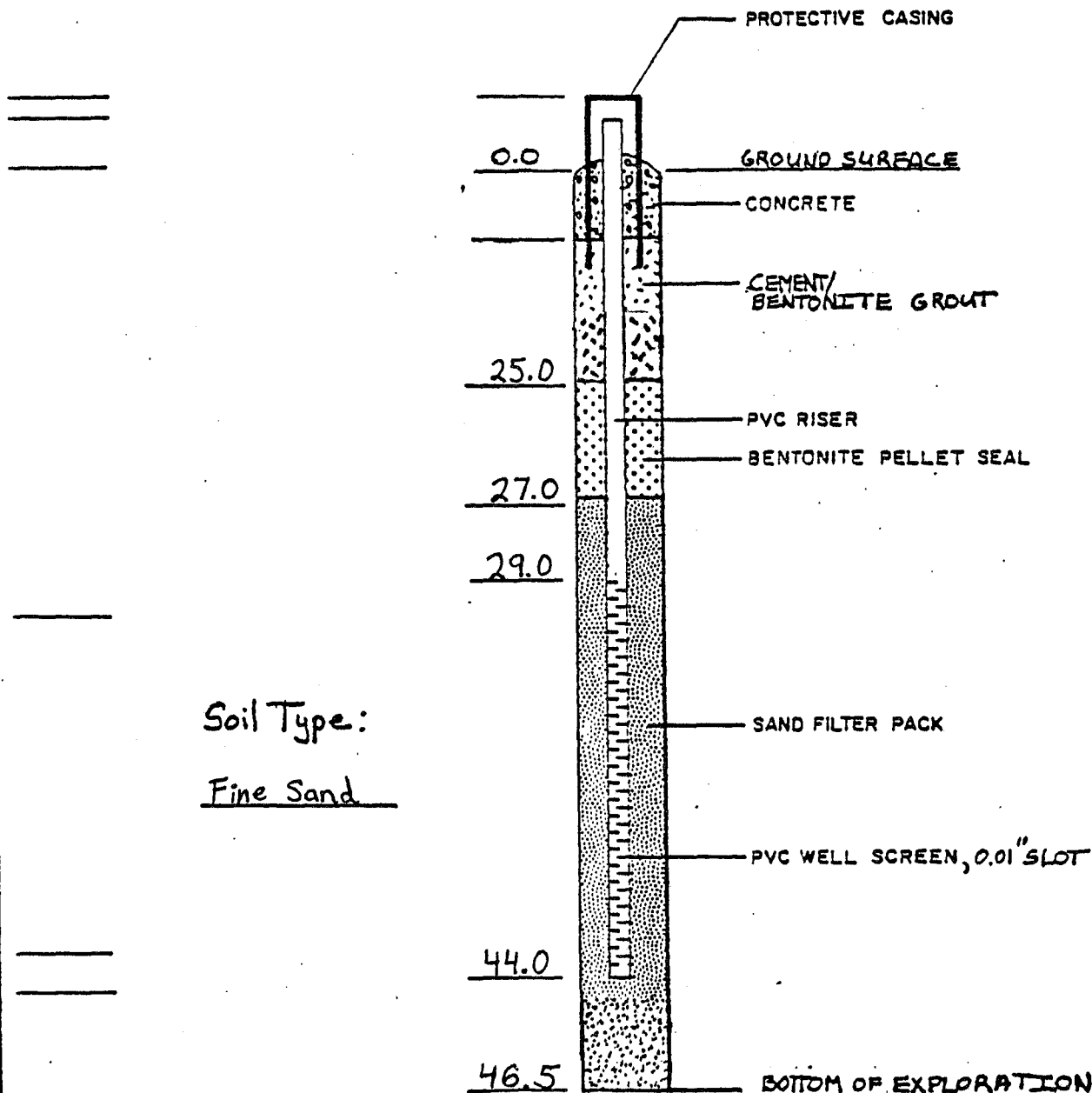


MONITORING WELL INSTALLATION DIAGRAM

PROJECT NO. 3971-63	PROJECT NAME <i>Winthrop Landfill Hydrogeologic Investigation</i>	WELL NO. MW-304B
INSTALLED BY: MATHES DRILLING INSPECTED BY:	DATE INSTALLED 11/22/88	BORING DIAMETER 6" 5" ~~4~~" 3"
WELL DIAMETER 2" I.D.	WELL MATERIAL Sch. 80 PVC	Drilling Method: Drive + Wash Casing

ELEV, FT.

DEPTH, FT.

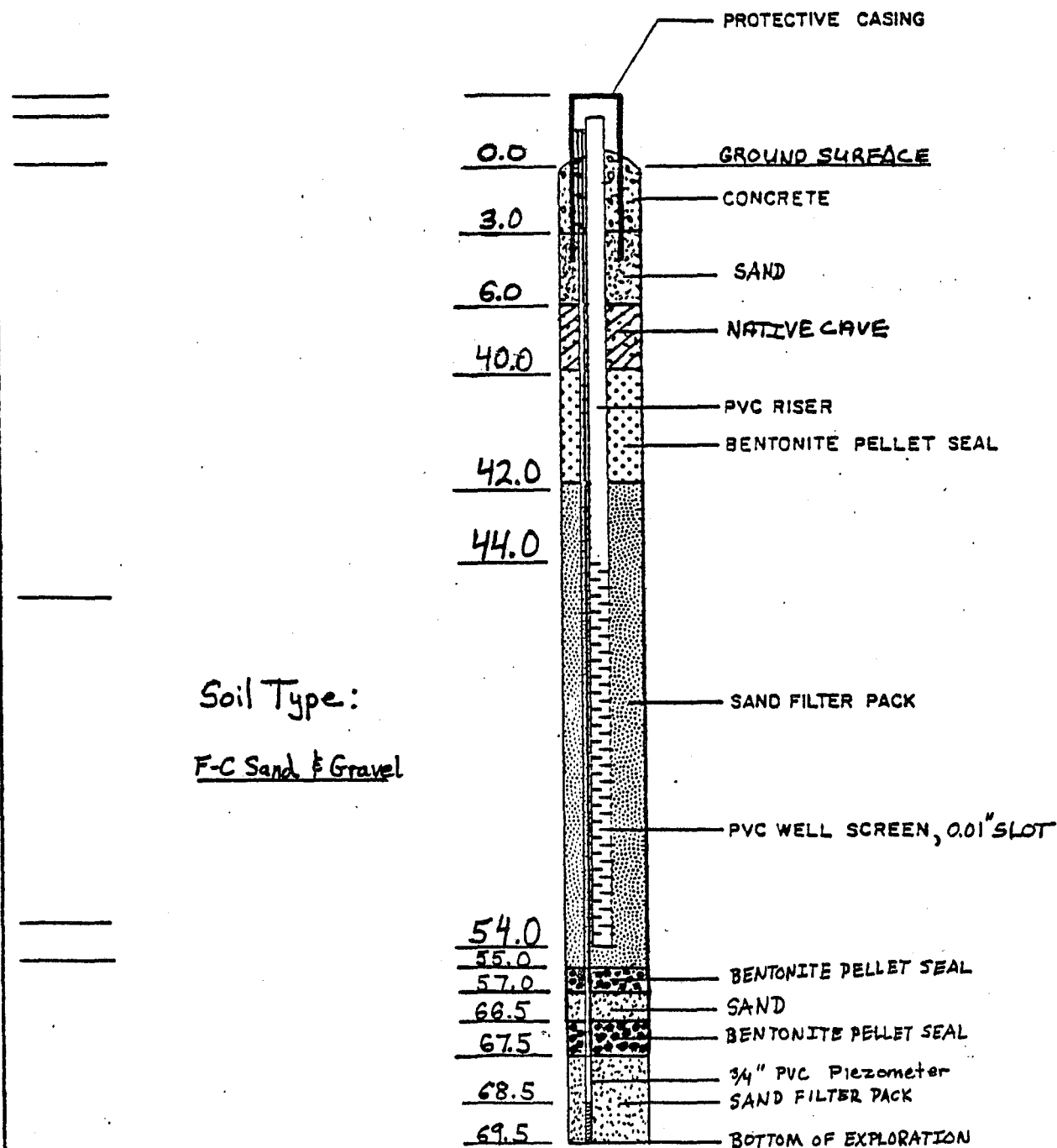


MONITORING WELL INSTALLATION DIAGRAM

PROJECT NO. 3971-63	PROJECT NAME <i>Winthrop Landfill</i> <i>Hydrogeologic Investigation</i>	WELL NO. MW-305A
INSTALLED BY: MATHES DRILLING	DATE INSTALLED 1/8/89	BORING DIAMETER 6" 5" ~~4~~" 3"
INSPECTED BY:		
WELL DIAMETER 2" I.D.	WELL MATERIAL Sch. 80 PVC	Drilling Method: Drive + Wash Casing

ELEV., FT.

DEPTH, FT.

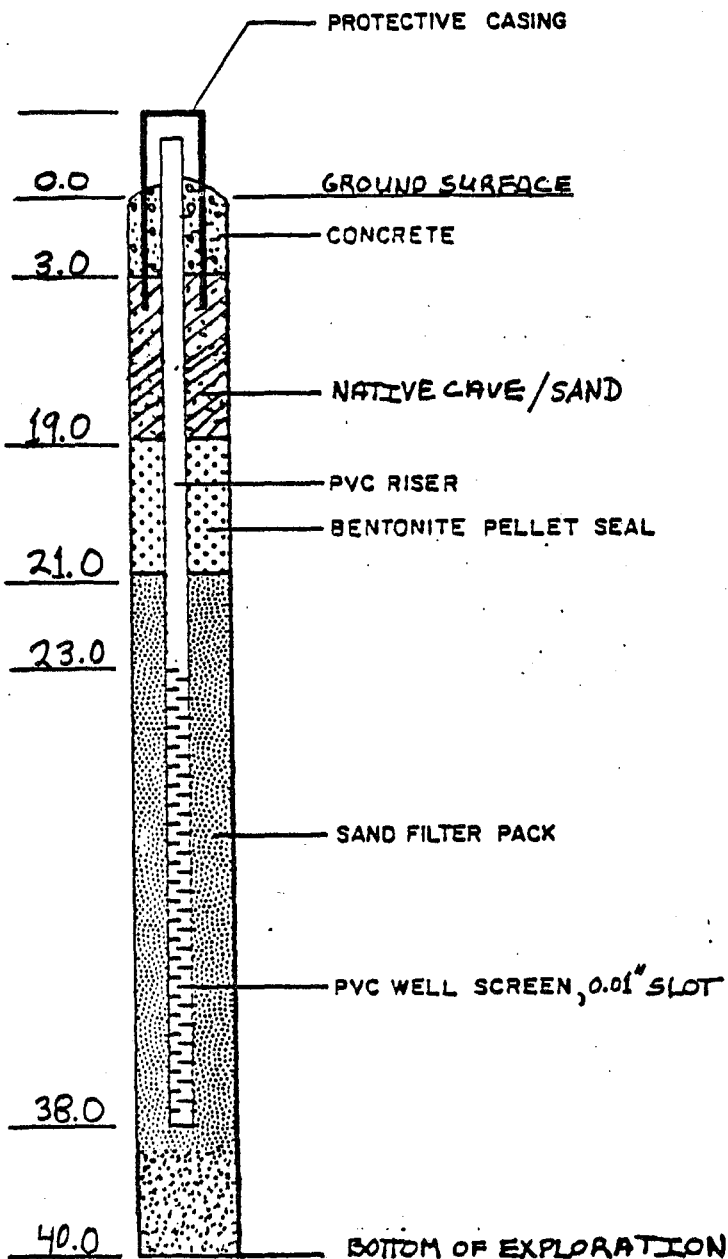


MONITORING WELL INSTALLATION DIAGRAM

PROJECT NO. 3971-63	PROJECT NAME <i>Winthrop Landfill Hydrogeologic Investigation</i>	WELL NO. MW- 305 B
INSTALLED BY: MATHES DRILLING INSPECTED BY:	DATE INSTALLED 1/22/89	BORING DIAMETER 6" 5" ~~4~~ 3"
WELL DIAMETER 2" I.D.	WELL MATERIAL Sch. 80 PVC	Drilling Method: Drive + Wash Casing

ELEV., FT.

DEPTH, FT.

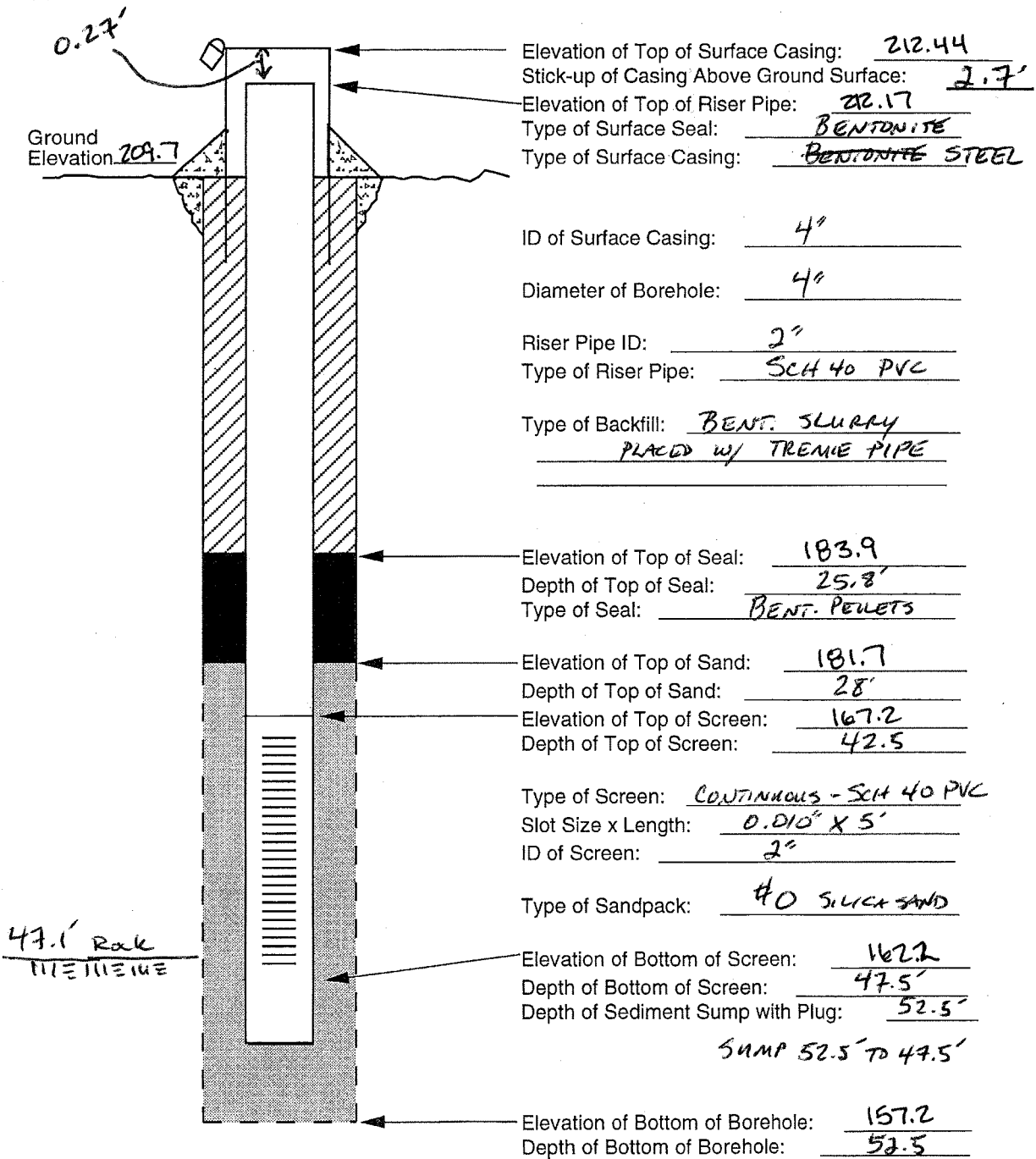


Soil Type:

F-C Sand, tr. Silt

MONITORING WELL CONSTRUCTION DIAGRAM

Project UTC WINTHROP Study Area ORC Driller GREAT WORKS
Project No. 51351-1 Boring No. OW-5 Drilling Method WASH & DRIVE TO ROCK
Date Installed 8-25-00 Development Method PUMP & SURGE
Field Geologist T.D. Longley



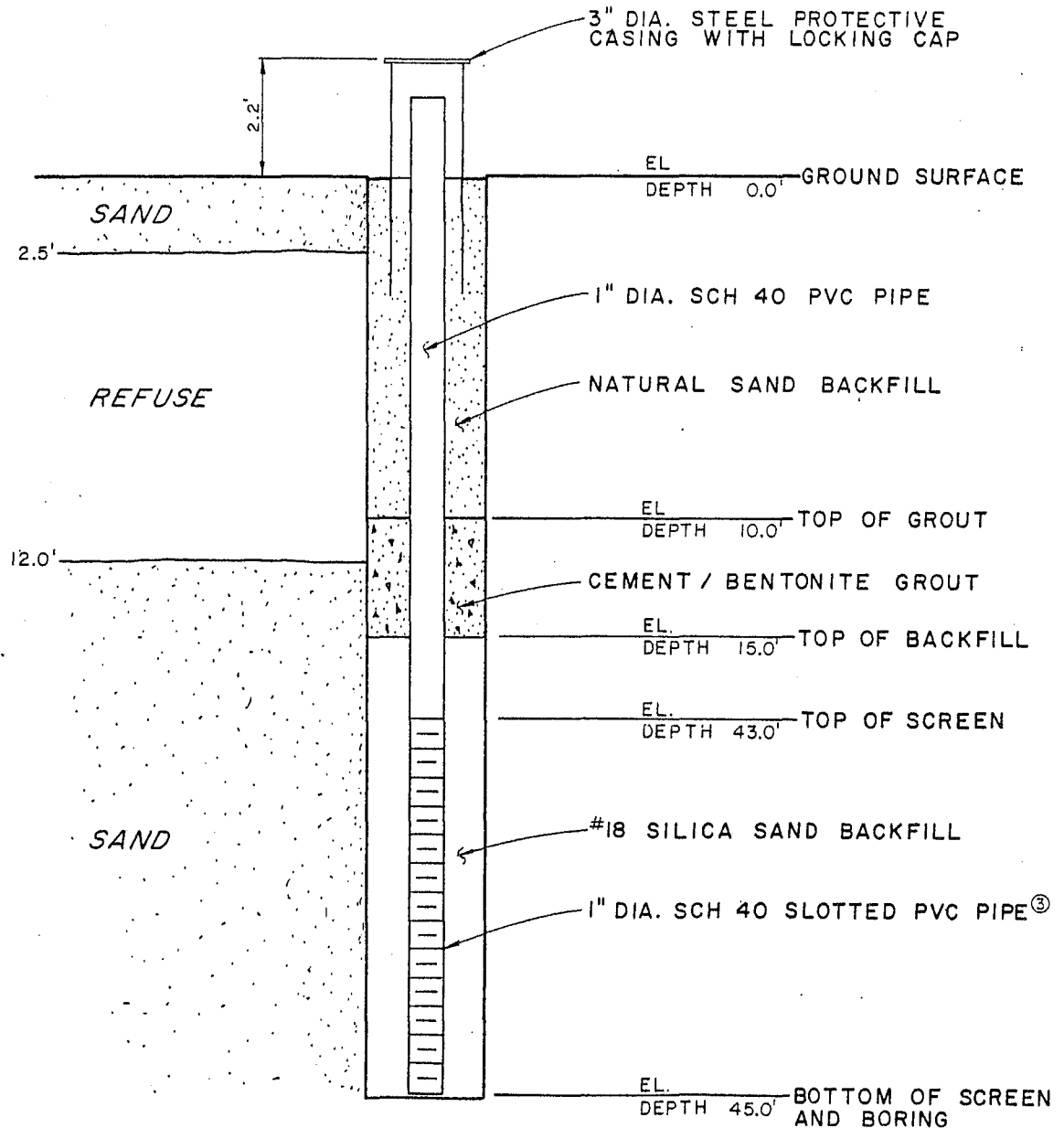
Piezometer Detail

PROJECT No. CH 85-031

PIEZOMETER No. P-110

PROJECT NAME UNITED TECHNOLOGIES INMONT/WINTHROP SITE

BORING LOCATION _____ DATE 11-1-85 BY KMB



NOTES:

1. NOT DRAWN TO SCALE.
2. SEE BORING LOG FOR DETAILED SOIL DESCRIPTION.
3. A HACKSAW BLADE WAS USED TO CUT SLOTS INTO PVC.

APPENDIX C

Data and Laboratory Procedures

APPENDIX C-1

Data Management, Validation and Qualification Procedures

APPENDIX C DATA MANAGEMENT, VALIDATION, AND QUALIFICATION PROCEDURES

The following procedures are used by Wood Environment and Infrastructure Solutions, Inc. (Wood) to manage and evaluate the usability of laboratory data from groundwater and surface water collected at the Winthrop Landfill site in Winthrop, Maine (Site).

DATA MANAGEMENT

Data management of chemical data includes the following tasks:

- Organization and storage of project field records including logbooks, instrument calibration records, exploration records, field sample collection records, and sample handling and Chain of Custody (COC) documents.
- Tracking of off-site laboratory samples and receipt of laboratory deliverables.
- Receipt, organization, and storage of laboratory data packages.
- Receipt of electronic data and entry of results into the project database.
- Data quality review at a validation level specified in the project-specific plan.
- Entry of data validation qualifiers and preparation of final data tables.
- Preparation of tables and figures for use in contamination assessments.

Electronic Data and Technical Environmental Database (TED)

The data management process will include procedures necessary to ensure consistent and complete collection of field data, tracking of the laboratory analytical and validation processes, consistent and timely production of electronic data deliverables (EDDs) from laboratories, and accurate entry of laboratory EDDs into Wood's environmental database, herein referred to as TED.

COC information will be documented by Wood when samples are collected. The COC data will be compared to the laboratory EDDs to track the completeness of the laboratory data deliverables.

The Wood Data Manager will upload the analytical result EDDs into TED upon receipt of the laboratory EDDs. Additional field data may be uploaded to TED using an EXCEL template after it has been documented as being checked.

HOLDING TIMES

Technical holding time for each analytical parameter is determined from the date of collection. If the analytical holding time is exceeded, sample results are considered estimated and qualified with a "J" for positive sample results and a "UJ" for non-detected sample results. If holding times are grossly exceeded (>2x holding time), all non-detected sample results are considered unusable and qualified with an "R".

If samples were not properly preserved or iced (maintained at 4°C) upon receipt at the laboratory or if VOC vials for a sample have air bubbles, professional judgment is used to evaluate the effect on sample data.

FIELD DUPLICATE PRECISION

If field duplicates are submitted for analysis, a comparison of reported results is calculated which should comply with a relative percent difference (RPD) <30 for aqueous samples and RPD <50 for sediment samples where:

$$RPD = [(original - duplicate)/(original + duplicate)/2] \times 100.$$

Generally, an RPD is calculated for a sample and its field duplicate for all results > PQL. If one result is > PQL and one sample result is non-detected or < PQL, an RPD is calculated using the PQL. If RPD between the sample result and field duplicate result exceed the 30 RPD criterion for aqueous samples or 50 RPD criterion for sediment samples, then the sample result and field duplicate result are considered estimated and qualified "J" for positive results or "UJ" for the non-detected result. RPDs are not calculated if both sample results for the duplicate pair are non-detect or detected below the PQL.

METHOD BLANK AND FIELD QC BLANK CONTAMINATION

Samples are evaluated for contamination introduced from the laboratory or the field by reviewing associated method blank, trip blank, source blank, and equipment blank results. If any sample does not have an associated method blank, then the laboratory should be notified and professional judgment should be used in evaluating the sample data. If any of these blanks have positive results for constituents, then the associated data are qualified as follows:

For an analyte detection in any blank, with the exception of common laboratory contaminants, an action level is established at 5 times the blank concentration. For detections of common laboratory contaminants acetone, 2-butanone, or methylene chloride, an action level is established at 10 times the blank concentration. If the analyte is detected in the associated sample at a concentration less than the reporting limit and less than the established action level, the sample result is qualified as not detected (U) at the reporting limit. If the analyte is detected in the associated sample at a concentration greater than the reporting limit and less than the established action level, the sample result is qualified as not detected (U). If the analyte is detected in the associated sample at a concentration greater the established action level, no action is required.

Action levels established for blank contaminants are adjusted for sample dilutions and percent moisture in sediment samples. Field and trip blanks are not qualified because of contamination in other blanks.

SURROGATE RECOVERIES

Volatile organic compound analyses are reviewed and evaluated for compliance to laboratory-specific surrogate standard recovery criteria. The percent recovery (%R) is defined as (sample surrogate concentration/expected surrogate concentration) x 100. The following actions are taken for surrogate recoveries falling outside laboratory-specific control limits.

Surrogate Standard	%R < 10	%R < LL	%R > UL
Action	(J) positive (R) non-detects	(J) positive (J) non-detects	(J) positive

Notes: LL = lower limit; UL = upper limit

LABORATORY CONTROL SAMPLE

Laboratory Control Sample (LCS) and LCS duplicate results are reviewed and evaluated for laboratory compliance. LCS percent recovery ranges and duplicate precision criteria are provided by the laboratory. If the LCS recovery is below the laboratory acceptance level, then all data (positive and non-detected) are considered estimated "J". If the LCS recovery is greater than the upper level, then positive data results are estimated "J".

INITIAL AND CONTINUING CALIBRATIONS

The laboratory case narratives and non-conformance reports are reviewed to identify any issues relating to the initial and continuing calibrations. Each non-conformance is further investigated to determine if and how it effects the data quality and usability for the project.

MATRIX SPIKE/MATRIX SPIKE DUPLICATES

Matrix spike and matrix spike duplicate (MS/MSD) samples are selected by the laboratory upon receipt. The MS/MSD results are reviewed and evaluated for laboratory compliance. The MS/MSD percent recovery ranges and duplicate precision criteria are provided by the laboratory. In organic samples, if the percent recovery is below the laboratory acceptance level, then only the sample associated with the sample is (positive and non-detected) considered estimated "J". If the percent recovery is greater than the acceptable level then the positive result in the associated sample is qualified as estimated "J." In inorganic samples, if the percent recovery is below the acceptable level, all samples of the same matrix are considered estimated "J." If the LCS recovery is greater than the upper level, then positive data results are estimated "J".

DATA VALIDATION QUALIFIERS

The following qualifiers will be used to flag results:

U = the analyte was not detected; value represents the PQL
 UJ = estimated PQL
 J = estimated value
 R = rejected value (unusable result)

The data quality evaluation for Winthrop Landfill will include examining the results of any data generated when an analytical method was not within established criteria. If possible, these qualified data will be examined for possible direction and magnitude of bias. The evaluation of usability will include an evaluation of representativeness of the results with site history and conditions.

As part of the data quality evaluation, analytical data are reviewed to determine if the data were generated by methods meeting the QC requirements for the methods. This is established during data validation which is a review of the quality control documentation provided by the laboratory.

After the sampling data and analytical data are established to be acceptable according to the validation guidelines, the data are reviewed for their site, environmental, and historical context.

Analytical DQOs Analytical DQOs are reviewed which include measuring the precision, accuracy, representativeness, comparability, and completeness (PARCC parameters) of the data set. These PARCC parameters are indicators of the data quality. These parameters are defined below.

Precision is a measure of the reproducibility of the measurements and is generally expressed as the variability of a group of measurements compared to their average value and may be expressed as a standard deviation, coefficient of variation, relative standard deviation (RSD), RPD, range (minimum to maximum value), or relative range.

Precision of analytical data from the monitoring program will be calculated from the laboratory duplicates or LCS/LCSD and the field duplicates according to the following formula.

$$RPD = \frac{|R1 - R2| \times 100\%}{(R1 + R2) / 2}$$

Where: R1 and R2 are duplicate sample results.

The RPD for the laboratory duplicates and LCS/LCSDs will be a measure of the method's precision, while the RPD for the field duplicates will include the site and constituent specific variations as well as the method precision. Laboratory duplicates and LCS/LCSDs are performed per analytical batch. Field duplicates are collected at a frequency of 5% (1 duplicate for every 20 samples).

Accuracy is the bias in the measurement system and reflects how close a measurement is to the true value. Accuracy and bias measurements include recovery of spiked samples, recovery of surrogates, chemicals in field QC samples (trip blanks, field blanks, equipment rinsates, source water), and chemicals in laboratory method blanks.

Accuracy for the monitoring program will be calculated from the percent recoveries (%R) of the surrogate standards and LCS/LCSDs according to the following formula:

$$\%R = \frac{(\text{Measured amount} - \text{amount in unspiked sample}) \times 100}{\text{Amount spiked}}$$

The %R for the laboratory surrogates will be a measure of the method's accuracy, while the %R for the LCS/LCSDs include constituent specific variations. Accuracy goals are summarized in Table 3.3 of the text.

Representativeness is a qualitative measure that expresses the degree to which sample data accurately and precisely represent the environmental conditions. It is considered during planning and data evaluation. Representativeness in the monitoring program is assured in two ways: through the use of analytical data that meet method requirements and program precision and accuracy requirements, and through a data review process in which data are reviewed for their context in the site, historical database, and environmental conditions. The result of the process is acceptance of the result or classification as provisional or non-representative. In any case, the datum is retained in the database and flagged accordingly. No result is removed from the database.

Comparability is a data quality indicator that affects the combination of data for use. Comparability is assured for the monitoring program by using standard USEPA methods whenever possible and by using methods which give comparable results to those used to define the PALs. Comparability is also assured by collecting and handling the samples in the same manner each sampling event. This is accomplished by using sampling Standard Operating Procedures (SOPs) and reporting results with consistent units.

Completeness is the percentage of valid measurements taken. For the monitoring program, there are two measures of completeness, one for the sampling program and one for the analytical data. The completeness of data for the sampling program may be calculated by the following formula:

$$\text{Percent completeness} = \frac{(\text{Number of acceptable data points}) \times 100}{\text{Total number of samples scheduled}}$$

The percent completeness for the analytical data may be calculated by the following formula:

$$\text{Percent completeness} = \frac{(\text{Number of acceptable data points}) \times 100}{\text{Total number of samples analyzed}}$$

The completeness for the sampling program may be affected by several factors including inability to collect samples from frozen locations, dry surface water locations, and damaged wells.

According to USEPA guidance, the percent completeness goal for analytical data for DQO Levels III and V is approximately 80 percent. The analytical data for the monitoring program are expected to meet this goal.

LABORATORY DATA DELIVERABLE PACKAGES

Deliverable packages will include a narrative that summarizes activities and any problems or issues, forms summarizing sample and QC blank results, and forms summarizing laboratory QC measurement parameters including laboratory control data summaries, matrix spike/matrix spike duplicate summaries (for samples requested on the COC), surrogate summaries, and laboratory duplicate summaries.

APPENDIX C-2

Laboratory Standard Operating Procedure





Document Information

Document Number: ENV-SOP-MTJL-0081	Revision: 01
Document Title: Semi-volatile Organics by GCMS using Capillary Column (EPA Methods 8270C, EPA 8270D, EPA Method 625, SM 6410B), Including Provisions for Analysis in SIM Mode	
Department(s): SVOA	

Date Information

Effective Date: 19 Sep 2019

Notes

Document Notes:

All Dates and Times are listed in: Central Time Zone

Signature Manifest

Document Number: ENV-SOP-MTJL-0081**Revision:** 01**Title:** Semi-volatile Organics by GCMS using Capillary Column (EPA Methods 8270C, EPA 8270D, EPA Method 625, SM 6410B), Including Provisions for Analysis in SIM Mode

All dates and times are in Central Time Zone.

ENV-SOP-MTJL-0081

QM Approval

Name/Signature	Title	Date	Meaning/Reason
Steven Miller (006597)	QA Director	19 Sep 2019, 03:59:33 PM	Approved

Management Approval

Name/Signature	Title	Date	Meaning/Reason
Christopher Johnson (006487)	Organics Director	19 Sep 2019, 09:07:12 PM	Approved



STANDARD OPERATING PROCEDURE

TITLE: ENV-SOP-MTJL-0081 Semivolatile Organics by GCMS using Capillary Column (EPA Methods 8270C, 8270D, 625, 625.1 and SM6410 B) Including Provisions for Analysis in SIM Mode

ISSUER: Pace National – Mt. Juliet, Tennessee

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1.0 SCOPE AND APPLICATION

STATE NOTE: For samples analyzed in conjunction with the Ohio Voluntary Action Program (VAP) please utilize SOP #330345OH.

- 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 IV.
- 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 on ESC's intranet.
 - 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), Limits of Detection (LOD) and Limits of Quantitation (LOQ)*. Should the procedure be utilized for DOD support; then the frequency of these studies must meet the requirements of the current DOD QSM (see Attachment IX).
 - 1.8.2 Lower Limit of Quantitation (LOQ) – For analyses performed per the requirements of Method 8000D, the LLOQ is established at concentrations where both quantitative and qualitative requirements can consistently be met (see Sections 2.10 and 10.4).



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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. Soil analysis using the same technology can also be performed with extraction as noted in ESC SOP #330707 and no concentration performed on the extract. This process is termed throughout this SOP as non-concentrated soil. Solid samples can also be extracted traditionally using methylene chloride-acetone (1:1) and a sonication process (SOP #330705) or with methylene chloride using the microwave process (SOP #330707), where permitted. These extracts are denoted in this procedure using the terminology “concentrated soil” extracts.
- 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.
- 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 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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- 2.9 Isotope dilution calibration - Isotope dilution calibration is essentially a special case of internal standard calibration. In isotope dilution, the internal standards are stable isotopically-labeled analogs of the target analytes *and* they are added to the sample prior to any sample handling steps, including sample extraction. Because the spiked compounds differ from the target compounds only in the presence of the stable isotopes, the physical and chemical behavior of each labeled compound is virtually the same as its unlabeled "native" analog. Thus, any losses of the target compound that may occur during any of the sample preparation, extraction, cleanup, or determinative steps will be mirrored by a similar loss of the labeled standard.
 - 2.10 Lower Limit of Quantitation (LLOQ) – For analyses performed according to the requirements of Method 8000D, the lowest concentration at which the laboratory has demonstrated target analytes can be reliably measured and reported with a certain degree of confidence, which must be greater than or equal to the lowest point in the calibration curve.
 - 2.11 Large Volume Injection (LVI): any injection volume >5ul. Technique is dependent upon type of GC inlet used and sensitivity of detection.
 - 2.12 Minimum Level (ML): A term used in Method 625.1 which refers to either the sample concentration equivalent to the lowest calibration point in a method or a multiple of the MDL, whichever is higher. Minimum levels may be obtained in several ways: They may be published in a method; they may be based on the lowest acceptable calibration point used by a laboratory; or they may be calculated by multiplying the MDL in a method, or the MDL determined by a laboratory, by a factor of 3. For the purposes of NPDES compliance monitoring, EPA considers the following terms to be synonymous: "quantitation limit," "reporting limit," and "minimum level."
 - 2.13 See the current Quality Assurance Manual for other definitions associated with terms found in this document.
- 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. A reference file of safety data sheets (SDSs) is made available on ESC's intranet to all personnel. Use hazardous 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 protocols.
 - 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.



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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.
- 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% $\text{Na}_2\text{S}_2\text{O}_3$ 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 $<6^\circ\text{C}$ (not frozen).
- 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 and Reporting*.

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.
- 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 IV and are recommended from the published 8270 methods. There is no method stated ions for the following: Pyridine, 1-



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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. Targets have strongly-responding, analyte-specific secondary ions suitable for quantitative use. Refer to Attachment IV 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.
- 5.4.7 Benzenethiol, or thiophenol, can be found in refinery wastes at caustic pH values. Benzenethiol is unstable in water/soils of neutral or acidic pH values. Benzenethiol rapidly degrades in organic solvents used to prepare the instrument calibration standards. Benzenethiol is part of Appendix VIII and the 1985 Skinner List, but was never included in Appendix IX to 40 CFR 264, due to its instability in the environment

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 (Phenomex ZB-5MS or equivalent).
- 6.1.3 Column 2 – J&W 30m x 0.25mm x 0.5µm film DB5MS or an equivalent is used. Ultrapure (99.999%) Helium gas is used for a mobile phase.
- 6.1.4 Syringes: Agilent (or equivalent) syringes sizes 10µL, 25µL, 50µL, 100µL and 1.0mL.



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- 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

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 #030230, *Standard Logger – Tree Operation*. 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.
- 7.6 Stock standard solutions
 - 7.6.1 Restek 8270 MegaMix – 31850, or equivalent, at 1000ppm
 - 7.6.2 Agilent ULTRAgold Custom Standard – CUS-28559, at 1000ppm
 - 7.6.3 NSI Lab Solutions Custom BNA Mix – Q6330, or equivalent, at 2000ppm
 - 7.6.4 Restek Custom a-Terpineol/Quinoline Standard – 572011, or equivalent, at 2000ppm



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- 7.6.5 Restek 8270 Benzidines Mix #2 – 31852, or equivalent, at 1000ppm
- 7.6.6 Restek Benzoic Acid Mix – 31879, or equivalent, at 2000ppm
- 7.6.7 NSI Lab Solutions 8270 BNA Mix – C-701, or equivalent, at 1000ppm
- 7.6.8 Phenova Custom 8270 Appendix IX Mix – ALO-130094, or equivalent, at 1000ppm
- 7.6.9 Phenova Benzidines Standard – ALO-101244, or equivalent, at 2000ppm
- 7.6.10 Phenova Benzoic Acid Mix – ALO-101246, or equivalent, at 2000ppm
- 7.6.11 Phenova Custom Appendix IX Mix 2 – ALO-130203, or equivalent, at 2000ppm
- 7.6.12 Phenova Methapyrilene – ALO-130204, or equivalent, at 2000ppm
- 7.6.13 Phenova Hexachlorophene – ALO-130233, or equivalent, at 4000ppm
- 7.6.14 Phenova Custom Appendix IX Mix 1 – ALO-130202, or equivalent, at 2000ppm
- 7.6.15 Phenova 8270 OP Pesticides Mix – ALO-101256, or equivalent, at 2000ppm
- 7.6.16 Phenova 1,4-Dioxane – ALO-101313, or equivalent, at 2000ppm
- 7.6.17 Restek 1,4-Dioxane – 31853, or equivalent, at 2000ppm
- 7.6.18 Phenova Benezenethiol – ALO-130085, or equivalent, at 1000ppm
- 7.6.19 Phenova Sulfolane Mix – ALO-130201, or equivalent, at 800ppm
- 7.6.20 Restek Sulfolane Standard – 36413, or equivalent, at 800ppm
- 7.6.21 Restek Custom BNA Subgroup Standard #1 – 574999, or equivalent, at 200ppm
- 7.6.22 Agilent ULTRAgold Custom Standard – CUS-23897, or equivalent, at 1000ppm
- 7.6.23 Agilent ULTRAgold Custom Standard – CUS-23952, or equivalent, at 1000ppm
- 7.6.24 Restek TX TPH Calibration Mix – 569373, or equivalent, at 10000ppm
- 7.7 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.

STANDARD NAME	TOTAL VOLUME (mL)	RECIPE
BNA INTERNAL STANDARD	10	Open Ampule - No dilution required, Custom BNA Internal Standard, NSI Lab Solutions, Cat. No. Q-6343-O
1,4-DIOXANE-D8 IS @ 50ppm	10	250uL 1,4-Dioxane-d8 Standard, Restek, 30614 9.75mL Methylene Chloride

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NDMA-D6 IS @ 50ppm	10	500uL 521 Surrogate Std., Restek, Cat. No. 33910 9.5mL Methylene Chloride
TUNE @ 50ppm	10	500uL GC/MS tuning std, Accu Standard, Cat. No. M-625-TS-20X 9.5mL Methylene Chloride
TUNE @ 10ppm	10	100 uL GC/MS tuning std, Accu Standard, Cat. No. M-625-TS-20X 9.9 mL Methylene Chloride
8270 PRIMARY INT @ 200ppm	10	2mL 8270 MegaMix, Restek, Cat. No. 31850 2mL ULTRAgold Custom Standard, Agilent, Cat. No. CUS-28559 6mL Methylene Chloride
8270 TCL INT @ 200ppm	10	1mL Custom BNA Mix, NSI Lab Solutions, Cat. No. Q6330 1mL Custom a-Terpineol/Quinoline Standard, Restek, Cat. No. 572011 1mL 8270 Benzidines Mix #2, Restek, Cat. No. 31852 1mL Benzoic Acid Mix, Restek, Cat. No. 31879 6mL Methylene Chloride
8270 SVMS SSCV @ 10ppm	1	10uL 8270 BNA Mix, NSI Lab Solutions, Cat. No. C-701 10uL ULTRAgold Custom Standard, Agilent, CUS-28559 980uL Methylene Chloride
8270 TCL SSCV @ 200ppm	10	2mL Custom 8270 Appendix IX Mix, Phenova, Cat. No. ALO-130094 1mL Benzidies Standard, Phenova, ALO-101244 1mL Benzoic Acid Mix, Phenova, ALO-101246 1mL Custom Appendix IX Mix 2, Phenova, Cat. No. ALO-130203 5mL Methylene Chloride
8270 TCL SSCV @ 10ppm	1	100uL 8270 TCL SSCV @ 200ppm, 900uL Methylene Chloride
SVAM INT @ 200ppm	10	1mL Custom Appendix IX Mix 2, Phenova, ALO-130203 1mL Methapyrilene, Phenova, Cat. No. ALO-130204 1mL Hexachlorophene, Phenova, Cat. No. ALO-130233 7mL Methylene Chloride
SVAP INT @ 200ppm	10	1mL Custom Appendix IX Mix 1, Phenova, Cat. No. ALO-130202 9mL Methylene Chloride
SVOP INT @ 200ppm	10	1mL 8270 OP Pesticides Mix, Phenova, Cat. No. ALO-101256 9mL Methylene Chloride
NDMA/1,4-DIOXANE INT @ 20ppm	10	100uL 1,4-Dioxane, Phenova, Cat. No. ALO-101313 1mL 8270 Primary Int. @ 200ppm, 8.9mL Methylene Chloride
NDMA/1,4-DIOXANE INT @ 2ppm	5	500ul NDMA/1,4-Dioxane Int @ 20 4.5mL Methylene Chloride
1,4-DIOXANE SSCV @ 200ppm	10	1mL 1,4-Dioxane, Restek, Cat. No. 31853 9mL Methylene Chloride
1,4-DIOXANE SSCV @ 10ppm	10	500uL 1,4-Dioxane SSCV @ 200pm 9.5mL Methylene Chloride
NDMA SSCV @ 10ppm	10	100uL 8270 BNA Mix, NSI Lab Solutions, Cat. No. C-701 9.9mL Methylene Chloride
BENZENETHIOL CAL INT @ 100ppm	10	1mL Benzenethiol, Phenova, Cat. No. ALO-130085 1mL Ultragold Custom Standard, Agilent, CUS-28559 8mL Methylene Chloride
BENZENETHIOL SSCV @ 100ppm	10	1mL Benzenethiol, Phenova, Cat. No. ALO-130085 9mL Methylene Chloride

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SULFOLANE CAL INT @ 50ppm	10	625uL Sulfolane Mix, Phenova, Cat. No. ALO-130201 500uL Ultragold Custom Standard, Agilent, CUS-28559 8.875mL Methylene Chloride
SULFOLANE SSCV @ 50ppm	10	625uL Sulfolane, Restek, Cat. No. 36413 9.375mL Methylene Chloride

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 LVI work. Internal standard intermediates at 16µg/mL and 4µg/mL are prepared for spiking, RV/LVI 8270PAHand RV/LVI 8270SIM analyses, respectively.

7.8.1 For all concentrated soil and 8270 full run water reduced volume extracts, use the 8000µg/mL internal standard solution. Each sample extract undergoing analysis is spiked with 5µL of internal standard intermediate solution, resulting in a concentration of 8µg/mL of each internal standard.

7.8.2 For non-concentrated soil, reduced volume water and 3511 water analyses, including PAH and DROMO, use the 16µg/mL ISTD intermediate. For non-concentrated soil, reduced volume and EPA 3511 water 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.

7.9 Using a volumetric syringe, measure each of the solutions listed in Section 7.9 and place into a 10mL volumetric flask.

7.10 Preparation of Working Standards

Standards must be stored at ≤6°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 7.11.1 through 7.11.6 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 concentrated soil and 1L concentrated 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 7.10 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 concentration per extraction method for quantitation purposes.



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SVOC mix (200ppm) μL	ISTD mix μL	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 (soil and water) 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 0.01 $\mu\text{g/mL}$ up to 1 $\mu\text{g/mL}$. Surrogates are included at the same concentrations. Internal standards are spiked at a constant of 160 $\mu\text{g/L}$ for quantitation purposes.

SVOC mix (2ppm) μL	ISTD mix μL	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 concentrated 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, 10,000 $\mu\text{g/L}$. 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 8 $\mu\text{g/mL}$.

SIM Standard Concentration ($\mu\text{g/L}$)	Amount Added (μL) 5 $\mu\text{g/mL}$ 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

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SIM Standard Concentration (ug/L)	Amount Added (uL) 5µg/mL Int.	Final Volume (mL)
2000	200.0	1.0
4000	400.0	1.0
10000	1000.0	1.0

7.10.4 For SIM analyses using reduced volume, non-concentrated soil, 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 40µg/L.

SIM RV 3511 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 Missouri DRO analysis by 3511 and non-concentrated soil, 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

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. Alternative GCMS tuning criteria from that specified in EPA 8270C is acceptable as permitted in Section 7 of the published method.

8.1 GC Conditions: The GC conditions are listed in each instrument maintenance log and are updated as necessary.

8.1.1 Due to the tuning and calibration requirements outlined in this method, a liner change is necessary prior to beginning an analytical sequence. Any additional maintenance performed on the instrumentation will be documented as performed



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in the specific instrument maintenance log. (i.e., column clip/change, septa change, inlet cleaning, detector cleaning/maintenance, etc.)

- 8.2 Mass Spectrometer Tuning Criteria: The GC/MS is hardware-tuned using a 50ng (or less) injection of DFTPP. Analyses must not begin until the tuning criteria are met. 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. If Autofind is not utilized, select the mass spectrum at the peak apex for evaluation, or use an average mass spectrum across the entire DFTPP peak. 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.

The mass spectrometer must be tuned every 12 hours if samples, standards, etc. are to be analyzed for Methods 8270C, 8270D, and 625.1. ESC uses 8270D evaluation criteria per method allowances.

TABLE 8.2
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.

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 total ion chromatograms 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



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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 and 625.1, 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.

- 8.3 The use of selected ion monitoring (SIM) is acceptable for applications requiring quantitation limits below the normal range of electron impact mass spectrometry. However, SIM may provide a lesser degree of confidence in the compound identification since less mass spectral information is available. Using the primary ion for quantitation and the secondary ions for confirmation set up the collection groups based on their retention times. The selected ions are nominal ions and most compounds have small mass defect, usually less than 0.2 amu, in their spectra. These mass defects should be used in the acquisition table. The dwell time may be automatically calculated by the laboratory's GC/MS software or manually calculated using the following formula. The total scan time should be less than 1,000 msec and produce at least 5 to 10 scans per chromatographic peak. The start and stop times for the SIM groups are determined from the full scan analysis using the formula below: Additional guidance for performing SIM analyses, in particular for PAHs and phenol target analyte compounds, can be found in the most recent CLP semivolatile organic methods statement of work (SOW). See the SIM sections from the following CLP SOW for further details: EPA CLP Organics SOW. (Reference 14)

SIM Groups for PAHs and including pentachlorophenol and hexachlorobenzene

SIM Group	1	2	3	4	5	6	7	8
RT start	Solvent delay	Before 2-Methyl naphthalene	Before Acenaphthalene	Before Fluorene	Before Fluoranthene	Before Benzo (a)-anthracene	Before Benzo (b)-fluoranthene	Before Dibenz (a,h)-anthracene
Ions	82, 128, 129, 136, 137	127, 141, 142, 162, 171, 172	139, 151, 152, 153, 154, 162, 164, 168	94, 165, 166, 176, 178, 179, 188, 264, 266, 268, 282, 284, 286	200, 202, 203, 244, 245	226, 228, 229, 240, 241	252, 253, 260, 264	138, 139, 276, 277, 278, 279
Dwell	40	35	25	30	40	50	75	50

8.4 Calibration

8.4.1 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



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by linear or quadratic regression instead of RF. If the RSD of any target analyte is $\leq 15\%$, 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 0.995 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	

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 $\leq 20\%$. 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 $\leq 20\%$, 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 0.995 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

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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.

Table 8.3.1c: Recommended Minimum Response Factors for Each Calibration Level (Initial and Continuing Calibration)

<i>Compound</i>	<i>Minimum Response Factor</i>	<i>Compound</i>	<i>Minimum Response Factor</i>
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		

EPA 8270C GC/MS SIM: When analyzing samples using SW-846 8270C SIM, all target compounds must be treated as CCCs and must have an average RSD of $\leq 30\%$.



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EPA 8270D GC/MS SIM: If analyzing samples by EPA 8270D SIM, follow the initial calibration criteria for the specified referenced method as found in section 8.3.1 (EPA Method 8270D).

EPA Method 625: The working standards prepared in section 7.10 are injected and average response factors are calculated. A minimum of 3 points calibration is required for method 625. The %RSD is calculated for the standards analyzed and must be $\leq 35\%$ for all compounds in order to assume linearity.

EPA Method 625.1: One of the calibration standards should be at a concentration at or below the reporting limit. The resulting calibration must meet all applicable acceptance criteria in Section 10, based on the RSD, RSE, or r^2 . The concentrations of the other calibration standards should correspond to the expected range of concentrations found in real samples or should define the working range of the GC/MS system for full-scan and/ or SIM operation, as appropriate. A minimum of six concentration levels is required for a second order, non-linear (i.e., quadratic) calibration.

Calculate the mean (average) and relative standard deviation (RSD) of the responses factors. If the RSD is less than 35%, the RF can be assumed to be invariant and the average RF can be used for calculations. Alternatively, the results can be used to fit a linear or quadratic regression of response ratios, A_s/A_{is} , vs. concentration ratios C_s/C_{is} . If used, the regression must be weighted inversely proportional to concentration. The coefficient of determination (r^2) of the weighted regression must be greater than 0.920 (this value roughly corresponds to the RSD limit of 35%). Alternatively, the relative standard error (RSE) may be used as an acceptance criterion. As with the RSD, the RSE must be less than 35%. If an RSE less than 35% cannot be achieved for a quadratic regression, system performance is unacceptable and the system must be adjusted and re-calibrated.

All Published Methods: Reference spectra must be updated upon analysis of each new calibration curve.

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.

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 $\pm 40\%$ 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.



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STATE NOTE: When analyzing samples in conjunction with the Ohio VAP or South Carolina DHEC programs, the calibration model must be RSD or linear. Quadratic curve modeling is not permitted unless historical performance of analytes exhibited a nonlinear response (i.e., Benzoic Acid and problematic phenols). Quadratic models cannot be used to extend the calibration range or bypass instrument maintenance.

8.4.2 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.

- 8.4.3 **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%~~ 50% of the true standard concentration.

STATE NOTE: For the analysis of South Carolina samples, all target analytes, including 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.

- 8.4.4 Quadratic Regression: Quadratic regression may be used for the following compounds: Pentachlorophenol, 4-Nitrophenol, 2,3,4,6-Tetrachlorophenol, 4,6-Dinitro-2-methylphenol, 2,4-Dinitrophenol. Plots must have a minimum of 6 points and a correlation coefficient of 0.995 or better.

STATE NOTE: Quadratic curve modeling is not permitted for samples originating in South Carolina or for samples reported for the Ohio VAP unless historical performance of analytes exhibited a nonlinear response (e.g., Benzoic Acid).

- 8.4.5 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.



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8.4.6 Daily Tuning and Continuing Calibration

As with the initial calibration, the system must be tuned with 50ng of DFTPP or less 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 and every 12 hours for 8270C/D and every 24 hours for EPA 625 (see the method note in Section 8.2 for Method 625.1 requirements).

EPA Method 8270C: The percent difference of the CCCs (see Table 8.3.1a & b) in the mid-level standard must be $\leq 20\%$ 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 625.1: The RF or calibration curve must be verified immediately after calibration and at the beginning of each 12-hour shift, by analysis of a standard at or near the concentration of the mid-point calibration standard. The standard(s) must be obtained from a second manufacturer or a manufacturer's batch prepared independently from the batch used for calibration. Include the surrogates in this solution. It is necessary to verify calibration for the analytes of interest only.

Compare the recoveries for the analytes of interest against the acceptance criteria for recovery (Q) in Attachment VII and the recoveries for surrogates against the acceptance criteria in Attachment VIII. If recovery of the analytes of interest and surrogates meet acceptance criteria, system performance is acceptable and analysis of samples may continue. If any individual recovery is outside its limit, system performance is unacceptable for that analyte.

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 ($\pm 20\%$). 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 $\pm 50\%$ criteria for the CCV.

EPA 8270C GC/MS SIM: When analyzing samples using SW-846 EPA 8270C SIM, all compounds in the CCV must be treated as CCCs and must meet the minimum requirements of $\leq 20\%$ difference.

EPA 8270D GC/MS SIM: If analyzing samples by EPA 8270D SIM, follow the initial calibration criteria for the specified referenced method as found in section 8.3.6 (EPA Method 8270D below).

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 $\leq 20\%$. When using regression fit calibration, the percent drift of the CCV



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must be $\leq 20\%$. 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 $+100\%$.

8.4.7 For corrective action regarding tuning and calibration, see sections 11.1 and 11.2.

8.5 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.

8.6 GC/MS analysis of field samples and preparation QC.

8.6.1 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.

8.6.2 Allow the extracts to warm to room temperature. Just prior to analysis, add $10\mu\text{L}$ of the internal standard solution to the 1mL concentrated extract or $5\mu\text{L}$ of the internal standard solution to the 0.5mL extract obtained from sample preparation.

8.6.3 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 $8\text{ng}/\mu\text{L}$, unless a more sensitive GC/MS system is being used). For example, if performing a $1:10$ dilution on a concentrated extract, take $100\mu\text{L}$ of the extract and dilute to a volume of 1mL with the appropriate solvent. Add $9\mu\text{L}$ of the appropriate internal standard solution to the diluted extract and inject on the analytical system. It can be assumed that $1\mu\text{L}$ of internal standard was contained in the $100\mu\text{L}$ extract used for the initial dilution.

8.6.4 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 $\pm 0.06\text{RRT}$ 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 $\pm 40\%$ of the expected concentration. See section 11.10 for additional information.

8.7 Qualitative Identification

8.7.1 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.



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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.

8.7.1.1 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.

8.7.1.2 The RRT of the sample component is within ± 0.06 RRT units of the RRT of the standard component.

8.7.1.3 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.

8.7.1.4 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.

8.7.1.5 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.

8.7.1.6 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.

8.7.1.7 Absolute retention times are used for compound identification in all GC methods that do *not* employ internal standard calibration. Retention time windows are established to compensate for minor shifts in absolute retention times as a result of sample loadings and normal



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chromatographic variability. The width of the retention time window should be carefully established to minimize the occurrence of both false positive and false negative results. Tight retention time windows may result in false negatives and/or may cause unnecessary reanalysis of samples when surrogates or spiked compounds are erroneously not identified. Overly wide retention time windows may result in false positive results that may not be confirmed.

- 8.7.1.7.1 Before establishing retention time windows, make sure that the chromatographic system is operating reliably and that the system conditions are optimized for the target analytes and surrogates in the sample matrix to be analyzed. Make three injections of all standard mixtures over the course of a 72-hour period. Serial injections or injections over a period of less than 72 hours may result in retention time windows that are too tight.
- 8.7.1.7.2 Record the retention time (in minutes) for each single component analyte and surrogate to three decimal places. Calculate the mean and standard deviation of the three absolute retention times for each single component analyte and surrogate. For multi-component analytes, choose three to five major peaks (see the determinative methods for more details) and calculate the mean and standard deviation of those peaks.
- 8.7.1.7.3 If the standard deviation of the retention times for a target compound is 0.000 (i.e., no difference between the absolute retention times), then either collect data from additional injections of standards or use a default standard deviation of 0.01 minutes.
- 8.7.1.7.4 The width of the retention time window for each analyte, surrogate, and major constituent in multi-component analytes is defined as ± 3 times the standard deviation of the mean absolute retention time established during the 72-hour period or 0.03 minutes, whichever is greater.
- 8.7.1.7.5 Establish the center of the retention time window for each analyte and surrogate by using the absolute retention time for each analyte and surrogate from the calibration verification standard at the beginning of the analytical shift. For samples run during the same shift as an initial calibration, use the retention time of the mid-point standard of the initial calibration.
- 8.7.1.7.6 Calculate absolute retention time windows for each analyte and surrogate on each chromatographic column and instrument. New retention time windows must be established when a new GC column is installed or if a GC column has been shortened during maintenance.



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8.8 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 $\pm 20\%$. (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.
- 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 80 for tentative identifications and a minimum concentration of 10ppb. Peaks below a Q value of 80 but above 10ppb are reported as “Unknown”. Any identified peaks below 10ppb are removed as these could result from baseline noise or other interferences, not necessarily attributable to the field sample or reliably quantifiable using GCMS technology. Additionally, any peaks that are attributable to instrument contamination (i.e., siloxanes) are also removed.

8.9 Quantitative analysis

8.9.1 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.

8.9.1.1 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*.

DOD samples must include a reason for each integration performed on the manual integration documentation.



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- 8.9.2 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.
- 8.9.3 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.
- 8.9.4 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.
- 8.9.5 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 8081 or 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.
- 8.9.6 **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, but not <70%. Resolution must be adequate at lower levels and not worsen as concentration increases.
- 8.9.7 Indeno(1,2,3-cd)pyrene and dibenz(a,h)anthracene share a similar structure and physical properties. Under routine analytical production conditions it is very difficult to achieve resolved chromatographic separation. The mass-spectra of these compounds exhibit base peaks separated by 2 AMUs (276 and 278 respectively) and these unique ions are used for quantitation of the respective compounds as defined by Method 8270. It has been found that the major base ion, 276, for indeno(1,2,3-cd)pyrene includes a significant contribution from dibenz(a,h)anthracene when the targets are present together at equal concentrations; however, indeno(1,2,3-cd)pyrene presence *does not* contribute significant ion 278 abundance to dibenz(a,h)anthracene quantitation at equal concentrations. For these reasons when dibenz(a,h)anthracene is found to be present at similar or lesser concentrations than indeno(1,2,3-cd)pyrene, the results are normalized by the calibration conditions and considered to be non-impacted. Alternatively, when dibenz(a,h)anthracene is found to be present at relatively greater concentrations than indeno(1,2,3-cd)pyrene, the indeno(1,2,3-cd)pyrene results are considered to be elevated and may be confirmed by a secondary acquisition and analysis utilizing a technique for chromatographic separation of the targets. Concentrations shall be considered similar up to a factor of two.

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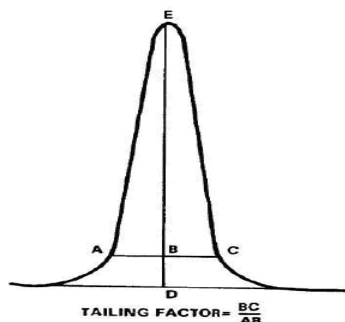
9.0 DATA ANALYSIS AND CALCULATIONS

9.1 GC/MS Tune: DDT Breakdown Determination during Tuning:

$$\% \text{ breakdown of DDT} = \frac{\text{sum of degradation peak areas (DDD + DDE)}}{\text{sum of all peak areas (DDT + DDE + DDD)}} \times 100$$

9.2 GC/MS Tune: Benzidine and Pentachlorophenol Tailing Factor

$$\text{Tailing Factor} = \frac{BC}{AB}$$



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)

$$\overline{RF} = \frac{\sum_{i=1}^n RF_i}{n} \quad SD = \sqrt{\frac{\sum_{i=1}^n (RF_i - \overline{RF})^2}{n-1}} \quad RSD = \frac{SD}{\overline{RF}} \times 100\%$$

where:

- $\overline{RSD}$ = Relative standard deviation.
- $\overline{RF}$ = Mean of 5 initial RFs for a compound.



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SD = Standard deviation of average RFs for a compound.

- Concentration of an analyte in an extract using RF (on column):

$$X_s = \frac{(Conc_{ISld})(Area_{Analyte})}{(Average RF_{analyte})(Area_{ISld})}$$

where:

X_s = Calculated raw concentration of analyte (in ppb)

- Quantitation Report Multiplier"

$$M_a = \frac{(V_t)(D)}{(V_s)} \quad \text{or} \quad M_s = \frac{(V_t)(D)}{(W_s)}$$

where:

M_a = Quantitation Report Multiplier for Aqueous Samples

M_s = Quantitation Report Multiplier for Solid Samples

V_t = Total volume of concentrated extract (in mL)

D = Dilution factor. If no dilution, D=1. Always dimensionless

V_s = Volume of aqueous sample extracted (in mL)

W_s = Weight sample extracted (in grams)

- Sample concentration by volume (ug/L) for aqueous samples:

$$\text{Concentration in } \frac{\mu\text{g}}{\text{L}} = (X_s)(M_a)$$

- Sample concentration by weight (ug/kg) for solid samples and non-aqueous liquids:

$$\text{Concentration in } \frac{\mu\text{g}}{\text{kg}} = \frac{(X_s)(M_s)}{(\%S)}$$

where:

%S = Percent solids expressed as a decimal

9.4 Relative Retention Time (RRT):

$$RRT = \frac{RT \text{ of Target Analyte}}{RT \text{ of Internal Standard}}$$

9.5 Percent Error (%Error)

$$\%Error = \frac{x_i - x'_i}{x_i} * 100$$



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where:

x'_i = Measured amount of analyte at the calibration level i , in mass or concentration units

x_i = True amount of analyte at calibration level i , in mass or concentration units

- 9.6 Relative Standard Error (%RSE) – As an alternative to using the average response factor when using Method 625.1, the quality of the calibration may be evaluated using the Relative Standard Error (RSE). The acceptance criterion for the RSE is the same as the acceptance criterion for Relative Standard Deviation (RSD), in the method. RSE is calculated as:

$$\%RSE = 100 \times \frac{\sum_{i=1}^n \left[\frac{x'_i - x_i}{x_i} \right]^2}{(n - p)}$$

where:

x'_i = Calculated concentration at level i

x_i = Actual concentration of the calibration level i

n = Number of calibration points

p = Number of terms in the fitting equation (average = 1; linear = 2; quadratic = 3)

- 9.7 See the current Quality Assurance Manual for other equations associated with common calculations.

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.

10.1.1 Method 625.1 Demonstration of Capability (DOC) Requirements

10.1.1.1 For the DOC, a QC check sample (LCS) concentrate containing each analyte of interest is prepared in a water miscible solvent. The QC check sample concentrate must be prepared independently from those used for calibration, but may be from the same source as the second-source standard used for calibration verification. The concentrate should produce concentrations of the analytes of interest in water at the midpoint of the calibration range, and may be at the same concentration as the LCS.

10.1.1.2 Prepare four QC check samples by adding an appropriate volume of the concentrate and of the surrogate(s) to each of four aliquots of reagent water and mix well. The volume of reagent water must be the same as the volume that will be used for the sample, blank, and MS/MSD.

10.1.1.3 Extract and analyze the four LCSs samples.



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- 10.1.1.4 Calculate the average percent recovery ($\bar{X}$) and the standard deviation (s) of the percent recovery for each analyte using the four results.
- 10.1.1.5 For each analyte, compare s and $\bar{X}$ with the acceptance criteria for precision and recovery presented in Attachment VII. For analytes that are not listed, QC acceptance criteria must be developed by the laboratory.

If s and $\bar{X}$ for all analytes of interest meet the acceptance criteria, system performance is acceptable and analysis of blanks and samples can begin. If any individual s exceeds the precision limit or any individual $\bar{X}$ falls outside the range for recovery, system performance is unacceptable for that analyte.
- 10.1.1.6 When one or more of the analytes tested fail at least one of the acceptance criteria, repeat the test for only the analytes that failed. If results for these analytes pass, system performance is acceptable and analysis of samples and blanks may proceed. If one or more of the analytes again fail, system performance is unacceptable for the analytes that failed the acceptance criteria. Correct the problem and repeat the test.
- 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 method blank, 1 Laboratory Control Sample (LCS), 1 Laboratory Control Sample Duplicate (LCSD), 1 Matrix Spike/Spike Duplicate (MS/MSD) (if client has supplied sufficient sample volume). All batch information must be maintained in the preparation documentation assigned to the department.
- 10.4 For sample analyzed per the requirements of Method 8000D, the LLOQ (see Section 1.8.2) must be verified at least annually, and whenever significant changes are made to the preparation and/or analytical procedure, to demonstrate quantitation capability at lower analyte concentration levels
 - 10.4.1 The LLOQ verification (to be performed after the initial calibration) is prepared by spiking a clean control material with the analyte(s) of interest at 0.5-2 times the LLOQ concentration level(s).
 - 10.4.2 The LLOQ check is carried through the same preparation and analytical procedures as environmental samples and other QC samples.
 - 10.4.3 It is recommended to analyze the LLOQ verification on every instrument where data is reported; however, at a minimum, the lab must rotate the verification among similar analytical instruments such that all are included within 3 years.
 - 10.4.4 Recovery of target analytes in the LLOQ verification must be within established in-house limits or within other such project-specific acceptance limits to demonstrate acceptable method performance at the LLOQ. Until the laboratory has sufficient data to determine acceptance limits, the LCS criteria $\pm 20\%$ (i.e.,



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lower limit minus 20% and upper limit plus 20%) may be used for the LLOQ acceptance criteria.

- 10.5 For acceptance criteria for calibration standards, QC samples and field samples and corrective actions, see section 11.0.

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 using the total ion chromatograms for each. 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 625.1: 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 models cannot be used to extend the calibration range or bypass instrument maintenance. If the method criteria are 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.

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 (0.995) for alternate curve fits, then the chromatographic system is considered too



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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.

TNI: If the ICV or CCV results obtained are outside the established acceptance criteria and analysis of a second consecutive (immediate) calibration verification fails to produce results within acceptance criteria, corrective actions shall be performed. The laboratory shall demonstrate acceptable performance after the final round of corrective action with two consecutive calibration verifications, or a new initial instrument calibration shall be performed.

Method 8000D: To determine calibration function acceptability, refit the initial calibration data back to the calibration model and calculate %Error (see Section 9.5). Percent error between the calculated and expected amounts of an analyte must be $\leq 30\%$ for all standards. ($\leq 50\%$ is acceptable for the lowest calibration point.)

Method 625.1: The RF or calibration curve must be verified immediately after calibration and at the beginning of each 12-hour shift, by analysis of a standard at or near the concentration of the mid-point calibration standard. The standard(s) must be obtained from a second manufacturer or a manufacturer's batch prepared independently from the batch used for calibration.

When one or more analytes fail acceptance criteria, analyze a second aliquot of the calibration verification standard and compare ONLY those analytes that failed the first test with their respective acceptance criteria. If these analytes now pass, system performance is acceptable and analysis of samples may continue. A repeat failure of any analyte that failed the first test, however, will confirm a general problem with the measurement system. If this occurs, repair the system and repeat the test, or prepare a fresh calibration standard and repeat the test. If calibration cannot be verified after maintenance or injection of the fresh calibration standard, re-calibrate the instrument.

- 11.3 The method blank must be extracted and analyzed with each set of samples at a frequency of at least 5% and must be free of the analytes of interest at the method detection limit. If the method blank contains target analytes at a detectable concentration, 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. A passing method blank must be analyzed before any samples are analyzed; otherwise 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 (see the LIMS). 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.

General guidelines for qualifying sample results with regard to method blank quality are as follows:

- If the method blank concentration is less than the MDL and sample results are greater than the RL, then no qualification is required.



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- No qualification is necessary when an analyte is detected in the method blank but not in the associated samples.
- If the concentration in a sample is more than ten times the concentration in the method blank, then no qualification is required.
- If the method blank concentration is greater than the MDL but less than the RL and sample results are greater than the MDL, then qualify associated sample results to indicate that analyte was detected in the method blank.
- If the method blank concentration is greater than the RL, further corrective action and qualification is required. An analyst should consult their supervisor for further instruction.

11.4 Second Source Calibration Verification

Method 8270D: The value determined from the second source calibration verification (SSCV) should be within $\pm 30\%$ drift 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/SM6410B: The value determined from the second source calibration verification (SSCV) must be $\leq 50\%$ drift for non-CCC compounds; $\leq 20\%$ drift 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.

STATE NOTE: If the samples are analyzed in conjunction with South Carolina DHEC, alternate recovery limits can only be used if they are more stringent than method criteria.

Method 625.1: Method 625.1 utilizes CCV limits for evaluation.

- 11.5 Surrogates: If the surrogate recoveries in the samples do not fall within the appropriate acceptance criteria presented in the LIMS, ensure that there were no errors in calculations, internal standard, or instrument performance. If the recovery of any one surrogate is critically low (, then the sample must be re-extracted unless otherwise directed by the client or a clear, documented matrix interference is exhibited. If two of three acid and two of three base/neutral surrogates are within acceptance criteria, then the sample may be reported. If surrogate recoveries present a high bias, and samples are BDL, then data is not impacted and may be reported. If re-extraction is required and 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% or greater than 200% recovery, and matrix interferences are not confirmed as the cause of the failure, the sample must be re-extracted.



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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 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.

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. ISTD failures <50% of daily ICV may be reported if all corresponding analytes are BDL as the high quantitation bias created by the reduced internal standard recovery has not adversely impacted the reported analyte 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 acceptance criteria given in the LIMS. 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 exceedance 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



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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 exceedances must be random events.

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

Number of allowable marginal exceedances:

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 the LIMS, 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 or Ohio VAP samples, marginal exceedances 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 evaluated within 70-130% and the MS/MSD recoveries must be within in-house derived recovery limits; however, if the limits given in Method 625 Table 6 are more stringent, then those limits must be used. The following are the current limits:

Parameter	LCS/LCSD	MS/MSD
1,2,4-TRICHLOROBENZENE	70 - 130%	44 – 104%
2,4,6-TRICHLOROPHENOL	70 - 130%	37 - 132%
2,4-DICHLOROPHENOL	70 - 130%	39 - 117%
2,4-DIMETHYLPHENOL	70 - 119%	32 - 119%
2,4-DINITROPHENOL	70 - 130%	10 - 141%
2,4-DINITROTOLUENE	70 - 130%	45.4 - 139%
2,6-DINITROTOLUENE	70 - 130%	50 - 134%
2-CHLORONAPHTHALENE	70 - 118%	60 - 118%
2-CHLOROPHENOL	70 - 130%	23 - 111%
2-NITROPHENOL	70 - 130%	29 - 135%
3,3-DICHLOROBENZIDINE	70 - 130%	10 - 143%
4,6-DINITRO-2-METHYLPHENOL	70 - 130%	10 - 143%
4-BROMOPHENYL-PHENYLETHER	70 - 127%	53 - 127%
4-CHLORO-3-METHYLPHENOL	70 - 130%	38.4 - 123%
4-CHLOROPHENYL-PHENYLETHER	70 - 130%	49.8 - 127%
4-NITROPHENOL	70 - 130%	10 - 52.8%
ACENAPHTHENE	70 - 130%	47 - 141%
ACENAPHTHYLENE	70 - 130%	40 - 132%
ANTHRACENE	70 - 130%	44.5 - 130%
BENZO(A)ANTHRACENE	70 - 130%	46.4 - 130%
BENZO(A)PYRENE	70 - 130%	34.6 - 129%



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Parameter	LCS/LCSD	MS/MSD
BENZO(B)FLUORANTHENE	70 - 130%	36.3 - 137%
BENZO(G,H,I)PERYLENE	70 - 130%	10 - 140%
BENZO(K)FLUORANTHENE	70 - 130%	30.3 - 136%
BENZYL BUTYL PHTHALATE	70 - 130%	44.8 - 152%
BIS(2-CHLOROETHOXY)METHANE	70 - 130%	39.2 - 128%
BIS(2-CHLOROETHYL)ETHER	70 - 130%	14.8 - 131%
BIS(2-CHLOROISOPROPYL)ETHER	70 - 130%	36 - 117%
BIS(2-ETHYLHEXYL)PHTHALATE	70 - 130%	12.6 - 153%
CHRYSENE	70 - 130%	40 - 133%
DIBENZ(A,H)ANTHRACENE	70 - 130%	10 - 143%
DIETHYL PHTHALATE	70 - 114%	50.4 - 114%
DIMETHYL PHTHALATE	70 - 112%	9.1 - 112%
DI-N-BUTYL PHTHALATE	70 - 118%	53.3 - 118%
DI-N-OCTYL PHTHALATE	70 - 130%	13.3 - 146%
FLUORANTHENE	70 - 130%	42.9 - 137%
FLUORENE	70 - 121%	59 - 121%
HEXACHLORO-1,3-BUTADIENE	70 - 116%	28.9 - 116%
HEXACHLOROBENZENE	70 - 130%	47 - 121%
HEXACHLOROCYCLOPENTADIENE	70 - 130%	10 - 128%
HEXACHLOROETHANE	70 - 113%	40 - 109%
INDENO(1,2,3-CD)PYRENE	70 - 130%	10 - 141%
ISOPHORONE	70 - 130%	31.9 - 118%
NAPHTHALENE	70 - 130%	29 - 115%
NITROBENZENE	70 - 130%	35 - 118%
N-NITROSODI-N-PROPYLAMINE	70 - 130%	35.4 - 129%
PENTACHLOROPHENOL	70 - 130%	14 - 128%
PHENANTHRENE	70 - 130%	54 - 120%
PHENOL	70 - 112%	10 - 55.7%
PYRENE	70 - 115%	52 - 115%

Method 625.1: For LCS analyses, repeat the test only for those analytes that failed to meet the acceptance criteria (PS). If these analytes now pass, system performance is acceptable and analysis of blanks and samples may proceed. Repeated failure, however, will confirm a general problem with the measurement system. If this occurs, repeat the test using a fresh LCS or an LCS prepared with a fresh QC check sample concentrate, or perform and document system repair. Subsequent to analysis of the LCS prepared with a fresh sample concentrate, or to system repair, repeat the LCS test. If failure of the LCS indicates a systemic problem with samples in the batch, re-extract and re-analyze the samples in the batch.

The large number of analytes tested in performance tests in this method present a substantial probability that one or more will fail acceptance criteria when many analytes are tested simultaneously, and a retest is allowed if this situation should occur. If, however, continued re-testing results in further repeated failures, the laboratory must document and report the failures (e.g., as qualifiers on results), unless the failures are not required to be reported as determined by the regulatory/control authority. Results associated with a QC failure for an analyte regulated in a discharge cannot be used to demonstrate regulatory compliance. QC failures do not relieve a discharger or permittee of reporting timely results.



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NOTE: To maintain the validity of the test and re-test, system maintenance and/or adjustment is not permitted between the pair of tests.

For MS/MSD analyses, compare the percent recoveries (P1 and P2) and the RPD for each analyte in the MS/MSD aliquots with the corresponding QC acceptance criteria in Attachment VII. A laboratory may develop and apply QC acceptance criteria more restrictive than the criteria in Attachment VII, if desired.

If any individual P falls outside the designated range for recovery in either aliquot, or the RPD limit is exceeded, the result for the analyte in the unspiked sample is suspect. The large number of analytes tested in performance tests in this method present a substantial probability that one or more will fail acceptance criteria when many analytes are tested simultaneously, and a retest is allowed if this situation should occur. If, however, continued re-testing results in further repeated failures, the laboratory must document and report the failures (e.g., as qualifiers on results), unless the failures are not required to be reported as determined by the regulatory/control authority. Results associated with a QC failure for an analyte regulated in a discharge cannot be used to demonstrate regulatory compliance. QC failures do not relieve a discharger or permittee of reporting timely results.

- 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 concentrations consistent with the calibration standards.

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 $\pm 40\%$ of the expected concentration. If this criterion 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.

- 11.9 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.
- 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.



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- 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 surrogate recoveries must be checked to ensure that they are within QC acceptance criteria or that corrective action has occurred.
- 11.10.3 Blanks must be free of all interfering peaks.
- 11.10.4 Quality control criteria must be checked for the LCS, LCSD, MS, and MSD.
- 11.10.5 Data must be checked to determine the need for appropriate flags. Comments are noted when results are flagged.
- 11.10.7 All manual integrations must be verified through checking the before/after shot of the sample, method blank, and/or QC (LCS/LCSD/MS/MSD).
- 11.10.8 See SOP #030201, *Data Handling and Reporting* and SOP #030227, *Data Review*.

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, *Environmental Sustainability & 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 concentrated soil or 1L water extractions and calibration levels for EPA 3511 extracted water samples and non-concentrated Soil range from 5-200ppm from a 200ppm intermediate. Retention time windows are set using C₁₀, C₂₁, and C₃₅. For DRO, set RT 0.1 minutes after C₁₀ to 0.1 minutes after C₂₁. For ORO, set RT 0.1 minutes after C₂₁ to 0.1 minutes after C₃₅. 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 (C₁₀-C₂₁) **must** be used for C₂₁-C₃₅. 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.
- 13.4 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/625method. The reduction in volume extracted along with increased sensitivity at detection and/or analysis of the resulting extract using large volume injection (>5uL) on each GCMS allows for low



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detection limits typical of those obtained using a 1L extraction. 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.

- 13.5 **Low level NDMA and 1,4-Dioxane by SIM scan/isotope dilution.** Tuning and frequency requirements are the same as 8270C. Extract samples the same as 8270BNA using the appropriate extraction method. 250ng of N-nitrosodimethylamine-d₆ or 1,4-Dioxane-d₈ is added to each sample per every 0.5mL of final extract volume prior to extraction resulting in a true value of 500ppb in extract. Only base/neutral surrogates monitoring is necessary. GC/MS is set to scan for masses 42, 43, 46, 48, 54, 74, 80, 82, 115, 128, 150 and 152 for NDMA-d₆ or masses 57, 58, 62, 64, and 88 for 1,4-Dioxane-d₈ in SIM mode. Calibrate at least 5 points using 8270BNA mega mix or 1,4-Dioxane ICAL standard. 500ng of N-nitrosodimethylamine-d₆ is added per every 1mL of calibration standard to each level of the calibration resulting in a true value of 500ppb. Calibration standards range from 5ppb to 10,000ppb for 3510RV extracted water samples. Quantitate using Chemstation auto-integration software unless a significant discrepancy is noted in which case manually adjust integrations to best represent the calibration. Select ion monitoring should be used for acquisition and quantitation. NDMA and 1,4-Dioxane are quantitated using the isotope dilution method as described in 8000C. The %RSD determined for NDMA RFs **must** be <15% in order to use the average of response factors for quantitation, otherwise linear regression is to be used. Run a DFTPP tune and CCV every 12 hours near mid-point of calibration, %Diff must be <20% for the calibration to be deemed in control and sample analysis to proceed. Run a method blank, LCS and LCSD with every extraction batch. MS/MSDs will be processed with batches when requested by the client as matrix spiking and duplication does not yield reliable precision data when analyzed by the isotope dilution method. Quantitation of low level NDMA and 1,4-Dioxane should be performed using the isotope dilution process.

14.0 REFERENCES

- 14.1 *Semivolatile Organic Compounds by Gas Chromatography/Mass Spectrometry (GC/MS)*, SW846 Method 8270C, Revision 3, December 1996.
- 14.2 *Semivolatile Organic Compounds by Gas Chromatography/Mass Spectrometry (GC/MS)*, SW846 Method 8270D, Revision 4, February 2007.
- 14.3 *Determinative Chromatographic Separations*, SW846 Method 8000B, Revision 2, December 1996.
- 14.4 *Determinative Chromatographic Separations*, SW846 Method 8000C, Revision 3, March 2003.
- 14.5 *Determinative Chromatographic Separations*, SW846 Method 8000D, Revision 4, July 2014.
- 14.6 *Base/Neutrals and Acids*, 40 CFR Part 136, Appendix A, EPA Method 625, October 1991.
- 14.7 *Extractable Base/Neutrals and Acids*, Standard Methods for the Examination of Water and Wastewater, Method 6410B-2000.



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- 14.8 *Extractable Base/Neutrals and Acids*, Standard Methods for the Examination of Water and Wastewater, Method 6410B-1997 (20th Ed).
- 14.9 *Base/Neutrals and Acids by GC/MS*, EPA Method 625.1, Federal Register, Volume 82, Number 165, August 28, 2017.

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Attachment I: Revision History**Current Version (Pace National):**

Date	Description of Revisions
9/19/2019	Technical and quality review and revision. Additional update in response to SC DHEC 8/29/2019 technical review letter. Replaced existing header and footer with corporate header and footer. Revised Sections 4.7, 7.61 through 7.6.24, 7.7, 7.8 and all subsections, 7.9, 7.10 and all subsections, 8.2, 8.4.1, 8.4.3, 8.4.6, 10.5, 11.1, 11.2, 11.3, 11.4, 11.5, 11.6, and 11.7. Deleted all subsections of 10.5 as well as Sections 11.10.2, 11.10.7, 11.10.9, 11.10.10, 11.10.11, 11.11 and all subsections, 13.2, 13.3, 13.4, and re-numbered as appropriate.

Superseded Versions (ESC Lab Sciences SOP #330345):

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.
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.
16	3/26/13	Technical and Quality Review and update. Revised title, Attachment IV and sections 2.1, 7.8, 7.9, 7.10, 8.0 (state note), 8.3, 11.6, and 13.1; Added sections 7.13, 14.6 and state notes in sections 1.0 and 11.4; Removed sections 7.10.5 and 8.5.3.

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Version	Date	Description of Revisions
17	6/10/14	Technical and Quality Review and update. Revised Attachments II, IVc through IVk, and V along with sections 5.3, 8.3.1, 8.3.6; 11.7; Added sections 5.4.7, 7.5.10 through 7.5.13, 7.7, 8.3.4; Removed sections 6.1.4 and 11.11.2.
18	8/14/14	Technical and Quality Review and update. Added sections 2.35, 7.5.14, and 13.6.
19	11/17/2015	Technical and quality review and update. Header and signature block re-formatting. Revised Sections 1.5, 1.8.1, 5.3, 7.9.1, 7.11.1, 8.1.1, 8.4.1, 8.4.4, 8.4.6, 8.7.1, 8.8, 8.9.1.1, 11.3, 11.5, 11.7, 11.11.4, 11.11.5, and 13.5. Added Section 8.6.1.7, 8.6.1.7.1 through 8.6.1.7.6, and Attachment VI. Deleted Attachment Iva, Attachment IVb, Attachment IVc, Attachment IVd, Attachment IVe, Attachment IVf, Attachment IVg, Attachment IVh, Attachment IVi, Attachment IVj, and Attachment IVk.
20	4/1/2016	Technical and quality review and update. Revised Sections 1.8.1, 2.8, 2.9, 2.10, 4.8, 7.1, 9.4, 9.5, 11.2, 11.4, 11.11.7, 12.2, and 13.6. Deleted Sections 2.11 through 2.35 and 9.6 through 9.13. Added Sections 7.5.16, 7.5.17, 7.5.18, and 8.9.7.
21	10/24/16	Technical and quality review and update to satisfy the requirements of SC DHEC (see correspondence dated 6/24/16) Header and signature block re-formatting. Revised Sections 11.2, 11.3, 11.4, 11.7, 14.1, 14.2, 14.3, 14.4, 14.6, 14.7, 14.8, and Attachment VII Table 2. Added Sections 1.8.2, 2.10, 9.5, 10.4 and all subsections, and 11.11.7.1.
22	6/19/2017	Technical and quality review and update. Revised Sections 1.8, 3.1, 7.5.14, 7.8, 7.11.1, 8.4.1, 8.8, 9.3, 10.3, 11.7, and Attachment III.
23	7/10/2017	Technical and quality review and update. Revised Sections 2.1, 2.11, 2.12, 7.9.2, 7.10.2, 7.10.3, 7.10.4, 7.11.4, 7.11.5, 9.3, 13.1, 13.5
24	10/6/2017	Technical and quality review and update. Revised Sections 7.9.2, 11.5, and Attachment IV. Added Table 8 in Attachment VII.
25	11/29/2017	Update in response to A2LA audit finding CAR2872. Revised Attachment IV.
26	3/22/2018	Technical and quality review and update. Added 625.1 requirements. Revised SOP Title and Sections 1.8.1, 2.11, 8.2, 8.4.1, 8.4.6, 9.7, 10.6, 11.1, 11.2, 11.3, 11.4, 11.5, 11.6, and 11.7. Added Sections 2.12, 9.6, 10.1.1 and all subsections, 10.5 and all subsections, 14.9, Attachment VII, and Attachment VIII.

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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
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

Analyte	Water mg/L	Soil mg/Kg
Phenanthrene	0.001	0.033
Benzylbutyl phthalate	0.003	0.033
Bis(2-ethylhexyl)phthalate	0.003	0.033
Di-n-butyl phthalate	0.003	0.033
Diethyl phthalate	0.003	0.033
Dimethyl phthalate	0.003	0.033
Di-n-octyl phthalate	0.003	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
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

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**STANDARD OPERATING PROCEDURE**

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ISSUER: Pace National – Mt. Juliet, Tennessee

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Analyte	Water mg/L	Soil mg/Kg
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
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	0.33
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

Analyte	Water mg/L	Soil mg/Kg
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-Triethylphosphorothioate	0.05	1.65
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
Pentachlorophenol (SIM)	0.001	-
Sulfolane	0.0002	0.33
Mirex	0.02	NA
Dicofol	0.02	NA
Quinoline	0.05	0.33

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ISSUER: Pace National – Mt. Juliet, Tennessee

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Analyte	Water mg/L	Soil mg/Kg
Indene	NA	0.33
Benzenethiol	0.02	3.3

*Alternate reporting levels may be possible using different technologies (i.e. SIM, LVI, etc.). Please contact the laboratory for additional information.

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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
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

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ISSUER: Pace National – Mt. Juliet, Tennessee

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ANALYTE:	3510*	3520	3540/3541	3550*	3580*	CAS #(a)
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-phenylenediamine	X	X	ND	ND	ND	95-83-0
4-Chloro-1,3-phenylenediamine	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
Crotoxypfos	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
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

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ISSUER: Pace National – Mt. Juliet, Tennessee

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ANALYTE:	3510*	3520	3540/3541	3550*	3580*	CAS #(a)
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
3,4-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
3,4-Dimethylbenzidine	X	ND	ND	ND	X	119-93-7
α,α-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
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
Fensulfthion	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

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ANALYTE:	3510*	3520	3540/3541	3550*	3580*	CAS #(a)
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
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

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ANALYTE:	3510*	3520	3540/3541	3550*	3580*	CAS #(a)
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
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 Pyrophosphoramidate	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,HE(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	DC, OE(10)	ND	ND	ND	X	94-59-7
Safrole	X	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

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**STANDARD OPERATING PROCEDURE**

TITLE: ENV-SOP-MTJL-0081 Semivolatile Organics by GCMS using Capillary Column (EPA Methods 8270C, 8270D, 625, 625.1 and SM6410 B) Including Provisions for Analysis in SIM Mode

ISSUER: Pace National – Mt. Juliet, Tennessee

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ANALYTE:	3510*	3520	3540/3541	3550*	3580*	CAS #(a)
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
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) Non-reproducible 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.

**STANDARD OPERATING PROCEDURE**

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Attachment IV: 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
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

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Compound	Primary Characteristic Ion	Secondary Characteristic Ion(s)
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
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

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Compound	Primary Characteristic Ion	Secondary Characteristic Ion(s)
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
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

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ISSUER: Pace National – Mt. Juliet, Tennessee

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Compound	Primary Characteristic Ion	Secondary Characteristic Ion(s)
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
Mirex	272	274, 237,270
Kelthane (Dicofol)	251	139.111.253
Indene	115	116,117
Quinoline	129	128,130,102
Benzenethiol	110	109,66
Diphenyl Disulfide	218	109,65
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
2-Methylnaphthalene-d10 (surr)	152	150, 122, 151
Fluoranthene-d10 (surr)	212	208, 313, 210
PAH by SIM		
Naphthalene	128	129
2-Methylnaphthalene	142	141
1-Methylnaphthalene	142	141
2-Chloronaphthalene	162	127
Acenaphthylene	152	153, 151
Acenaphthene	153	154, 152, 151
Dibenzofuran	168	139
Fluorene	166	165
Phenanthrene	178	179
Anthracene	178	179, 176
Fluoranthene	202	203, 200
Pyrene	202	203, 200
Benzo(a)anthracene	228	226
Chrysene	228	226, 229
Benzo(b)fluoranthene	252	253
Benzo(k)fluoranthene	252	253
Benzo(a)pyrene	252	253
Indeno(1,2,3-cd)pyrene	276	277, 138
Dibenz(a,h)anthracene	278	279, 139, 138
Benzo(g,h,i)perylene	276	138

**STANDARD OPERATING PROCEDURE**

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Attachment V - 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
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

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Compound	Test conc. (µg/L)	Limit for s (µg/L)	Range for x (µg/L)	Range p, p(s) (%)
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.



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Attachment VI - 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
Hexachlorophene

**STANDARD OPERATING PROCEDURE**

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Attachment VII – Method 625.1 Criteria

Analyte	Range for Q (%)	Limit for s (%)	Range for $\bar{X}$ (%)	Range for P1, P2 (%)	Limit for RPD (%)
Acenaphthene*	70-130	29	60-132	47-145	48
Acenaphthylene*	60-130	45	54-126	33-145	74
Anthracene*	58-130	40	43-120	27-133	66
Benidine*					
Benzo(a)anthracene*	42-133	32	42-133	33-143	53
Benzo(a)pyrene*	32-148	43	32-148	17-163	72
Benzo(b)fluoranthene*	42-140	43	42-140	24-159	71
Benzo(k)fluoranthene*	25-146	38	25-146	11-162	63
Benzo(ghi)perylene*	13-195	61	D-195	D-219	97
Benzyl butyl phthalate*	43-140	36	D-140	D-152	60
bis(2-Chloroethoxy)methane	52-164	32	49-165	33-184	54
bis(2-Ethylhexyl)phthalate*	43-137	50	29-137	8-158	82
bis(2-Chloroisopropyl) ether (2,2'-Oxybis[1-chloropropane])*	63-139	46	63-139	36-166	76
4-Bromophenyl phenyl ether*	70-130	26	65-120	53-127	43
2-Chloronaphthalene*	70-130	15	65-120	60-120	24
4-Chlorophenyl phenyl ether*	57-145	36	38-145	25-158	61
Chrysene*	44-140	53	44-140	17-168	87
Dibenz(a,h)anthracene*	13-200	75	D-200	D-227	126
Di-n-butylphthalate*	52-130	28	8-120	1-120	47
3,3'-Dichlorobenzidine*	18-213	65	8-213	D-262	108
Diethyl phthalate*	47-130	60	D-120	D-120	100
Dimethyl phthalate*	50-130	110	D-120	D-120	183
2,4-Dinitrotoluene*	53-130	25	48-127	39-139	42
2,6-Dinitrotoluene*	68-137	29	68-137	50-158	48
Di-n-octylphthalate*	21-132	42	19-132	4-146	69
Fluoranthene*	47-130	40	43-121	26-137	66
Fluorene*	70-130	23	70-120	59-121	38
Hexachlorobenzene*	38-142	33	8-142	D-152	55
Hexachlorobutadiene*	68-130	38	38-120	24-120	62
Hexachloroethane*	55-130	32	55-120	40-120	52
Indeno(1,2,3-cd)pyrene*	13-151	60	D-151	D-171	99
Isophorone*	52-180	56	47-180	21-196	93
Naphthalene*	70-130	39	36-120	21-133	65
Nitrobenzene*	54-158	37	54-158	35-180	62
N-Nitrosodi-n-propylamine*	59-170	52	14-198	D-230	
Phenanthrene*	67-130	24	65-120	54-120	39
Pyrene*	70-130	30	70-120	52-120	49
1,2,4-Trichlorobenzene*	61-130	30	57-130	44-142	50
4-Chloro-3-methylphenol	68-130	44	41-128	22-147	73
2-Chlorophenol	55-130	37	36-120	23-134	61
2,4-Dichlorophenol	64-130	30	53-122	39-155	50
2,4-Dimethylphenol	58-130	35	42-120	32-120	58
2,4-Dinitrophenol	39-173	79	D-173	D-191	132
2-Methyl-4,6-dinitrophenol	56-130	122	53-130	D-181	203

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Analyte	Range for Q (%)	Limit for s (%)	Range for $\bar{X}$ (%)	Range for P1, P2 (%)	Limit for RPD (%)
2-Nitrophenol	61-163	33	45-167	29-182	55
4-Nitrophenol	35-130	79	13-129	D-132	131
Pentachlorophenol	42-152	52	38-152	14-176	86
Phenol	48-130	39	17-120	5-120	64
2,4,6-Trichlorophenol	69-130	35	52-129	37-144	58
4-Chloro-3-methylphenol					
2-Chlorophenol					
2,4-Dichlorophenol					
2,4-Dimethylphenol					
2,4-Dinitrophenol					
2-Methyl-4,6-dinitrophenol					
2-Nitrophenol					
4-Nitrophenol					
Acetophenone					
2-Acetylaminofluorene					
1-Acetyl-2-thiourea					
Alachlor					
Aldrin	7-152	39	7-152	D-166	81
Ametryn					
2-Aminoanthraquinone					
Aminoazobenzene					
4-Aminobiphenyl					
3-Amino-9-ethylcarbazole					
Anilazine					
Aniline					
o-Anisidine					
Aramite					
Atraton					
Atrazine					
Azinphos-methyl					
Barban					
Benzanthrone					
Benzenethiol					
Benzoic acid					
2,3-Benzofluorene					
p-Benzoquinone					
Benzyl alcohol					
alpha-BHC					
beta-BHC	42-131	37	42-131	24-149	61
gamma-BHC (Lindane)					
delta-BHC	D-130	77	D-120	D-120	129
Biphenyl					
Bromacil					
2-Bromochlorobenzene					
3-Bromochlorobenzene					
Bromoxynil					

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Analyte	Range for Q (%)	Limit for s (%)	Range for $\bar{X}$ (%)	Range for P1, P2 (%)	Limit for RPD (%)
Butachlor					
Butylate					
n-C10 (n-decane)					
n-C12 (n-undecane)					
n-C14 (n-tetradecane)					
n-C16 (n-hexadecane)					
n-C18 (n-octadecane)					
n-C20 (n-eicosane)					
n-C22 (n-docosane)					
n-C24 (n-tetracosane)					
n-C26 (n-hexacosane)					
n-C28 (n-octacosane)					
n-C30 (n-triacontane)					
Captafol					
Captan					
Carbaryl					
Carbazole					
Carbofuran					
Carboxin					
Carbophenothion					
Chlordane 3,5					
bis(2-Chloroethyl) ether	52-130	65	43-126	12-158	108
Chloroneb					
4-Chloroaniline					
Chlorobenzilate					
Chlorfenvinphos					
4-Chloro-2-methylaniline					
3-(Chloromethyl)pyridine hydrochloride					
4-Chloro-2-nitroaniline					
Chlorpropham					
Chlorothalonil					
1-Chloronaphthalene					
3-Chloronitrobenzene					
4-Chloro-1,2-phenylenediamine					
4-Chloro-1,3-phenylenediamine					
2-Chlorobiphenyl					
Chlorpyrifos					
Coumaphos					
m + p-Cresol					
o-Cresol					
p-Cresidine					
Crotoxyphos					
2-Cyclohexyl-4,6-dinitro-phenol					
Cyanazine					
Cycloate					

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p-Cymene					
Dacthal (DCPA)					
4,4'-DDD	D-135	56	D-135	D-145	93
4,4'-DDE	19-130	46	19-120	4-136	77
4,4'-DDT	D-171	81	D-171	D-203	135
Demeton-O					
Demeton-S					
Diallate (cis or trans)					
2,4-Diaminotoluene					
Diazinon					
Dibenz(a,j)acridine					
Dibenzofuran					
Dibenzo(a,e)pyrene					
Dibenzothiophene					
1,2-Dibromo-3-chloropropane					
3,5-Dibromo-4-hydroxybenzonitrile					
2,6-Di-tert-butyl-p-benzoquinone					
Dichlone					
2,3-Dichloroaniline					
2,3-Dichlorobiphenyl					
2,6-Dichloro-4-nitroaniline					
2,3-Dichloronitrobenzene					
1,3-Dichloro-2-propanol					
2,6-Dichlorophenol					
Dichlorvos					
Dicrotophos					
Dieldrin 3	70-130	38	44-119	29-136	62
1,2:3,4-Diepoxybutane					
Di(2-ethylhexyl) adipate					
Diethylstilbestrol					
Diethyl sulfate					
Dilantin (5,5-Diphenylhydantoin)					
Dimethoate					
3,3'-Dimethoxybenzidine					
Dimethylaminoazobenzene					
7,12-Dimethylbenz(a)anthracene					
3,3'-Dimethylbenzidine					
N,N-Dimethylformamide					
3,6-Dimethylphenathrene					
alpha, alpha-Dimethylphenethylamine					
Dimethyl sulfone					
1,2-Dinitrobenzene					
1,3-Dinitrobenzene					
1,4-Dinitrobenzene					
Dinocap					

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Analyte	Range for Q (%)	Limit for s (%)	Range for $\bar{X}$ (%)	Range for P1, P2 (%)	Limit for RPD (%)
Dinoseb					
Diphenylamine					
Diphenyl ether					
1,2-Diphenylhydrazine					
Diphenamid					
Diphenyldisulfide					
Disulfoton					
Disulfoton sulfoxide					
Disulfoton sulfone					
Endosulfan I					
Endosulfan II					
Endosulfan sulfate	D-130	42	D-120	D-120	70
Endrin					
Endrin aldehyde	D-189	45	D-189	D-209	75
Endrin ketone					
EPN					
EPTC					
Ethion					
Ethoprop					
Ethyl carbamate					
Ethyl methanesulfonate					
Ethylenethiourea					
Etridiazole					
Ethynylestradiol-3-methyl ether					
Famphur					
Fenamiphos					
Fenarimol					
Fensulfothion					
Fenthion					
Fluchloralin					
Fluridone					
Heptachlor	D-172	44	D-172	D-192	74
Heptachlor epoxide	70-130	61	71-120	26-155	101
2,2',3,3',4,4',6-Heptachlorobiphenyl					
2,2',4,4',5',6-Hexachlorobiphenyl					
Hexachlorocyclopentadiene					
Hexachlorophene					
Hexachloropropene					
Hexamethylphosphoramide					
Hexanoic acid					
Hexazinone					
Hydroquinone					
Isodrin					
2-Isopropyl-naphthalene					
Isosafrole					

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Analyte	Range for Q (%)	Limit for s (%)	Range for $\bar{X}$ (%)	Range for P1, P2 (%)	Limit for RPD (%)
Kepone					
Leptophos					
Longifolene					
Malachite green					
Malathion					
Maleic anhydride					
Merphos					
Mestranol					
Methapyrilene					
Methoxychlor					
2-Methylbenzothiazole					
3-Methylcholanthrene					
4,4'-Methylenebis(2-chloroaniline)					
4,4'-Methylenebis(N,N-dimethylaniline)					
4,5-Methylenephenanthrene					
1-Methylfluorene					
Methyl methanesulfonate					
2-Methylnaphthalene					
Methylparaoxon					
Methyl parathion					
1-Methylphenanthrene					
2-(Methylthio)benzothiazole					
Metolachlor					
Metribuzin					
Mevinphos					
Mexacarbate					
MGK 264					
Mirex					
Molinate					
Monocrotophos					
Naled					
Napropamide					
1,4-Naphthoquinone					
1-Naphthylamine					
2-Naphthylamine					
1,5-Naphthalenediamine					
Nicotine					
5-Nitroacenaphthene					
2-Nitroaniline					
3-Nitroaniline.					
4-Nitroaniline.					
5-Nitro-o-anisidine					
4-Nitrobiphenyl					
Nitrofen					
5-Nitro-o-toluidine					

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Analyte	Range for Q (%)	Limit for s (%)	Range for $\bar{X}$ (%)	Range for P1, P2 (%)	Limit for RPD (%)
Nitroquinoline-1-oxide					
N-Nitrosodi-n-butylamine					
N-Nitrosodiethylamine					
N-Nitrosodimethylamine					
N-Nitrosodiphenylamine					
N-Nitrosomethylethylamine					
N-Nitrosomethylphenylamine					
N-Nitrosomorpholine					
N-Nitrosopiperidine					
N-Nitrosopyrrolidine					
trans-Nonachlor					
Norflurazon					
2,2',3,3',4,5',6,6'-Octachlorobiphenyl					
Octamethyl pyrophosphoramidate					
4,4'-Oxydianiline					
Parathion					
PCB-1016					
PCB-1221					
PCB-1232					
PCB-1242					
PCB-1248					
PCB-1254					
PCB-1260	19-130	77	19-130	D-164	128
PCB-1268					
Pebulate					
Pentachlorobenzene					
Pentachloronitrobenzene					
2,2',3,4',6-Pentachlorobiphenyl					
Pentachloroethane					
Pentamethylbenzene					
Perylene					
Phenacetin					
cis-Permethrin					
trans-Permethrin					
Phenobarbital					
Phenothiazene					
1,4-Phenylenediamine					
1-Phenylnaphthalene					
2-Phenylnaphthalene					
Phorate					
Phosalone					
Phosmet					
Phosphamidon					
Phthalic anhydride					
alpha-Picoline (2-Methylpyridine)					

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Analyte	Range for Q (%)	Limit for s (%)	Range for $\bar{X}$ (%)	Range for P1, P2 (%)	Limit for RPD (%)
Piperonyl sulfoxide					
Prometon					
Prometryn					
Pronamide					
Propachlor					
Propazine					
Propylthiouracil					
Pyridine					
Resorcinol (1,3-Benzenediol)					
Safrole					
Simazine					
Simetryn					
Squalene					
Stirofos					
Strychnine					
Styrene 9					
Sulfallate					
Tebuthiuron					
Terbacil..					
Terbufos					
Terbutryn					
alpha-Terpineol					
1,2,4,5-Tetrachlorobenzene					
2,2',4,4'-Tetrachlorobiphenyl					
2,3,7,8-Tetrachlorodibenzo-p-dioxin					
2,3,4,6-Tetrachlorophenol					
Tetrachlorvinphos					
Tetraethyl dithiopyrophosphate					
Tetraethyl pyrophosphate					
Thianaphthene (2,3-Benzothiophene)					
Thioacetamide					
Thionazin					
Thiophenol (Benzenethiol)					
Thioxanthone					
Toluene-1,3-diisocyanate					
Toluene-2,4-diisocyanate					
o-Toluidine					
Toxaphene 3,5					
Triadimefon					
1,2,3-Trichlorobenzene					
2,4,5-Trichlorobiphenyl					
2,3,6-Trichlorophenol					
2,4,5-Trichlorophenol					
Tricyclazole					
Trifluralin					

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Analyte	Range for Q (%)	Limit for s (%)	Range for $\bar{X}$ (%)	Range for P1, P2 (%)	Limit for RPD (%)
1,2,3-Trimethoxybenzene					
2,4,5-Trimethylaniline					
Trimethyl phosphate					
Triphenylene					
Tripropyleneglycolmethyl ether					
1,3,5-Trinitrobenzene					
Tris(2,3-dibromopropyl) phosphate					
Tri-p-tolyl phosphate					
O,O,O-Triethyl phosphorothioate.					
Trithiane					
Vernolate					

Many of the analytes in this table do not have QC acceptance criteria. If calibration is to be verified and other QC tests are to be performed for these analytes, acceptance criteria must be developed and applied. EPA has provided guidance for development of QC acceptance criteria (see 40 CFR 136.6(b)(2)(i) and *Protocol for EPA Approval of New Methods for Organic and Inorganic Analytes in Wastewater and Drinking Water* (EPA-821-B-98-003) March 1999). Alternatively, analytes that do not have acceptance criteria may be based on laboratory control charts, or 60 to 140% may be used.

* At a minimum, these compounds must be spiked into the MS/MSD analyses when direction cannot be obtained from the data user.

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Attachment VIII – Method 625.1 Suggested Internal Standards and Surrogates

Compound	Range for Surrogate Recovery	
	Calibration Verification	Recovery from Samples
Base/Neutral Fraction		
Acenaphthalene-d ₈	66-152	33-168
Acenaphthene-d ₁₀	71-141	30-180
Aniline-d ₅		
Anthracene-d ₁₀	58-171	53-142
Benzo(a)anthracene-d ₁₂	28-357	22-329
Benzo(a)pyrene-d ₁₂	32-194	32-194
4-Chloroaniline-d ₄	1-145	1-145
bis(2-Chloroethyl)ether-d ₈	52-194	25-222
Chrysene-d ₁₂	23-290	23-290
Decafluorobiphenyl		
4,4'-Dibromobiphenyl		
4,4'-Dibromooctafluorobiphenyl		
1,4-Dichlorobenzene-d ₄	65-153	11-245
2,2'-Difluorobiphenyl		
Dimethyl phthalate-d ₆	47-211	1-500
Fluoranthene-d ₁₀	61-164	38-172
4-Fluoroaniline		
1-Fluoronaphthalene		
2-Fluoronaphthalene		
2-Methylnaphthalene-d ₁₀	50-150	50-150
Naphthalene-d ₈	71-141	22-192
Nitrobenzene-d ₅	46-219	15-314
2,3,4,5,6-Pentafluorobiphenyl		
Perylene-d ₁₂		
Phenanthrene-d ₁₀	67-149	34-168
Pyrene-d ₁₀	48-210	28-196
Pyridine-d ₅		
Acid Fraction		
2-Chlorophenol-d ₄	55-180	33-180
2,4-Dichlorophenol-d ₃	64-157	34-182
4,6-Dinitro-2-methylphenol-d ₂	56-177	22-307
2-Fluorophenol		
4-Methylphenol-d ₈	25-111	25-111
2-Nitrophenol-d ₄	61-163	37-163
4-Nitrophenol-d ₄	35-287	6-500
Pentafluorophenol		
2-Perfluoromethylphenol		
Phenol-d ₅	48-208	8-424

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Many of the surrogates in this table do not have QC acceptance criteria. If calibration is to be verified and other QC tests are to be performed for these surrogates, acceptance criteria must be developed and applied. EPA has provided guidance for development of QC acceptance criteria (see 40 CFR 136.6(b)(2)(i) and *Protocol for EPA Approval of New Methods for Organic and Inorganic Analytes in Wastewater and Drinking Water* (EPA-821-B-98-003) March 1999). Alternatively, surrogates that do not have acceptance criteria may be based on laboratory control charts, or 60 to 140% may be used.



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Attachment IX – DoD Requirements

1.0 Equipment/Instrument Maintenance

Instrument maintenance must be performed routinely to optimize instrument performance and improve chromatography. Commonly performed maintenance includes baking columns, changing injection port liners, changing pump oil, etc. A new calibration curve must be analyzed following any major maintenance performed on the analytical system.

2.0 Computer Hardware and Software

Software name and version: HP Chemstation G1701CA Version C.00.00 or equivalent

3.0 Troubleshooting

Table 1. GCMS Troubleshooting Guide		
Problem	Cause	Treatment
Peaks broaden and tail	Poor column installation causing dead volume in the injector	Reinstall column in injector. Check seal at ferrule. Check insertion depth. Ensure a good column cut.
	Solvent flashing in hot injector	Reduce injection speed on hot injectors and if possible reduce injector temperature
	Injector not being purged properly after splitless injection	For splitless injection, the vent flow should be 70 ml/min, and the injector should be switched to the split mode 0.5_1.5 min after injection.
Tailing sample peaks for active components	Active sites in the injector insert or liner	Change or clean the injector insert
	Active sites or degraded phase in column	Remove the front 15 cm of the column and reinstall. If retention times are changing or cutting the column does not help, replace the column.
	Injector not hot enough for higher boiling compounds	Increase the injector temperature and lower the injection speed. Check that the graphite ferrule is free of cracks and the septum support is tight.
Low response and tailing of high boiling point compounds	Injector is not hot enough to vaporize high boilers	Increase injector temperature
	Interface/ion source not getting to adequate temperature	Change the manifold heater
Leading sample peaks	Column overload due to excess amount of component injected	Dilute the sample or do split injection
	Degradation of stationary phase	Change the column
	Carrier gas velocity too low	Increase carrier gas flow rate
Poor chromatographic resolution	Column temperature or program not optimized	Modify method by changing temperature ramp segment slopes

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Table 1. GCMS Troubleshooting Guide

Problem	Cause	Treatment
	Carrier gas flow rate not optimized	Decrease carrier gas linear velocity
	Stationary phase has degraded	Replace the column
Peak splitting, especially low boilers	Sample is flashing in the injector simulating two injections	Lower injector temperature
Retention times shift in chromatogram	Unstable carrier gas flow controller/regulator	Check pneumatics for leaks. Replace flow controller/ regulator if necessary.
	Column contamination or degradation	Condition or replace column
	Leaks at septum or column to injector connection	Replace septum regularly and check that the septum nut and the capillary column nut are tight
Cannot reach operating vacuum	Analyzer contaminated by diffusion pump oil	Shut down and clean mass spec
	Major air leak around column fitting into interface	Replace column ferrule and reseal compression fitting
No calibration gas peaks	Cal gas valve not open	Open cal gas valve
	Calibration gas solenoid valve stuck open. All calibration gas evaporated.	Have solenoid replaced. Put fresh PFBTA in the cal gas vial.
Analysis sensitivity has decreased	Background has increased	Check column bleed, septum bleed, pump oil, and ion source contamination
	Detector needs replacement	Replace detector
	Defective syringe	Try a new or proven syringe
	“Blown” septum or other massive leaks at the inlet or with carrier gas flow. Poor peak shapes usually result from bad leaks.	Find and fix leaks and adjust gas flow.
	Purge flow or split ratio too high	Adjust gas flow rates

4.0 Other Requirements

- 4.1 All hardcopy laboratory notebooks must be reviewed by the Supervisor, or their designee, on a monthly basis.
- 4.2 If not self-explanatory (e.g., a typo or transposed number), corrections to technical and quality records shall also include a justification for the change.
- 4.3 A storage blank must be stored with all volatile organic samples, regardless of suspected concentration levels.
- 4.4 A person performing a manual integration must sign and date each manually integrated chromatogram and record the rationale for performing manual integration. Electronic signatures are acceptable.

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- 4.5 The results of calibration and verification of support equipment must be within the specifications required of the application for which this equipment is used or the equipment must be removed from service until repaired. Calibration and verification records, including those of established correction factors, must be maintained. In the absence of method-specific requirements, the minimum requirements are as follows:

Table 2. Support Equipment Checks

Performance Check	Frequency	Acceptance Criteria
Balance calibration check [Using two standard weights that bracket the expected mass]	Daily prior to use	Top-loading balance: $\pm 2\%$ or $\pm 0.02\text{g}$, whichever is greater Analytical balance: $\pm 0.1\%$ or $\pm 0.5\text{mg}$, whichever is greater
Verification of standard mass [Using weights traceable to the International System of Units (SI) through a NMI]	Every 5 years	Certificate of Calibration from ISO/IEC 17025 accredited calibration laboratory
Monitoring of refrigerator/freezer temperatures	Daily (i.e. 7 days per week) [use MIN/MAX thermometers or data loggers equipped with notification of out of control event capabilities if personnel not available to record daily]	Refrigerators: 0°C to 6°C Freezers: $\leq -10^{\circ}\text{C}$
Thermometer verification check [Using a thermometer traceable to the SI through an NMI] [Performed at two temperatures that bracket the target temperature(s). Assume linearity between the two bracketing temperatures.] [If only a single temperature is used, at the temperature of use]	Liquid in glass: Before first use and annually Electronic: Before first use and quarterly	Apply correction factors or replace thermometer
Volumetric labware	Class B: By lot before first use Class A and B: Upon evidence of deterioration	Bias: Mean within $\pm 2\%$ of nominal volume Precision: $\text{RSD} \leq 1\%$ of nominal volume (based on 10 replicate measurements)
Non-volumetric labware [Applicable only when used for measuring initial sample volume and final extract/ digestates volume]	By lot before first use or upon evidence of deterioration	Bias: Mean within $\pm 3\%$ of nominal volume Precision: $\text{RSD} \leq 3\%$ of nominal volume (based on 10 replicate measurements)
Mechanical volumetric pipette	Quarterly	Bias: Mean within $\pm 2\%$ of nominal volume Precision: $\text{RSD} \leq 1\%$ of nominal volume (based on minimum of 3 replicate measurements) [Note: for variable volume pipettes, the nominal volume is the volume of use]

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Table 2. Support Equipment Checks

Performance Check	Frequency	Acceptance Criteria
Glass microliter syringe	Upon receipt and upon evidence of deterioration	General Certificate of Bias & Precision upon receipt Replace if deterioration is evident
Drying oven temperature check	Daily prior to and after use	Within $\pm 5\%$ of set temperature
Water purification system	Daily prior to use	See method blank criteria given in Section 4.20 of this addendum

- 4.6 The expiration date of the prepared standard shall not exceed the expiration date of the primary standard. All containers must bear a preparation date.
- 4.7 To avoid preparing non-representative samples, the laboratory shall not “target” within a relatively small mass range (e.g., $1.00 \pm 0.01\text{g}$) because such targeting will produce non-representative subsamples if the sample has high heterogeneity. The laboratory shall not manipulate the sample material so the sample aliquot weighs exactly $1.00\text{g} \pm 0.01\text{g}$, as an example.
- 4.8 In the absence of project-specific requirements, the minimum standard data qualifiers to be used are:
- U Analyte was not detected and is reported as less than the LOD or as defined by the customer. The LOD has been adjusted for any dilution or concentration of the sample.
 - J The reported result is an estimated value (e.g., matrix interference was observed or the analyte was detected at a concentration outside the quantitation range).
 - B Blank contamination. The recorded result is associated with a contaminated blank.
 - N Non-target analyte. The analyte is a tentatively identified compound using mass spectrometry or any non-customer requested compounds that are tentatively identified.
 - Q One or more quality control criteria failed (e.g., LCS recovery, surrogate spike recovery, or CCV recovery).
- Additional data qualifiers may be used, or different letters or symbols to denote the qualifiers listed above, as long as they are appropriately defined and their use is consistent with project-specific requirements (e.g., QSM 5.0, the contract, and project-planning documents).
- 4.9 If the time of the sample collection is not provided, assume the most conservative time of day. For the purpose of batch processing, the start and stop dates and times of the batch preparation shall be recorded.
- 4.10 Each preparation method listed on the scope of accreditation must have quarterly LOD/LOQ verifications. However, not all possible combinations of preparation and cleanup techniques are required to have LOD/LOQ verifications. If LOD/LOQ verifications are not performed on all combinations, the laboratory must base the LOD/LOQ verifications on the worst case basis (preparation method with all applicable cleanup steps).



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- 4.11 After each MDL determination, the laboratory must establish the LOD by spiking a quality system matrix at a concentration of at least 2 times but no greater than four times the MDL. This spike concentration establishes the LOD and the concentration at which the LOD shall be verified. It is specific to each suite of analyte, matrix, and method (including sample preparation). The following requirements apply to the initial LOD establishment and to the LOD verifications:
- The apparent signal to noise (S/N) ratio at the LOD must be at least three and the results must meet all method requirements for analyte identification (e.g., ion abundance, second column confirmation, or pattern recognition). For data systems that do not provide a measure of noise, the signal produced by the verification sample must produce a result that is at least three standard deviations greater than the mean method blank concentration. This is initially estimated based on a minimum of four method blank analyses and later established with a minimum of 20 method blank results.
 - If the LOD verification fails, then the laboratory must repeat the MDL determination and LOD verification or perform and pass two consecutive LOD verifications at a higher spike concentration and set the LOD at the higher concentration.
 - The laboratory shall maintain documentation for all MDL determinations and LOD verifications.
 - The DL and LOD must be reported for all analyte-matrix-methods suites, unless it is not applicable to the test or specifically excluded by project requirements.
- 4.12 The LOD shall be verified quarterly. In situations where methods are setup and used on an infrequent basis, the laboratory may choose to perform LOD verifications on a one per batch basis. All verification data will be in compliance, reported, and available for review.
- 4.13 For DoD, at a minimum, the LOQ shall be verified quarterly. In situations where methods are setup and used on an infrequent basis, the laboratory may choose to perform LOQ verifications on a one per batch basis.
- 4.14 All initial instrument calibrations must be verified with a standard obtained from a second manufacturer prior to analyzing any samples. The use of a standard from a second lot obtained from the same manufacturer (independently prepared from different source materials) is acceptable for use as a second source standard. The concentration of the second source standard shall be at or near the midpoint of the calibration range. The acceptance criteria for the initial calibration verification must be at least as stringent as those for the continuing calibration verification.
- 4.15 Exclusion of calibration points without documented scientifically valid technical justification is not permitted.
- 4.16 The concentration of the CCV standard shall be greater than the low calibration standard and less than or equal to the midpoint of the calibration range.
- 4.17 All CCVs analyzed must be evaluated and reported. If a CCV fails, reanalysis or corrective actions must be taken.
- If a CCV fails, the laboratory can immediately analyze two additional consecutive CCVs (immediately is defined as starting a consecutive pair within one hour; no samples can be run between the failed CCV and the two additional CCVs). This approach allows for spurious failures of analytes to be reported without reanalysis of samples. Any corrective actions that change the dynamics of the system (e.g., clip



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- column, clean injection port, run blanks) requires that all samples since the last acceptable CCV be reanalyzed.
- Both of these CCVs must meet acceptance criteria in order for the samples to be reported without reanalysis.
 - If either of these two CCVs fail or if the laboratory cannot immediately analyze two CCVs, the associated samples cannot be reported and must be reanalyzed.
 - Corrective action(s) and recalibration must occur if the above scenario fails. All affected samples since the last acceptable CCV must be reanalyzed.
 - Flagging of data for a failed CCV is only appropriate when the affected samples cannot be reanalyzed. The laboratory must notify the client prior to reporting data associated with a failed CCV.
- 4.18 The results of all MS/MSDs must be evaluated using the same acceptance criteria used for the DoD LCS limits (see Addendum Tables 3 through 6) or project limits, if specified. If the specific analyte(s) are not available in the Addendum Tables 3 through 6, the laboratory shall use their LCS in-house limits (see the LIMS) as a means of evaluating MS/MSDs. The MS and MSD must be spiked with all reported analytes.
- 4.19 Surrogate spike results shall be compared with DoD LCS limits (see Addendum Tables 3 through 6) or acceptance criteria specified by the client. If these criteria are not available, the laboratory shall compare the results with its in-house statistically established LCS criteria (see the LIMS).
- 4.20 The method blank shall be considered to be contaminated if:
- The concentration of any target analyte (chemical of concern) in the blank exceeds 1/2 the LOQ and is greater than 1/10th the amount measured in any associated sample, or 1/10th the regulatory limit, whichever is greater;
 - The concentration of any common laboratory contaminant in the blank exceeds the LOQ;
 - If a method blank is contaminated as described above, then the laboratory shall reprocess affected samples in a subsequent preparation batch, except when sample results are below the LOD. If insufficient sample volume remains for reprocessing, the results shall be reported with appropriate data qualifiers.
- 4.21 Sporadic Marginal Exceedances are not allowed for target analytes (chemicals of concern as identified by a project) without project-specific approval. Target analytes are considered those few analytes that are critical for the success of a project (such as risk drivers) where sporadic marginal exceedances cannot be allowed. Laboratories should consult with clients whenever long lists of analytes are requested for analysis to determine if marginal exceedances will not be allowed.
- 4.22 DoD considers the same analyte exceeding the LCS control limit two (2) out of three (3) consecutive LCS to be indicative of non-random behavior, which requires corrective action and reanalysis of the LCS.

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Table 3. LCS Control Limits – Method 8270 Solid Matrix

CAS ID	Analyte	N Records	Mean	Standard Deviation	Lower Control Limit	Upper Control Limit
92-52-4	1,1-Biphenyl	1645	78.5	13	40	117
95-94-3	1,2,4,5-Tetrachlorobenzene	1810	77.8	13.7	37	119
120-82-1	1,2,4-Trichlorobenzene	3577	75.7	13.9	34	118
95-50-1	1,2-Dichlorobenzene	3352	74.6	14	33	117
528-29-0	1,2-Dinitrobenzene [1,2-DNB]	203	79.4	11.9	44	115
122-66-7	1,2-Diphenylhydrazine [Azobenzene]	2039	83	13.9	41	125
99-35-4	1,3,5-Trinitrobenzene [1,3,5-TNB]	154	89.2	10.7	57	121
541-73-1	1,3-Dichlorobenzene	3288	72.6	14.1	30	115
99-65-0	1,3-Dinitrobenzene [1,3-DNB]	598	84.6	14	43	127
106-46-7	1,4-Dichlorobenzene	3793	73.1	13.9	31	115
100-25-4	1,4-Dinitrobenzene	248	84.4	15.7	37	132
130-15-4	1,4-Naphthoquinone	150	81.2	8.8	55	108
90-13-1	1-Chloronaphthalene	119	81.1	11.1	48	115
90-12-0	1-Methylnaphthalene	3004	79.2	13.2	40	119
58-90-2	2,3,4,6-Tetrachlorophenol	1724	84.7	13.6	44	125
935-95-5	2,3,5,6-Tetrachlorophenol	227	75.9	11.9	40	112
608-27-5	2,3-Dichloroaniline	108	82.4	13	44	121
95-95-4	2,4,5-Trichlorophenol	4014	82.6	13.7	41	124
118-79-6	2,4,6-Tribromophenol	2930	85.7	15.4	39	132
88-06-2	2,4,6-Trichlorophenol	4183	82.1	14.5	39	126
120-83-2	2,4-Dichlorophenol	3794	80.9	13.7	40	122
105-67-9	2,4-Dimethylphenol	3886	78.4	16.2	30	127
121-14-2	2,4-Dinitrotoluene	4075	86.8	12.9	48	126
87-65-0	2,6-Dichlorophenol	1364	79.2	12.6	41	117
606-20-2	2,6-Dinitrotoluene	3706	85	13	46	124
53-96-3	2-Acetylaminofluorene	175	94	13.3	54	134
91-58-7	2-Chloronaphthalene	3569	77.5	12.1	41	114
95-57-8	2-Chlorophenol	3977	77.3	14.5	34	121
321-60-8	2-Fluorobiphenyl	3191	79.5	11.8	44	115
367-12-4	2-Fluorophenol	3008	75.2	13.3	35	115
91-57-6	2-Methylnaphthalene	5059	80.1	14	38	122
95-48-7	2-Methylphenol (o-Cresol)	4016	77	14.9	32	122
88-74-4	2-Nitroaniline	3639	85.4	13.8	44	127
119-75-5	2-Nitrodiphenylamine	279	88.1	11.6	53	123
88-75-5	2-Nitrophenol	3804	79.6	14.5	36	123
109-06-8	2-Picoline [2-Methylpyridine]	181	64.5	12.7	27	103
91-94-1	3,3'-Dichlorobenzidine	3521	71.3	16.5	22	121
56-49-5	3-Methylcholanthrene	188	95.1	13	56	134
99-09-2	3-Nitroaniline	3454	75.9	14.3	33	119
65794-96-9	3/4-Methylphenol [m/p-Cresol]	2900	76.5	14.1	34	119
534-52-1	4,6-Dinitro-2-methylphenol	3739	80.7	17.2	29	132
101-55-3	4-Bromophenyl phenyl ether	3708	85.1	13	46	124

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Table 3. LCS Control Limits – Method 8270 Solid Matrix

CAS ID	Analyte	N Records	Mean	Standard Deviation	Lower Control Limit	Upper Control Limit
59-50-7	4-Chloro-3-methylphenol	3880	83.3	12.9	45	122
106-47-8	4-Chloroaniline [p-Chloroaniline]	3435	61.3	14.9	17	106
7005-72-3	4-Chlorophenyl phenyl ether	3673	83	12.7	45	121
106-44-5	4-Methylphenol [p-Cresol]	1555	84.1	14.1	42	126
100-02-7	4-Nitrophenol	3976	80.6	17	30	132
99-55-8	5-Nitro-o-toluidine [2-Amino-4-nitrotoluene]	187	69.8	15.8	23	117
57-97-6	7,12-Dimethylbenz(a)-anthracene	338	96.2	15.3	50	142
83-32-9	Acenaphthene	5300	81.3	13.7	40	123
208-96-8	Acenaphthylene	5194	81.8	16.8	32	132
98-86-2	Acetophenone	2101	73.9	13.6	33	115
120-12-7	Anthracene	5250	85.2	12.7	47	123
1912-24-9	Atrazine	1428	87.1	13.4	47	127
103-33-3	Azobenzene	378	82.1	14.2	39	125
56-55-3	Benz(a)anthracene	5385	87.4	12.9	49	126
50-32-8	Benzo(a)pyrene	5500	86.9	13.9	45	129
205-99-2	Benzo(b)fluoranthene	5323	88.3	14.5	45	132
191-24-2	Benzo(g,h,i)perylene	5263	88.5	15.1	43	134
207-08-9	Benzo(k)fluoranthene	5386	89.6	14.2	47	132
100-51-6	Benzyl alcohol	2895	75.7	15.6	29	122
111-91-1	bis(2-Chloroethoxy)methane	3705	78.4	14.2	36	121
111-44-4	Bis(2-chloroethyl) ether	3711	75.4	14.9	31	120
39638-32-9	bis(2-Chloroisopropyl) ether	769	82	16.3	33	131
117-81-7	Bis(2-ethylhexyl) phthalate	4018	91.9	13.7	51	133
103-23-1	bis(2-Ethylhexyl)adipate	156	90.8	10.1	61	121
85-68-7	Butyl benzyl phthalate	3956	90.3	14	48	132
105-60-2	Caprolactam	1203	81.3	11.9	46	117
86-74-8	Carbazole	3095	86.3	12	50	123
510-15-6	Chlorobenzilate	172	99.7	16.9	49	150
218-01-9	Chrysene	5395	87.1	12.2	50	124
84-74-2	Di-n-butyl phthalate	4041	89.4	12.8	51	128
117-84-0	Di-n-octyl phthalate	3985	92.4	16	45	140
2303-16-4	Diallate [cis or trans]	173	93.7	12.7	56	132
53-70-3	Dibenzo(a,h)anthracene	5393	89.5	14.7	45	134
132-64-9	Dibenzofuran	3749	81.5	12.7	44	120
84-66-2	Diethyl phthalate	4012	87.2	12.3	50	124
60-51-5	Dimethoate	137	68	13.3	28	108
131-11-3	Dimethyl phthalate	4023	85.9	12.6	48	124
60-11-7	Dimethylaminoazobenzene	177	98.7	11.6	64	134
88-85-7	Dinoseb	123	67.3	17.1	16	119
101-84-8	Diphenyl ether	114	95.6	6	78	114
122-39-4	Diphenylamine	854	79.5	10.6	48	111
62-50-0	Ethyl methanesulfonate	174	85.1	16.9	34	136

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Table 3. LCS Control Limits – Method 8270 Solid Matrix

CAS ID	Analyte	N Records	Mean	Standard Deviation	Lower Control Limit	Upper Control Limit
206-44-0	Fluoranthene	5340	88.3	12.9	50	127
86-73-7	Fluorene	5150	84.2	13.8	43	125
118-74-1	Hexachlorobenzene	4138	83.5	13	45	122
87-68-3	Hexachlorobutadiene	4003	77.3	15.3	32	123
67-72-1	Hexachloroethane	4049	72.2	14.9	28	117
1888-71-7	Hexachloropropene	259	81.9	16.7	32	132
95-13-6	Indene	188	85.3	8.9	59	112
193-39-5	Indeno(1,2,3-cd)pyrene	5367	89.3	14.7	45	133
465-73-6	isodrin	167	93.8	12.8	56	132
78-59-1	Isophorone	3787	75.9	15.2	30	122
120-58-1	Isosafrole	174	89.5	15.4	43	136
66-27-3	Methyl methanesulfonate	150	77.9	13.1	38	117
100-75-4	N-Nitrosopiperidine	232	89.4	9.8	60	119
924-16-3	N-Nitrosodi-n-butylamine	236	91.7	10.8	59	124
621-64-7	N-Nitrosodi-n-propylamine	3857	78.2	13.9	36	120
55-18-5	N-nitrosodiethylamine	421	82.1	13.8	41	124
62-75-9	N-Nitrosodimethylamine	3170	71.6	16.2	23	120
86-30-6	N-Nitrosodiphenylamine	2968	82.7	14.8	38	127
10595-95-6	n-Nitrosomethylethylamine	265	78.7	14.9	34	123
59-89-2	n-Nitrosomorpholine	172	91.3	13.8	50	133
930-55-2	n-Nitrosopyrrolidine	326	85.5	13.6	45	126
91-20-3	Naphthalene	5342	78.8	14.7	35	123
98-95-3	Nitrobenzene	4103	77.8	14.7	34	122
4165-60-0	Nitrobenzene-d5	3226	79.3	14.2	37	122
56-57-5	Nitroquinoline-1-oxide	177	91.3	24.5	18	165
126-68-1	O,O,O-Triethyl phosphorothioate	138	91.6	10.8	59	124
593-45-3	Octadecane	113	87.4	14.5	44	131
608-93-5	Pentachlorobenzene	346	89.7	11.8	54	125
76-01-7	Pentachloroethane	131	70.4	10.6	39	102
87-86-5	Pentachlorophenol	4161	78.7	18	25	133
82-68-8	Pentachloronitrobenzene	579	86.1	16	38	134
62-44-2	Phenacetin	185	95	12.5	57	133
85-01-8	Phenanthrene	5259	85.4	12	50	121
108-95-2	Phenol	4029	77.3	14.4	34	121
4165-62-2	Phenol-d5	1016	77.4	14.9	33	122
23950-58-5	Pronamide	179	93	12.4	56	130
129-00-0	Pyrene	5518	87.2	13.3	47	127
91-22-5	Quinoline	219	90	11.9	54	126
94-59-7	Safrole	176	87.8	13.6	47	129
1718-51-0	Terphenyl-d14	3111	90.5	12.3	54	127
3689-24-5	Tetraethyl dithiopyrophosphate [Sulfotep]	136	94.4	14	52	137
297-97-2	Thionazine	139	94.6	10.7	62	127

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Table 4. LCS Control Limits – Method 8270 Water Matrix

CAS ID	Analyte	N Records	Mean	Standard Deviation	Lower Control Limit	Upper Control Limit
92-52-4	1,1-Biphenyl	2247	82.1	11.1	49	115
95-94-3	1,2,4,5-Tetrachlorobenzene	2326	77.9	14.5	35	121
120-82-1	1,2,4-Trichlorobenzene	4716	72.6	14.5	29	116
95-50-1	1,2-Dichlorobenzene	4442	71.4	13.3	32	111
528-29-0	1,2-Dinitrobenzene [1,2-DNB]	112	83.9	8.3	59	109
122-66-7	1,2-Diphenylhydrazine [Azobenzene]	2244	85.4	12.2	49	122
99-35-4	1,3,5-Trinitrobenzene [1,3,5-TNB]	241	89.1	16	41	137
541-73-1	1,3-Dichlorobenzene	4375	68.6	13.6	28	110
99-65-0	1,3-Dinitrobenzene [1,3-DNB]	601	88.2	13.1	49	128
106-46-7	1,4-Dichlorobenzene	5433	70.4	13.9	29	112
90-13-1	1-Chloronaphthalene	211	84.5	8.8	58	111
90-12-0	1-Methylnaphthalene	3742	80	13.1	41	119
134-32-7	1-Naphthylamine	258	73.7	16.6	24	124
58-90-2	2,3,4,6-Tetrachlorophenol	2293	89	13	50	128
935-95-5	2,3,5,6-Tetrachlorophenol	266	85.6	11.7	50	121
608-27-5	2,3-Dichloroaniline	150	99.2	9.8	70	129
95-95-4	2,4,5-Trichlorophenol	5707	88.1	11.8	53	123
118-79-6	2,4,6-Tribromophenol	2059	91.5	16	43	140
88-06-2	2,4,6-Trichlorophenol	6136	87.2	12.4	50	125
120-83-2	2,4-Dichlorophenol	5330	84	12.2	47	121
105-67-9	2,4-Dimethylphenol	5298	77.5	15.6	31	124
51-28-5	2,4-Dinitrophenol	5127	82.9	20	23	143
121-14-2	2,4-Dinitrotoluene	6032	92.3	11.8	57	128
87-65-0	2,6-Dichlorophenol	1583	84	11.4	50	118
606-20-2	2,6-Dinitrotoluene	5107	90.7	11.2	57	124
53-96-3	2-Acetylaminofluorene	228	98.9	12.9	60	138
91-58-7	2-Chloronaphthalene	5084	78	12.8	40	116
95-57-8	2-Chlorophenol	5571	77.5	13.2	38	117
93951-73-6	2-Chlorophenol-d4	119	79.9	8.7	54	106
321-60-8	2-Fluorobiphenyl	2263	81.2	12.4	44	119
367-12-4	2-Fluorophenol	2022	68.8	16.6	19	119
91-57-6	2-Methylnaphthalene	6330	80.7	13.6	40	121
95-48-7	2-Methylphenol (o-Cresol)	5800	73	14.5	30	117
88-74-4	2-Nitroaniline	4855	90.8	12.1	55	127
119-75-5	2-Nitrodiphenylamine	272	97.3	11.3	64	131
88-75-5	2-Nitrophenol	5097	84.6	12.7	47	123
109-06-8	2-Picoline [2-Methylpyridine]	195	71.6	12.6	34	109
91-94-1	3,3'-Dichlorobenzidine	4815	77.9	16.9	27	129
56-49-5	3-Methylcholanthrene	237	94	12.8	56	133
99-09-2	3-Nitroaniline	4808	84.4	14.5	41	128

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**STANDARD OPERATING PROCEDURE**

TITLE: ENV-SOP-MTJL-0081 Semivolatile Organics by GCMS using Capillary Column (EPA Methods 8270C, 8270D, 625, 625.1 and SM6410 B) Including Provisions for Analysis in SIM Mode

ISSUER: Pace National – Mt. Juliet, Tennessee

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Table 4. LCS Control Limits – Method 8270 Water Matrix

CAS ID	Analyte	N Records	Mean	Standard Deviation	Lower Control Limit	Upper Control Limit
65794-96-9	3/4-Methylphenol [m/p-Cresol]	3472	69.7	13.6	29	110
534-52-1	4,6-Dinitro-2-methylphenol	5097	90.1	15.5	44	137
101-55-3	4-Bromophenyl phenyl ether	5074	89.1	11.5	55	124
59-50-7	4-Chloro-3-methylphenol	5338	85.5	11.3	52	119
106-47-8	4-Chloroaniline [p-Chloroaniline]	4687	75.3	14	33	117
7005-72-3	4-Chlorophenyl phenyl ether	5071	86.7	11.3	53	121
106-44-5	4-Methylphenol [p-Cresol]	2798	72.5	15.8	25	120
99-55-8	5-Nitro-o-toluidine [2-amino-4-nitrotoluene]	260	82.1	14.6	38	126
57-97-6	7,12-Dimethylbenz(a)-anthracene	373	97.1	11.9	61	133
83-32-9	Acenaphthene	6952	84.5	12.3	47	122
208-96-8	Acenaphthylene	6662	85.3	14.7	41	130
98-86-2	Acetophenone	2877	82.1	12	46	118
120-12-7	Anthracene	6792	89.6	11	57	123
140-57-8	Aramite	100	82.8	16.3	34	132
1912-24-9	Atrazine	2328	92.8	16.4	44	142
103-33-3	Azobenzene	578	88.5	9.3	61	116
56-55-3	Benz(a)anthracene	6867	91.6	11.1	58	125
50-32-8	Benzo(a)pyrene	7045	90.8	12.4	54	128
205-99-2	Benzo(b)fluoranthene	6767	92	12.9	53	131
191-24-2	Benzo(g,h,i)perylene	6624	92	13.9	50	134
207-08-9	Benzo(k)fluoranthene	6803	93.2	12.1	57	129
100-51-6	Benzyl alcohol	3349	71.2	13.5	31	112
111-91-1	bis(2-Chloroethoxy)methane	5094	83.9	11.9	48	120
111-44-4	Bis(2-chloroethyl) ether	5139	80.8	12.6	43	118
39638-32-9	bis(2-Chloroisopropyl) ether	1140	83.4	15.4	37	130
117-81-7	Bis(2-ethylhexyl) phthalate	5288	95.2	13.3	55	135
85-68-7	Butyl benzyl phthalate	5173	93.3	13.5	53	134
86-74-8	Carbazole	4187	91.1	10.4	60	122
510-15-6	Chlorobenzilate	226	104.3	15.4	58	150
218-01-9	Chrysene	6779	91.3	10.7	59	123
124-18-5	Decane	126	66.9	12.8	29	105
84-74-2	Di-n-butyl phthalate	5329	93	11.4	59	127
117-84-0	Di-n-octyl phthalate	5222	95.5	15	51	140
2303-16-4	Diallate [cis or trans]	249	95.3	9.6	67	124
226-36-8	Dibenz(a,h)acridine	136	104.4	9.7	75	134
53-70-3	Dibenzo(a,h)anthracene	6840	92.7	13.8	51	134
132-64-9	Dibenzofuran	4963	85.3	10.8	53	118
84-66-2	Diethyl phthalate	5207	90.1	11.5	56	125
131-11-3	Dimethyl phthalate	4977	86	13.7	45	127
60-11-7	Dimethylaminoazobenzene	238	97.1	11.6	62	132
88-85-7	Dinoseb	144	93.4	10.8	61	126

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ISSUER: Pace National – Mt. Juliet, Tennessee

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Table 4. LCS Control Limits – Method 8270 Water Matrix

CAS ID	Analyte	N Records	Mean	Standard Deviation	Lower Control Limit	Upper Control Limit
101-84-8	Diphenyl ether	142	91.7	7.8	68	115
122-39-4	Diphenylamine	754	83	9.2	55	111
298-04-4	Disulfoton	122	92.5	12.5	55	130
62-50-0	Ethyl methanesulfonate	215	90.1	9.4	62	118
206-44-0	Fluoranthene	6826	92.6	11.9	57	128
86-73-7	Fluorene	6786	88.1	12	52	124
118-74-1	Hexachlorobenzene	6263	88.7	12.1	53	125
87-68-3	Hexachlorobutadiene	5878	73.1	16.9	22	124
39638-32-9	bis(2-Chloroisopropyl) ether	1140	83.4	15.4	37	130
117-81-7	Bis(2-ethylhexyl) phthalate	5288	95.2	13.3	55	135
85-68-7	Butyl benzyl phthalate	5173	93.3	13.5	53	134
86-74-8	Carbazole	4187	91.1	10.4	60	122
510-15-6	Chlorobenzilate	226	104.3	15.4	58	150
218-01-9	Chrysene	6779	91.3	10.7	59	123
124-18-5	Decane	126	66.9	12.8	29	105
84-74-2	Di-n-butyl phthalate	5329	93	11.4	59	127
117-84-0	Di-n-octyl phthalate	5222	95.5	15	51	140
2303-16-4	Diallate [cis or trans]	249	95.3	9.6	67	124
226-36-8	Dibenz(a,h)acridine	136	104.4	9.7	75	134
53-70-3	Dibenzo(a,h)anthracene	6840	92.7	13.8	51	134
132-64-9	Dibenzofuran	4963	85.3	10.8	53	118
84-66-2	Diethyl phthalate	5207	90.1	11.5	56	125
131-11-3	Dimethyl phthalate	4977	86	13.7	45	127
60-11-7	Dimethylaminoazobenzene	238	97.1	11.6	62	132
88-85-7	Dinoseb	144	93.4	10.8	61	126
101-84-8	Diphenyl ether	142	91.7	7.8	68	115
122-39-4	Diphenylamine	754	83	9.2	55	111
298-04-4	Disulfoton	122	92.5	12.5	55	130
62-50-0	Ethyl methanesulfonate	215	90.1	9.4	62	118
206-44-0	Fluoranthene	6826	92.6	11.9	57	128
86-73-7	Fluorene	6786	88.1	12	52	124
118-74-1	Hexachlorobenzene	6263	88.7	12.1	53	125
87-68-3	Hexachlorobutadiene	5878	73.1	16.9	22	124
67-72-1	Hexachloroethane	5904	68	15.7	21	115
95-13-6	Indene	253	93.8	13.7	53	135
193-39-5	Indeno(1,2,3-cd)pyrene	6880	92.6	13.6	52	134
465-73-6	isodrin	212	97.6	10	68	128
78-59-1	Isophorone	5190	83.3	13.7	42	124
120-58-1	Isosafrole	230	91.1	11.8	56	126
66-27-3	Methyl methanesulfonate	237	70.1	12.3	33	107
298-00-0	Methyl parathion	121	101.6	19	45	159
100-75-4	N-Nitrosopiperidine	299	88.6	10.8	56	121
924-16-3	N-Nitrosodi-n-butylamine	322	90.4	10.3	60	121
621-64-7	N-Nitrosodi-n-propylamine	5145	84	11.7	49	119
55-18-5	N-nitrosodiethylamine	488	81.8	12.9	43	121

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**STANDARD OPERATING PROCEDURE**

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ISSUER: Pace National – Mt. Juliet, Tennessee

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Table 4. LCS Control Limits – Method 8270 Water Matrix

CAS ID	Analyte	N Records	Mean	Standard Deviation	Lower Control Limit	Upper Control Limit
86-30-6	N-Nitrosodiphenylamine	3743	86.8	11.9	51	123
10595-95-6	n-Nitrosomethylethylamine	311	78.7	12.7	41	117
59-89-2	n-Nitrosomorpholine	214	86.2	10.3	55	117
930-55-2	n-Nitrosopyrrolidine	716	80.8	10.8	48	113
91-20-3	Naphthalene	6953	80	13.5	40	121
98-95-3	Nitrobenzene	5955	83	12.8	45	121
4165-60-0	Nitrobenzene-d5	2223	82.1	12.6	44	120
126-68-1	O,O,O-Triethyl phosphorothioate	212	92.6	8.8	66	119
95-53-4	o-Toluidine	296	69.9	13.2	30	110
593-45-3	Octadecane	151	89	13.1	50	128
56-38-2	Parathion	152	102.6	12.3	66	140
608-93-5	Pentachlorobenzene	401	91.1	10.7	59	123
76-01-7	Pentachloroethane	139	60.9	10.4	30	92
87-86-5	Pentachlorophenol	6083	86.4	17.1	35	138
82-68-8	Pentachloronitrobenzene	618	94.5	13.4	54	135
62-44-2	Phenacetin	241	97.9	8.9	71	124
85-01-8	Phenanthrene	6822	89.6	10.2	59	120
298-02-2	Phorate	126	88.6	16.8	38	139
23950-58-5	Pronamide	249	97	10.5	65	129
129-00-0	Pyrene	7013	91.1	11.5	57	126
91-22-5	Quinoline	249	100.1	10.5	69	132
94-59-7	Safrole	233	90	9.7	61	119
1718-51-0	Terphenyl-d14	1893	91.7	13.9	50	134
3689-24-5	Tetraethyl dithiopyrophosphate [Sulfotep]	200	96.7	11.9	61	133
297-97-2	Thionazine	196	102	10.1	72	132

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Table 5. LCS Control Limits – Method 8270 SIM Solid Matrix

CAS ID	Analyte	N Records	Mean	Standard Deviation	Lower Control Limit	Upper Control Limit
90-12-0	1-Methylnaphthalene	2267	76.6	11.3	43	111
95-95-4	2,4,5-Trichlorophenol	169	79.9	14.9	35	125
91-58-7	2-Chloronaphthalene	615	76.7	10.5	45	108
321-60-8	2-Fluorobiphenyl	1961	80.6	11.6	46	115
91-57-6	2-Methylnaphthalene	2535	76.8	12.5	39	114
83-32-9	Acenaphthene	2813	77.7	11.2	44	111
208-96-8	Acenaphthylene	2761	77.1	12.8	39	116
120-12-7	Anthracene	2812	82.1	10.7	50	114
56-55-3	Benz(a)anthracene	2827	88	11.4	54	122
50-32-8	Benzo(a)pyrene	2789	87.3	12.5	50	125
205-99-2	Benzo(b)fluoranthene	2790	90.3	12.6	53	128
191-24-2	Benzo(g,h,i)perylene	2739	87.8	13	49	127
207-08-9	Benzo(k)fluoranthene	2761	89.3	11.2	56	123
111-44-4	Bis(2-chloroethyl) ether	192	65.4	15.8	18	113
117-81-7	Bis(2-ethylhexyl) phthalate	181	108.9	13.9	67	150
85-68-7	Butyl benzyl phthalate	144	103.5	10.6	72	135
86-74-8	Carbazole	183	79.3	14.6	36	123
218-01-9	Chrysene	2812	87.5	10.2	57	118
84-74-2	Di-n-butyl phthalate	150	106.5	12.9	68	145
117-84-0	Di-n-octyl phthalate	144	105.5	16.8	55	156
53-70-3	Dibenzo(a,h)anthracene	2778	89.2	13.2	50	129
132-64-9	Dibenzofuran	282	71.9	12.2	35	108
84-66-2	Diethyl phthalate	147	99.3	10.9	67	132
131-11-3	Dimethyl phthalate	149	99.3	9.3	71	127
206-44-0	Fluoranthene	2782	87.3	10.7	55	119
86-73-7	Fluorene	2795	80.6	11.2	47	114
118-74-1	Hexachlorobenzene	201	81.9	14.2	39	125
193-39-5	Indeno(1,2,3-cd)pyrene	2812	89.6	13.5	49	130
62-75-9	N-Nitrosodimethylamine	117	90.7	10.9	58	124
91-20-3	Naphthalene	2823	74.7	12.2	38	111
4165-60-0	Nitrobenzene-d5	531	84.7	13.6	44	125
87-86-5	Pentachlorophenol	259	82.4	15.5	36	129
85-01-8	Phenanthrene	2792	80.8	10.6	49	113
129-00-0	Pyrene	2792	85.8	10.2	55	117
1718-51-0	Terphenyl-d14	1864	95.3	12.6	58	133

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Table 6. LCS Control Limits – Method 8270 SIM Water Matrix

CAS ID	Analyte	N Records	Mean	Standard Deviation	Lower Control Limit	Upper Control Limit
92-52-4	1,1-Biphenyl	106	77.3	7.3	56	99
90-12-0	1-Methylnaphthalene	2566	77.9	12.5	41	115
95-95-4	2,4,5-Trichlorophenol	488	84.1	13.4	44	124
118-79-6	2,4,6-Tribromophenol	164	83.7	12.7	46	122
606-20-2	2,6-Dinitrotoluene	118	67.2	15.8	20	115
91-58-7	2-Chloronaphthalene	717	72.4	12.7	34	111
321-60-8	2-Fluorobiphenyl	747	79.2	8.8	53	106
91-57-6	2-Methylnaphthalene	2984	76.5	12.6	39	114
83-32-9	Acenaphthene	3241	80.9	11.1	48	114
208-96-8	Acenaphthylene	3234	77.8	14.4	35	121
120-12-7	Anthracene	3224	85.8	11	53	119
56-55-3	Benz(a)anthracene	3277	89.3	10.1	59	120
50-32-8	Benzo(a)pyrene	3284	86.4	11.2	53	120
205-99-2	Benzo(b)fluoranthene	3248	89.7	12.3	53	126
191-24-2	Benzo(g,h,i)perylene	3178	86	14.1	44	128
207-08-9	Benzo(k)fluoranthene	3167	89.3	11.9	54	125
111-44-4	Bis(2-chloroethyl) ether	775	77.8	12.6	40	116
117-81-7	Bis(2-ethylhexyl) phthalate	275	114.1	19.6	55	173
85-68-7	Butyl benzyl phthalate	159	90.7	17.3	39	143
86-74-8	Carbazole	631	84	13.1	45	123
218-01-9	Chrysene	3215	88.3	10.4	57	120
84-74-2	Di-n-butyl phthalate	153	102.5	14.2	60	145
117-84-0	Di-n-octyl phthalate	157	103.3	19	46	160
53-70-3	Dibenzo(a,h)anthracene	3233	87.2	14.5	44	131
132-64-9	Dibenzofuran	864	77.5	14.1	35	120
84-66-2	Diethyl phthalate	142	94.5	13.5	54	135
206-44-0	Fluoranthene	3242	89.1	10.4	58	120
86-73-7	Fluorene	3232	84.1	11.3	50	118
118-74-1	Hexachlorobenzene	947	84.8	13	46	124
87-68-3	Hexachlorobutadiene	187	84.5	14.7	40	129
193-39-5	Indeno(1,2,3-cd)pyrene	3244	88.7	13.7	48	130
62-75-9	N-Nitrosodimethylamine	162	62.5	10	33	92
91-20-3	Naphthalene	3277	78.8	11.9	43	114
4165-60-0	Nitrobenzene-d5	444	83.1	9.2	55	111
87-86-5	Pentachlorophenol	808	88.4	17.6	36	141
85-01-8	Phenanthrene	3240	83.6	10.3	53	115
129-00-0	Pyrene	3252	87.1	11.3	53	121
1718-51-0	Terphenyl-d14	642	95.1	12.4	58	132

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Table 7. Quality Control Requirements – Organic Analysis by Gas Chromatography/Mass Spectrometry

QC Check	Minimum Frequency	Acceptance Criteria	Corrective Action	Flagging Criteria	Comments
Tune Check	Prior to ICAL and prior to each 12-hour period of sample analysis.	Specific ion abundance criteria of DFTPP from method.	Retune instrument and verify.	Flagging is not appropriate.	No samples shall be analyzed without a valid tune.
Performance Check	At the beginning of each 12-hour period, prior to analysis of samples.	Degradation $\leq 20\%$ for DDT. Benzidine and penta-chlorophenol shall be present at their normal responses, and shall not exceed a tailing factor of 2.	Correct problem, then repeat performance checks.	Flagging is not appropriate.	No samples shall be analyzed until performance check is within criteria. The DDT breakdown and Benzidine/Pentachlorophenol tailing factors are considered overall system checks to evaluate injector port inertness and column performance and are required regardless of the reported analyte list.
Initial calibration (ICAL) for all analytes (including surrogates)	At instrument set-up, prior to sample analysis	Each analyte must meet one of the three options below: Option 1: RSD for each analyte $\leq 15\%$; Option 2: linear least squares regression for each analyte: $r^2 \geq 0.99$; Option 3: non-linear least squares regression (quadratic) for each analyte: $r^2 \geq 0.99$.	Correct problem then repeat ICAL.	Flagging is not appropriate.	Minimum 5 levels for linear and 6 levels for quadratic. No samples shall be analyzed until ICAL has passed. If the specific version of a method requires additional evaluation (e.g., RFs or low calibration standard analysis and recovery criteria) these additional requirements must also be met.

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Table 7. Quality Control Requirements – Organic Analysis by Gas Chromatography/Mass Spectrometry

QC Check	Minimum Frequency	Acceptance Criteria	Corrective Action	Flagging Criteria	Comments
Retention Time window position establishment	Once per ICAL and at the beginning of the analytical sequence.	Position shall be set using the midpoint standard of the ICAL curve when ICAL is performed. On days when ICAL is not performed, the initial CCV is used.	NA.	NA.	Required for each analyte and surrogate.
Evaluation of Relative Retention Times(RRT)	With each sample.	RRT of each reported analyte within ± 0.06 RRT units.	Correct problem, then rerun ICAL.	NA	RRTs may be updated based on the daily CCV. RRTs shall be compared with the most recently updated RRTs.
Initial Calibration Verification (ICV)	Once after each ICAL, analysis of a second source standard prior to sample analysis	All reported analytes within $\pm 20\%$ of true value.	Correct problem. Rerun ICV. If that fails, repeat ICAL.	Flagging is not appropriate.	No samples shall be analyzed until calibration has been verified with a second source.
Continuing Calibration Verification (CCV)	Daily before sample analysis; after every 12 hours of analysis time; and at the end of the analytical batch run.	All reported analytes and surrogates within $\pm 20\%$ of true value. All reported analytes and surrogates within $\pm 50\%$ for end of analytical batch CCV.	Recalibrate, and reanalyze all affected samples since the last acceptable CCV; or Immediately analyze two additional consecutive CCVs. If both pass, samples may be reported without reanalysis. If either fails, take corrective action(s) and re-calibrate; then reanalyze all affected samples since the	If reanalysis cannot be performed, data must be qualified and explained in the case narrative. Apply Q-flag to all results for the specific analyte(s) in all samples since last acceptable calibration verification.	Results may not be reported without a valid CCV. Flagging is only appropriate in cases where the samples cannot be reanalyzed. If the specific version of a method requires additional evaluation (e.g., average RFs) these additional requirements must also be met.

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**STANDARD OPERATING PROCEDURE**

TITLE: ENV-SOP-MTJL-0081 Semivolatile Organics by GCMS using Capillary Column (EPA Methods 8270C, 8270D, 625, 625.1 and SM6410 B) Including Provisions for Analysis in SIM Mode

ISSUER: Pace National – Mt. Juliet, Tennessee

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Table 7. Quality Control Requirements – Organic Analysis by Gas Chromatography/Mass Spectrometry

QC Check	Minimum Frequency	Acceptance Criteria	Corrective Action	Flagging Criteria	Comments
Internal standards (IS)	Every field sample, standard and QC sample.	Retention time within ± 10 seconds from retention time of the midpoint standard in the ICAL; EICP area within - 50% to +100% of ICAL midpoint standard.	Inspect mass spectrometer and GC for malfunctions and correct problem. Reanalysis of samples analyzed while system was malfunctioning is mandatory.	If corrective action fails in field samples, data must be qualified and explained in the case narrative. Apply Q-flag to analytes associated with the non-compliant IS. Flagging is not appropriate for failed standards.	
Method Blank (MB)	One per preparatory batch.	No analytes detected $> \frac{1}{2}$ LOQ or $> 1/10$ the amount measured in any sample or $1/10$ the regulatory limit, whichever is greater. Common contaminants must not be detected $> \text{LOQ}$.	Correct problem. If required, reprep and reanalyze MB and all samples processed with the contaminated blank.	If reanalysis cannot be performed, data must be qualified and explained in the case narrative. Apply B-flag to all results for the specific analyte(s) in all samples in the associated preparatory batch.	Results may not be reported without a valid method blank. Flagging is only appropriate in cases where the samples cannot be reanalyzed.

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Table 7. Quality Control Requirements – Organic Analysis by Gas Chromatography/Mass Spectrometry

QC Check	Minimum Frequency	Acceptance Criteria	Corrective Action	Flagging Criteria	Comments
Laboratory Control Sample (LCS)	One per preparatory batch.	A laboratory must use the limits in Tables 3 through 6 for batch control if project limits are not specified. If the analyte(s) are not listed, use in-house LCS limits (see the LIMS) if project limits are not specified.	Correct problem, then reprep and reanalyze the LCS and all samples in the associated preparatory batch for failed analytes, if sufficient sample material is available	If reanalysis cannot be performed, data must be qualified and explained in the case narrative. Apply Q-flag to specific analyte(s) in all samples in the associated preparatory batch.	Must contain all surrogates and all analytes to be reported. Results may not be reported without a valid LCS. Flagging is only appropriate in cases where the samples cannot be reanalyzed.
Matrix Spike (MS)	One per preparatory batch.	A laboratory must use the limits in Tables 3 through 6 for batch control if project limits are not specified. If the analyte(s) are not listed, use in-house LCS limits (see the LIMS) if project limits are not specified.	Examine the project specific requirements. Contact the client as to additional measures to be taken.	For the specific analyte(s) in the parent sample, apply J-flag if acceptance criteria are not met and explain in the case narrative.	Must contain all surrogates and all analytes to be reported. If MS results are outside the limits, the data shall be evaluated to determine the source(s) of difference, i.e., matrix effect or analytical error.
Matrix Spike Duplicate (MSD) or Matrix Duplicate (MD)	One per preparatory batch.	A laboratory must use the limits in Tables 3 through 6 for batch control if project limits are not specified. If the analyte(s) are not listed, use in-house LCS limits (see the LIMS) if project limits are not specified. MSD or MD: RPD of all analytes $\leq 20\%$ (between MS and MSD or sample and MD).	Examine the project specific requirements. Contact the client as to additional measures to be taken.	For the specific analyte(s) in the parent sample, apply J-flag if acceptance criteria are not met and explain in the case narrative.	MSD: Must contain all surrogates and all analytes to be reported. The data shall be evaluated to determine the source of difference.

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QC Check	Minimum Frequency	Acceptance Criteria	Corrective Action	Flagging Criteria	Comments
Surrogate Spike	All field and QC samples.	QC acceptance criteria specified by the project, if available; otherwise use limits in Tables 3 through 6 or in-house LCS limits (see the LIMS) if analyte(s) are not listed.	Correct problem, then prep and reanalyze all failed samples for all surrogates in the associated preparatory batch, if sufficient sample material is available. If obvious chromatographic interference with surrogate is present, reanalysis may not be necessary.	Apply Q-flag to all associated analytes if acceptance criteria are not met and explain in the case narrative.	Alternative surrogates are recommended when there is obvious chromatographic interference.

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Table 8. Quality Control Requirements – Organic Semi-Volatile Analysis by GC/MS in SIM Mode

QC Check	Minimum Frequency	Acceptance Criteria	Corrective Action	Flagging Criteria	Comments
Tune Check	Prior to ICAL and prior to each 12-hour period of sample analysis.	Specific ion abundance criteria of DFTPP from method 8270. Tune check can be acquired as a full scan.	Retune instrument and verify.	Flagging is not appropriate.	No samples shall be analyzed without a valid tune. In addition to the full scan tune check, optimization for the analytes of interest is recommended.
Deuterated Monitoring Compounds (DMCs) (surrogates)	All field and QC samples.	PAH analysis: DMCs required for polyaromatic hydrocarbon (PAH) target analytes: fluoranthene-d10 and 2-methylnaphthalene-d10. Minimum RRF for PAH DMCs: 0.40. All DMCs: Requires 50-150% recovery until in-house limits can be established.	Correct problem, and then reprep and reanalyze all samples with failing DMCs if sufficient sample material is available. If obvious chromatographic interference is present, reanalysis may not be necessary, but the client must be notified prior to reporting data and the failures must be discussed in the Case Narrative.	Apply Q-flag to all associated samples and analytes if acceptance criteria are not met and explain in the Case Narrative.	For non-PAH target analytes, other DMCs with similar chemistry must be assigned. Laboratories may use the same extract for full scan and SIM analysis if the SIM-specific DMCs are added prior to extraction.

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Table 8. Quality Control Requirements – Organic Semi-Volatile Analysis by GC/MS in SIM Mode

QC Check	Minimum Frequency	Acceptance Criteria	Corrective Action	Flagging Criteria	Comments
Performance Checks	At the beginning of each 12-hour period, prior to analysis of samples.	Degradation $\leq$ 20% for DDT.	Correct problem, then repeat performance checks.	Flagging is not appropriate.	No samples shall be analyzed until the performance checks are within criteria. DDT breakdown and tailing factors are considered overall measures of port inertness and column performance and are required checks for SIM operation. DDT breakdown and tailing factor checks can be acquired as a full scan.
Initial Calibration (ICAL) for all analytes	At instrument set-up, prior to sample analysis.	Each analyte must meet one of the following options: RSD for each analyte $\leq$ 20% [If pentachlorophenol is a target analyte, an RSD of $\leq$ 40% allowed] Or Linear least squares regression for each analyte: $r^2 \geq 0.99$.	Correct problem then repeat ICAL.	Flagging is not appropriate.	Minimum 5 levels required for ICAL with one calibration point at the same concentration as the daily CCV. No samples shall be analyzed until ICAL has passed.

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Table 8. Quality Control Requirements – Organic Semi-Volatile Analysis by GC/MS in SIM Mode

QC Check	Minimum Frequency	Acceptance Criteria	Corrective Action	Flagging Criteria	Comments
Retention Time window position establishment	Once per ICAL and at the beginning of the analytical sequence.	Position shall be set using the midpoint standard of the ICAL curve when ICAL is performed. On days when ICAL is not performed, the initial CCV is used.	NA.	NA.	Calculated for each analyte.
Evaluation of Relative Retention Times (RRT)	With each sample.	RRT of each reported analyte within ± 0.06 RRT units of the mean RRT of the calibration standards. RRTs may be updated based on the daily CCV.	Correct problem, then rerun ICAL.	NA.	RRTs shall be compared with the most recently updated RRTs. Characteristic ions must maximize in the same scan or within one scan of each other. After any maintenance is performed which could affect retention times, RRTs may be updated based on the daily CCV.
Initial Calibration Verification (ICV)	Once after each ICAL, analysis of a second source standard prior to sample analysis.	All reported analytes within $\pm 20\%$ of true value. If pentachlorophenol is a target analyte, a %D from the true value of $\pm 50\%$ is allowed.	Correct problem. Rerun ICV. If that fails, repeat ICAL.	Flagging is not appropriate.	No samples shall be analyzed until calibration has been verified with a second source.

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Table 8. Quality Control Requirements – Organic Semi-Volatile Analysis by GC/MS in SIM Mode

QC Check	Minimum Frequency	Acceptance Criteria	Corrective Action	Flagging Criteria	Comments
Continuing Calibration Verification (CCV)	Daily before sample analysis; after every 12 hours of analysis time; and at the end of the analytical batch run.	Concentration the same as the mid-point calibration standard (or lower). All reported analytes within $\pm 20\%$ of true value. If pentachlorophenol is a target analyte, a %D from true value of $\pm 50\%$ is allowed. All reported analytes within $\pm 50\%$ for end of analytical batch within $\pm 50\%$ for end of analytical batch CCV.	Immediately analyze two additional consecutive CCVs. If both pass, samples may be reported without reanalysis. If either fails or if two consecutive CCVs cannot be run, perform corrective action(s) until a passing CCV is attained, and then reanalyze all associated samples since last acceptable CCV. Alternatively, perform an ICAL (including appropriate instrument QC) if necessary; then reanalyze all associated samples since the last acceptable CCV	If reanalysis cannot be performed, data must be qualified and explained in the Case Narrative. Apply Q-flag to all results for the specific analyte(s) in all samples since last acceptable calibration verification.	Results may not be reported without valid CCVs. Flagging is only appropriate in cases where the samples cannot be reanalyzed. If the specific version of a method requires additional evaluation (e.g., average RFs), these additional requirements must also be met.

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Table 8. Quality Control Requirements – Organic Semi-Volatile Analysis by GC/MS in SIM Mode

QC Check	Minimum Frequency	Acceptance Criteria	Corrective Action	Flagging Criteria	Comments
Internal Standards (IS)	Every field sample, Standards, blanks, and QC sample.	Retention time within ± 10 seconds from retention time of the midpoint standard in the ICAL; EICP area within 50% to +100% of ICAL midpoint standard. On days when ICAL is not performed, the initial CCV is used.	Inspect mass spectrometer and GC for malfunctions and correct problem. Reanalysis of samples analyzed while system was malfunctioning is mandatory.	If corrective action fails in field samples, data must be qualified and explained in the Case Narrative. Apply Q-flag to analytes associated with the non-compliant IS. Flagging is not appropriate for failed standards.	Internal Standard is spiked no greater than 0.40 ng/ μ L concentration. According to the EPA Contract Laboratory Program Statement of Work (CLP SOW), this is the concentration of internal standard specified for SIM analysis. The SOW indicates calibration standards range from 0.10 to 1.0 ng/ μ L, so 0.40 ng/ μ L is mid-range. 1, 4-dichlorobenzene-d4 is ignored for SIM
Method Blank (MB)	One per preparation batch, prior to analysis of any field samples.	No analytes detected $> \frac{1}{2}$ LOQ or $> 1/10$ th the amount measured in any sample or $1/10$ th the regulatory limit, whichever is greater.	Conduct investigation to determine the source of the contamination and take appropriate corrective actions. Correct problem. If required, reprep and reanalyze MB and all QC samples and field samples processed with the contaminated blank.	If reanalysis cannot be performed, data must be qualified and explained in the Case Narrative. Apply B-flag to all results for the specific analyte(s) in all samples in the associated analytical batch.	Laboratories may use the same extract for full scan and SIM analysis provided the applicable DMCs and IS are spiked at the appropriate concentrations. Results may not be reported without a valid Method Blank. Flagging is only appropriate in cases where the samples cannot be reanalyzed.

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Table 8. Quality Control Requirements – Organic Semi-Volatile Analysis by GC/MS in SIM Mode

QC Check	Minimum Frequency	Acceptance Criteria	Corrective Action	Flagging Criteria	Comments
Laboratory Control Sample (LCS)	One per preparation batch.	A laboratory must use Table 3 through Table 6 Limits (8270 SIM) for batch control if project specific limits are not specified. If the analyte(s) are not listed, use in-house LCS limits if project limits are not specified.	Correct problem, and then reanalyze the LCS and all samples in the associated analytical batch for failed analytes if sufficient sample material is available.	If reanalysis cannot be performed, data must be qualified and explained in the Case Narrative. Apply Q-flag to specific analyte(s) in all samples in the associated analytical batch.	Must contain all analytes to be reported. Results may not be reported without a valid LCS. Flagging is only appropriate in cases where the samples cannot be reanalyzed.
Matrix Spike (MS)	One per preparation batch.	A laboratory must use the QSM Appendix C Limits (8270 SIM) for batch control if project specific limits are not specified. If the analyte(s) are not listed, use in-house LCS limits if project limits are not specified.	Examine the project-specific requirements. Contact the client as to additional measures to be taken.	For the specific analyte(s) in the parent sample, apply the J-flag if acceptance criteria are not met and explain in the Case Narrative.	Must contain all analytes to be reported spiked at concentrations appropriate for SIM analysis. For matrix evaluation only. If MS results are outside the limits, the data shall be evaluated to determine the source(s) of difference (i.e., matrix effect or analytical error).

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QC Check	Minimum Frequency	Acceptance Criteria	Corrective Action	Flagging Criteria	Comments
Matrix Spike Duplicate (MSD) or Matrix Duplicate (MD)	One per preparation batch.	A laboratory must use the QSM Appendix C Limits (8270 SIM) for batch control if project specific limits are not specified. If the analyte(s) are not listed, use in-house LCS limits if project limits are not specified. MSD or MD: RPD of all analytes $\leq 40\%$ (between MS and MSD or sample and MD).	Examine the project-specific requirements. Contact the client as to additional measures to be taken.	For the specific analyte(s) in the parent sample, apply the J-flag if acceptance criteria are not met and explain in the Case Narrative.	The MSD must contain all analytes to be reported spiked at concentrations appropriate for SIM analysis. All data must be evaluated to determine the source of difference. For Sample/MD: RPD criteria only apply to analytes whose concentration in the sample is greater than or equal to the LOQ.
Characteristic ions for MS confirmation	Minimum 3 ions.	The relative intensities of the characteristic ions of target analytes agree within 30% of the relative intensities in the reference spectrum and the relative intensities must be > 0. Confirmation requires S/N ratio of ≥ 3 for each quant and confirmation ion.	No data can be reported without MS confirmation.	NA.	Need 3 structurally significant ions that are logical fragments – not isotopic clusters. Internal standard and DMC can use fewer than 3 ions.

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Document Title: Volatile Organic Compounds by GC/MS (EPA 8260B, 8260C, 624, 624.1 and SM 6200B)	
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Notes

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Signature Manifest**Document Number:** ENV-SOP-MTJL-0100**Revision:** 03**Title:** Volatile Organic Compounds by GC/MS (EPA 8260B, 8260C, 624, 624.1 and SM 6200B)

All dates and times are in Central Time Zone.

ENV-SOP-MTJL-0100**QM Approval**

Name/Signature	Title	Date	Meaning/Reason
Steven Miller (006597)	QA Director	19 Sep 2019, 02:49:02 PM	Approved

Management Approval

Name/Signature	Title	Date	Meaning/Reason
Heidi Ferrell (006481)	Volatiles Supervisor	20 Sep 2019, 08:22:33 AM	Approved



STANDARD OPERATING PROCEDURE

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1.0 SCOPE AND APPLICATION

STATE NOTE: For samples analyzed in conjunction with the Ohio Voluntary Action Program (VAP) please utilize ENV-SOP-MTJL-0099.

CLIENT NOTE: For clients, whose environment laboratory quality program is administered by Environmental Standards Inc. (ESI), see controlled document QUA-30 VOA.
<\\FAP\NovDiskH\QAQC\Controlled Docs>

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, 8260C, 8260D, 624.1, 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 ENV-SOP-MTJL-0016. 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 ENV-SOP-MTJL-0016, *Method Detection Limits (MDL), Limits of Detection (LOD) and Limits of Quantitation (LOQ)*. Should the procedure be utilized for DOD support; then the frequency of these studies must meet the requirements of the current DOD QSM.

1.3.2 Lower Limit of Quantitation (LOQ) – For analyses performed per the requirements of Method 8000D, the LLOQ is established at concentrations where both quantitative and qualitative requirements can consistently be met (see Sections 2.3 and 10.15).

1.4 Specific instructions for employees of the Pace Analytical National Center for Testing & Innovation (Pace National) Sacramento, California laboratory are included in this document as “SAC Notes”.

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 (ENV-SOP-MTJL-0131), 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



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to a mass spectrometer (MS) or a specific detector, depending on the determinative method selected.

METHOD NOTE: For Method 624.1, different sample sizes in the range of 5–25mL are allowed in order to meet differing sensitivity requirements. Calibration and QC samples must have the same volume as field samples.

- 2.2 For other samples, Method 5035 (ENV-SOP-MTJL-0129), volatile organic compounds are determined from a 5g sample combined with 5mL reagent water.
- 2.3 Lower Limit of Quantitation (LLOQ) – For analyses performed according to the requirements of Method 8000D, the lowest concentration at which the laboratory has demonstrated target analytes can be reliably measured and reported with a certain degree of confidence, which must be greater than or equal to the lowest point in the calibration curve.
- 2.4 See the current Quality Assurance Manual for definitions associated with terms found in this document.

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. A reference file of safety data sheets (SDSs) are made available on Pace National's intranet to all personnel. Use hazardous 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 protocols.
- 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 methanol 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 seven (7) days. It is also a Pace National requirement that water samples with 2-Chloroethyl vinyl ether (2-CEVE), as a compound of interest, be collected unpreserved and analyzed within 7 days of collection. 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 hours from the time of collection, added to preservative or otherwise frozen at $\leq -7^{\circ}\text{C}$. High-level soil samples collected in Encore™ or equivalent type sampling devices must be placed in vials of methanol according to Method 5035 (ENV-SOP-MTJL-0129).

STATE NOTE: The State of South Carolina 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



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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 minimal or no headspace to minimize the loss of highly volatile analytes. Sample kits can be configured to request additional vials per client request. Typically the pH is adjusted to <2 with HCl, but the preservation requirements may vary by EPA program and may be specified in a regulation or project planning document that requires compliance monitoring for a given contaminant. An example of this is some samples from Missouri or Kansas may be preserved with tri-sodium phosphate and will have a resulting pH>12.
- 4.4 Soil samples may 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 NaHSO₄ to a pH<2 and one 5g sample preserved in the field with methanol (for high level analysis or data generated on Agilent™ 5977A or 5977B instruments [with an extractor ion or high efficiency source] may be reported to low-level MDL/RL values due to the enhanced sensitivity associated with these instruments) 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 and for screening purposes. 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>12.

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.

4.5 Method 624.1 Considerations

- 4.5.1 If acrolein is to be determined, analyze the sample within three (3) days. To extend the holding time to 14 days, acidify a separate sample to pH 4–5 with HCl.
- 4.5.2 Experimental evidence indicates that some aromatic compounds, notably benzene, toluene, and ethyl benzene are susceptible to rapid biological degradation under certain environmental conditions. Refrigeration alone may not be adequate to preserve these compounds in wastewaters for more than seven days. To extend the holding time for aromatic compounds to 14 days, acidify the sample to approximately pH 2.
- 4.5.3 If halocarbons are to be determined, use an acidified sample.
- 4.5.4 Ethers are prone to hydrolysis at pH 2 when a heated purge is used. Aqueous samples should not be acid preserved if ethers are of interest or if the alcohols they would form upon hydrolysis are of interest and the ethers are anticipated to present.



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- 4.5.5 2-Chloroethyl vinyl ether is subject to hydrolysis at low pH; therefore, determine 2-Chloroethyl vinyl ether from the un-acidified sample.

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 may be analyzed to check for cross contamination. Alternatively, when analysis of a blank is not possible prior to the next sample, such as when an unattended autosampler is employed, the analyst should review the results for at least the next sample after the high-concentration sample. If analytes in the high-concentration sample are not present in the subsequent field sample, then the lack of carryover has been demonstrated.
- 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 by using a portable PID or equivalent.
- 5.4 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 III and are recommended from the published 8260 methods.
- 5.5 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



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exposed to methylene chloride fumes during liquid/liquid extraction procedures can contribute to sample contamination.

- 5.6 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 every two weeks to check for cross contamination of samples while samples are stored in the volatiles laboratory walk-in cooler. The storage blanks are purchased from a vendor and must matrix match the contents of the cold room or refrigerator and is placed in the cooler for a period of two weeks. Every two weeks, the applicable storage blanks are analyzed to verify that no contamination of client samples has taken place due to contamination in the storage unit.
- 5.7 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.
- 5.8 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.
- 5.9 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.

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. There are a total of 36 instruments; GCs include models 6890, 7890, Intuvo or equivalent and MSs include 5973, 5975, 5977b, HES (High efficiency source) or equivalent. Specific information for each instrument is included in the associated maintenance logbook.

Sample introduction system: Archon Autosampler, EST Centurion, OI 4760, Encon P & T, EST Evolution, OI 4660 or equivalent
- 6.2 **Glassware:** volumetric glassware equipped with penny head ground glass stopper. The volumetric flasks are cleaned by rinsing with methanol and laboratory reagent water. The volumetric flasks can be dried in a low temperature oven at less than 120 °C and are 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, 500µL, 1mL, and 5mL.



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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)
- 6.5.3 2mL amber vial with Teflon™/silicone/Teflon™ septa (used to store unused standards)

6.6 Miscellaneous

- 6.6.1 Stainless Steel Spatula or wooden tongue depressor
- 6.6.2 Teflon™-coated stir bars, 8mm x 16mm
- 6.6.3 Glass beads –VWR™ #EM1.04015.0500, or equivalent

6.7 Oven: Fisher IsoTemp Forced-Air Oven with capabilities of 100°C, or equivalent

6.8 Analytical 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 ENV-SOP-MTJL-0041, *Standards Logger – Tree Operation*. 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 six months or sooner if a problem is detected unless otherwise noted. Working standards for most compounds should be replaced after four weeks unless the integrity of the standard is suspected to of being compromised prior to that time. Working standards for gases should be replaced after one week unless the acceptability of the standard can be documented.

7.2 Nanopure water or equivalent: Nanopure water is used in all blanks to ensure it contains less than the method detection limit (MDL) of all compounds of interest. If volatile compounds are detected in the blank above MDL all samples associated with this blank must be flagged (see Section 11.5).

NOTE: For all DoD samples the laboratory water is used in all blanks to assure that it contains less than 1/2 LOQ 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 1/2 LOQ, then those samples must be flagged.

7.3 Methanol, CH₃OH – purge-and-trap grade, demonstrated to be free of analytes. Store apart from other solvents.

7.4 Sodium Bisulfate, Na₂S₂O₃ - QEC Level 3 Certified, or equivalent

7.5 STOCK SOLUTIONS – Primary and Secondary Sources

- Stock calibration solutions must be purchased as certified solutions.
- Certificates must be kept on file.
- All Stock standards must be stored ≤ 6°C or as recommended by the standard manufacturer.
- All non-gas stock standards must be replaced after six months, or sooner, if check standards indicate a problem.



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- Both gas and liquid standards must be monitored closely by comparison to the initial calibration curve and by comparison to a second source ICV, or Secondary Source Verification Standard (SSCV).
- 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 Primary Source - Primary source standards are used to prepare the initial 5-point calibration curve (additional levels may be used as needed), the continuing calibration verification (CCV) standard, LCS and matrix spikes. The CCV is analyzed to verify the initial calibration and is prepared using the primary source standard 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. The standard list of target LCS compounds are those compounds listed in Attachment VIII. The LCS 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."

STATE NOTE: South Carolina DHEC and the USACE require that all target analytes are present and evaluated in the LCS

Calibration Mix

The calibration mix is prepared in methanol in a 25mL volumetric flask by adding:

Manufacturer	Product	Cat. #	Amount added (mL)	Final Conc. (ppm)
Phenomenex	Custom 8260 Calibration Mix	AL0-130113	1	100
Phenomenex	Custom Ketones Mix	AL0-130115	1	500
Phenomenex	Acrolein	AL0-130159	1	500
Phenomenex	Custom 8260 Additions Mix	AL0-130114	2	100
Phenomenex	Vinyl Acetate	AL0-130117	1	500
Phenomenex	2-Chloroethyl vinyl ether	AL0-130116	1	500

7.5.1.1 The calibration standard mix is prepared by making a 1:1 ratio of the gas standard (Agilent, ULTRAgold™ Custom Standard CUS-27747 or equivalent at 100µg/mL) and the calibration mix standard.

7.5.1.2 For soil 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 ≤6° C.



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AP9/Oxygenates Calibration Mix

The AP9/Oxygenate ICV solution is prepared in methanol in a 25mL volumetric flask by adding:

Manufacturer	Product	Cat. #	Amount added (mL)	Final Conc. (ppm)
NSI	Ethyl Acetate	681	0.125	50
NSI	Custom AP9 Standard	Q-6603-O	3.125	5
Absolute	Isopropanol	70941	0.625	25

- 7.5.2 Secondary Source - Secondary source standards must be used to prepare the secondary source verification standard (SSCV) or initial calibration verification (ICV). 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 $\leq 6^{\circ}\text{C}$ 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.

SSCV

Prepare the SSCV mix in methanol in a 25mL 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)
NSI	8260 Custom Mix 2	Q-4147	1	500
Restek	Acrolein	30645	2.5	500
SPEX CertiPrep	AZ fuel additive 4 comps.	VO-ESCTN9	2	100
Ultra	Custom Standard	CUS-18354	1	100

- 7.5.2.1 The working second source (SSCV) or Initial Calibration Verification (ICV) is prepared by making a 1:1 ratio of the gas standard (Phenova, VOA Gas Calibration Mix AL0-130108 or equivalent at 200 $\mu\text{g/mL}$) and the second source standard.

- 7.5.2.2 For soil 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 .

AP9/Oxygenates SSCV

The AP9/Oxygenates SSCV is prepared in methanol in a 25mL volumetric flask by adding:



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Manufacturer	Product	Cat. #	Amount added (mL)	Final Conc. (ppm)
NSI	Custom AP9 Standard	Q-6603-O	3.125	5
NSI	Ethyl Acetate	681	.125	50
Absolute	Isopropanol	70941	.625	25

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 $\leq 6^{\circ}\text{C}$ and have a six month holding time, once opened. Surrogate spiking solutions are purchased from Phenova, Part# AL0-130491, 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 Pace National designated volatiles' analysis surrogates:

- Toluene-d8
- 4-Bromofluorobenzene
- 1,2-Dichloroethane-d4

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 $\leq 6^{\circ}\text{C}$ and have a six month holding time, once opened. [Internal standard and surrogate standard – Phenova, Part# AL0-130491, or equivalent]

7.7.1 The following are Pace National designated volatiles' analysis internal standards:

- 1,4-Dichlorobenzene-d4
- Fluorobenzene
- Chlorobenzene-d5

SAC NOTE: Internal Standard/Surrogate Mix – o2si 8260 Internal Surrogate Solution Catalog No.: 121722-08-20PAK. This mix is diluted 250x to produce a 50 $\mu\text{g/mL}$ internal standard and surrogate mix. During analysis, 5 μL of this solution is added to 5mL of sample, resulting in a concentration of 50 $\mu\text{g/L}$. The internal standards and surrogates are the same as listed in 7.6.1 and 7.7.1 with the addition of Tert-butyl Alcohol-d10 as an internal standard.

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 $\leq 6^{\circ}\text{C}$ and have a six month holding time, once opened.

7.9 Matrix spike (MS) standard - Stock standards must be stored at or below $\leq 6^{\circ}\text{C}$ 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.



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- 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 presented in the LIMS.

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%.

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. Soils require method 5035 for sample preparation, See ENV-SOP-MTJL-0129.

- 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 – 3×10^{-6} torr

8.2 Initial calibration

- 8.2.1 TUNING - Each GC/MS system must be hardware-tuned ($1\mu\text{L} \leq 50\text{ng}$) 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 624.1, 8260B, 8260C, and 6200B. BFB tuning for method 8260D is prior to each calibration.

BFB Key Ions and Ion Abundance Criteria for 8260B, 8260C, 8260D, 624.1	
Mass	Ion Abundance Criteria
95	50 - 200% of mass 174
96	5 - 9% of mass 95
173	< 2% of mass 174
174	50 - 200% of mass 95



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BFB Key Ions and Ion Abundance Criteria for 8260B, 8260C, 8260D, 624.1	
175	5 - 9% of mass 174
176	95 - 105% of mass 174
177	5 - 10% of mass 176

BFB Key Ions and Ion Abundance Criteria for 6200	
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

EPA 624.1 NOTE: The 12-hour tune clock begins after analysis of the BFB, the LCS, and the BLANK and ends 12 hours later. BFB, the LCS, and BLANK are outside of the 12-hour tune clock. The MS and MSD are treated as samples are analyzed within the 12-hour clock.

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: The reporting level standard must be refit 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.

STATE NOTE: When analyzing samples from Minnesota, 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



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separate reporting level standard following calibration curve update. This standard must recover $\pm 40\%$ of the expected concentration. If the criterion 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, ENV-SOP-MTJL-0129, 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 $\pm 30\%$ of the standard's true concentration.
 - **EPA 8260D 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. A minimum quantitation check on the viability of all calibration points should be performed by re-fitting the response from each calibration standard back into the curve. The recalculated concentration of the reporting level point should be within $\pm 50\%$ of the standard's true concentration. All other recalculated concentration calibration points should be within $\pm 30\%$ of the standard's true concentration. If a failure occurs in the low point and it is equivalent to the LLOQ, the analyte should be reported as estimated near that concentration or the LLOQ should be reestablished at a higher concentration.
- STATE NOTE:** South Carolina does not allow the use of quadratic regression for compounds that have previously demonstrated linearity.
- The method reference spectra must be updated from the mid-point of each calibration.

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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 Calibration Mix (See Section 7.5.1) into 5mL of reagent water. Surrogate standard is prepared by diluting the surrogate by 1:10 using (NSI lab solutions, 8260 Surrogate Mix Q-4392 or equivalent at 1000µg/mL).

8260 Calibration Curve - GC/MS Soil (into 5mL water)			
Intermediate solution volume (µL)	Concentration of standard (ppb)	Surrogate Added (µL)	Concentration of Surrogate (ppb)
0.025	0.25 (LOD Point)	0	n/a
0.05	0.5	0	n/a
0.1	1	1	1
0.2	2	2	2
0.5	5	3	3
2.5	25	4	4
7.5	75	5	5
10	100	6	6
20	200	7	7

8260 Calibration Curve - GC/MS Soil (into 50mL water) for use with Agilent 5977A or 5977B Only			
Intermediate solution volume (µL)	Concentration of standard (ppb)	Surrogate Added (µL)	Concentration of Surrogate (ppb)
0.25	0.25 (LOD Point)	0	n/a
0.5	0.5	0	n/a
1	1	1	1
2	2	2	2
5	5	3	3
25	25	4	4
75	75	5	5
100	100	6	6
200	200	7	7

AP9/Oxygenates Calibration Curve - GC/MS Soil (into 5mL of water)	
Intermediate solution volume (µL)	Concentration of standard (ppb)
1	1.0
5	5
10	10
15	15
20	20

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AP9/Oxygenates Calibration Curve - GC/MS Soil (into 50mL of water)	
Intermediate solution volume (µL)	Concentration of standard (ppb)
10	1.0
50	5
10	10
150	15
200	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.

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 Calibration Mix (See Section 7.5.1.) into 50mL of water. Surrogate standard is added to the curve points, (Phenova, Custom IS-SURR Mix AL0-130574 or equivalent at 1000µg/mL).

8260 Calibration Curve - GC/MS Water (into 50mL water)			
Intermediate solution volume (µL)	Concentration of standard (ppb)	Surrogate Added (µL)	Concentration of Surrogate (ppb)
0.25	0.25 (LOD Point)	0	n/a
0.5	0.5	0	n/a
1	1	1	1
2	2	2	2
5	5	3	3
25	25	4	4
75	75	5	5
100	100	6	6
200	200	7	7

AP9/Oxygenates Calibration Curve - GC/MS Water (into 50mL of water)	
Intermediate solution volume (µL)	Concentration of standard (ppb)
10	1.0
50	5
10	10
150	15

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AP9/Oxygenates Calibration Curve - GC/MS Water (into 50mL of water)	
Intermediate solution volume (µL)	Concentration of standard (ppb)
200	20

SAC NOTE: See Controlled Document DAV-03 VOA ICAL Reference for calibration table.

8.2.4 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 is equivalent of 16µg/L of each surrogate/internal 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.

SAC NOTE: During analysis, 5µL of this solution is added to 5mL of sample, resulting in a concentration of 50 µg/L

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.

8.2.5 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:

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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 or Argon) 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.

8.2.6 **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.

8.2.6.1 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

8.2.6.2 Target Analytes and other Non-CCC's - The RSD must meet the following criteria -

<15% RSD for all 8260B Target Analytes
<20% RSD for all 8260C/D Target Analytes
<35% RSD for all 624.1 Target Analytes
<20% RSD for all KSGRO Samples
<15% RSD for n-Hexane
<20% 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



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8.2.6.3 Linear Regression - Criteria

When any compound does not meet the calibration criteria for RF, the most appropriate curve fitting model is used. 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 (r) criteria of 0.995 or better. SM 6200 requires a linear regression correlation coefficient of >0.994.

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.

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.

The most appropriate curve fitting model from among the following choices must be utilized (given in the order of preference): Average Response Factor

- Linear – No Weighting
- Linear – 1/x Weighting
- Linear – 1/x² Weighting
- Quadratic

STATE NOTE: South Carolina does not allow the use of quadratic regression for compounds that have previously demonstrated linearity. 8.2.6.4 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:

- 8.2.6.4.1 Adjust the instrument and/or perform instrument maintenance and re-analyze the calibration standards until the RSD of the calibration meets criteria.
- 8.2.6.4.2 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



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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 acrylonitrile fall into this category.

8.2.7 **EPA 8260C:** Response Factors (RF's)

8.2.7.1 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 these 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.

8.2.7.2 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.

NOTE: To maximize batches calibration RSDs are held to the strictest method reported, the RSD used for all methods is 15%.

8.2.8 **EPA 8260D:** Response Factors (RF's)

8.2.8.1 Calibration Curve Criteria – Calculate the mean RF and the relative standard deviation (RSD) of the RFs for each target analyte. The RSD should be $\leq 20\%$ for each target analyte. Attachment IX contains minimum RFs that may be used as guidance in determining whether the system is behaving properly and as a check to see if calibration standards are prepared correctly. It is neither expected nor required that all analytes meet these minimum RFs in the guidance table, however target analytes must not have an RF < 0.01 . Due to the large number of compounds that may be analyzed by this method, some compounds will fail to meet these 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.



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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 as the inspection can be a useful diagnostic tool.

NOTE: To maximize batches calibration RSDs are held to the strictest method reported, the RSD used for all methods is 15%

8.2.9 EPA 624.1

Calculate the mean (average) and relative standard deviation (RSD) of the response factors. If the RSD is less than 35%, the RF can be assumed to be invariant and the average RF can be used for calculations. Alternatively, the results can be used to fit a linear or quadratic regression of response ratios vs. concentration ratios. If used the regression must be weighted inversely proportional to concentration. The coefficient of determination (R^2) of the weighted regression must be greater than 0.920 (this value roughly corresponds to the RSD limit of 35%). Alternatively, the relative standard error may be used as an acceptance criterion. As with the RSD, the RSE must be less than 35%. If an RSE less than 35% cannot be achieved for a quadratic regression, system performance is unacceptable, and the system must be adjusted and re-calibrated.

NOTE: Using capillary columns and current instrumentation, it is quite likely that a laboratory can calibrate the target analytes in this method and achieve a linearity metric (either RSD or RSE) well below 35%. Therefore, laboratories are permitted to use more stringent acceptance criteria for calibration than described here (e.g., to harmonize their application of this method with those from other sources).

STATE NOTE: South Carolina does not allow the use of quadratic regression for compounds that have previously demonstrated linearity.

8.3 Calibration Verification

8.3.1 Calibration Verification for EPA Methods 8260B, 624.1 and SM 6200B:

8.3.1.1 SSCV's

After a successful calibration, a Second Source Calibration Verification (SSCV) or Initial Calibration Verification (ICV) 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

8.3.1.2 CCC's

The curve must be verified daily by a calibration standard known as the Continuing Calibration Verification standard (CCV) and is analyzed every 12 hours. This standard is prepared at or near the mid-point of the calibration curve. A maximum of 20% criteria would be expected for



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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,

$$\% \text{ Difference} = (\text{RF}_v - \text{Rf}_{\text{ave}}) / \text{Rf}_{\text{ave}} \times 100\%$$

Compounds on regression fit model use percent drift,

$$\% \text{ Drift} = (\text{Calculated conc} - \text{Theoretic conc}) / \text{Theoretic conc} \times 100\%$$

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 8260B and 624.1 concurrently, calibration verifications are evaluated using both the 8260B criteria (sections 8.3.1.2 through 8.3.1.3 and the 624.1 criteria (Attachment VI). For analytes not on the 624.1 list, all target analytes (except for the poor performers (8.3.3) 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.

STATE NOTE: For South Carolina 624.1 samples, the target analytes reported must agree with the compound list found in the EPA 624.1 published method.

8.3.2 Calibration Verification for EPA Method 8260C/D:

8.3.2.1 SSCV's

After a successful calibration, a Second Source Calibration Verification (SSCV) or Initial Calibration Verification (ICV) 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	$\pm 30\%$
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 Continuing Calibration Verification standard (CCV) and is analyzed every 12 hours). 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.



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If the percent difference (average RSD) or percent drift (linear regression models) 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, a reporting level verification is analyzed. Reference QUA-12 *CCV Criteria for 8260C and 8270D* for assessing reporting level verification.

All compounds must have a minimum response factor. If these criteria are exceeded, then corrective action is required. The minimum response factors are generated in house based off of historical data.

8.3.3 Poor Performers:

The poor performers are as follows:

Propene	2-Chloroethylvinyl Ether
Dichlorodifluoromethane	Acrolein
Carbon Disulfide	Vinyl acetate
Bromomethane	trans-1, 4-dichloro-2-butene
Chloroethane	Alcohols (Ethanol, TBA, TAA, ETBA, Butanol)
1,3-Butadiene	Iodomethane
1,2-Dibromo-3-chloropropane	Naphthalene
1- Methylnaphthalene	2-Butanone
2- Methylnaphthalene	2-Hexanone
Acetone	4-Methyl-2-pentanone
Pentachloroethane	Cyclohexanone
Tert-butyl Formate	Methyl cyclohexane

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 found in the LIMS for LCS and MS/MSD.
- If the stated criteria are exceeded, then corrective action is required.
- See Section 11.6.1, on marginal exceedances.



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STATE NOTE: All **South Carolina** DHEC compliance testing, the LCS responses must be within 70 – 130% for Method 8260 and within the limits given in Attachment VI for Method 624.1. High failures are acceptable for 8260 or 624.1, as long as the sample results are below detection level. Qualifiers cannot be used therefore low failures require a batch re-analysis. See Section 10 for QC evaluation. For samples analyzed from South Carolina that are not utilized for compliance purposes, in house established acceptance limits are utilized to demonstrate controlled analyses.

STATE NOTE: For EPA Region IV only (AL, FL, GA, KY, MS, NC, SC, TN), when running Acrolein and Acrylonitrile by V624.1, the QC criteria from EPA Method 603 should be used for control demonstration. The LCS criteria from EPA Method 603 include Acrolein recovery within 88-118% and Acrylonitrile recovery within 71-135%.

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 CCV. 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 5-point for GRO (if analyzed)
Second Source Calibration Verification (SSCV) or Initial Calibration Verification(ICV)	Verify initial calibration with second source.
Laboratory Control Sample	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

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Sample/QC Type	Use
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

- 8.4.2 Typical sequence order for loading the autosampler with calibration verification only:

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 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 by screening of 5mL of sample using an HNU or equivalent portable PID. See ENV-SOP-MTJL-0102, *Volatile Organic Compounds Screening using the RAE Systems Photoionization Gas Detector Model MiniRAE 3000*.
- 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.
- 8.5.5 Load the unopened VOA vial onto the autosampler for analysis.



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After the sample has been analyzed 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 “G1” qualifier. All samples that report 2-CEVE as a target analyte and have a pH < 2 are qualified with a “G2”.

STATE NOTE: For West Virginia compliance samples, check for residual chlorine after sample analysis. Samples containing residual chlorine must be flagged as such.

8.5.6 Sample Dilution -- When necessary, samples can be diluted before purging. This can be performed in a clean 50mL volumetric flask. The sample is measured through the use of an appropriate microliter syringes and added to the flask which is then filled with reagent water to the meniscus.

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 All vials for the sample are combined in an appropriate sized volumetric flask that will allow for the least amount of headspace. (If 4 vials are to be composited then a 200mL volumetric flask will be used to combine the samples.). Practice special precautions to maintain zero headspace in the syringe.

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 Invert the volumetric flask three (3) times. Pour the volume out of the volumetric flask into the original 40mL VOA vial containers. The sample is now ready to be analyzed.

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 1µL of the IS/surrogate mix to each sample. The addition of 1µL of the surrogate spiking/internal standard solution is equivalent to 16µg/L of each surrogate standard. Internal standard and surrogate standard are contained within the same spiking mix.

SAC NOTE: During analysis, 5µL of this solution is added to 5mL of sample, resulting in a concentration of 50 µg/L of each surrogate and internal standard.

8.5.9 Refer to ENV-SOP-MTJL-0131, *Purge and Trap for Aqueous Samples* and ENV-SOP-MTJL-0129, *Closed-System Purge-and-Trap and Extraction for Volatile Organics in Soil and Waste Samples* 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-



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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 25-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 40mL vial filled with organic-free reagent water by adding at least 0.5µL, but not more than 25mLs of liquid sample. The sample is ready for addition of internal and surrogate standards. Proceed with Section 8.5.8.

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 - 1mg/Kg) or the high-concentration method (>1mg/Kg).

8.7.1 Low-concentration method -- This is designed for samples containing individual purgeable compounds of <1mg/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 glass beads or equivalent blank matrix.

STATE NOTE: This option cannot be used for South Carolina samples. Please refer to ENV-SOP-MTJL-0129 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 (ENV-SOP-MTJL-0129) 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



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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 nanopure 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.

- 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 may be purged at higher temperatures than ambient temperature as long as all calibration standards, field samples, and associated QC samples are purged at the same temperature and the laboratory demonstrates acceptable method performance. 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 ENV-SOP-MTJL-0129.

- 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 40mL 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 and vortex. See ENV-SOP-MTJL-0129.

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 200µL.

- 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 1µL of the IS/surrogate mix to all of the samples.



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- 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.
- 8.7.2.7 For a matrix spike in the high-concentration of sediment/soil samples, Add a 200µL 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).
- 8.7.2.8 Data generated from soil samples prepared in methanol on Agilent™ 5977A or 5977B instruments (with an extractor ion or high efficiency source) may be reported to low-level MDL/RL values due to the enhanced sensitivity associated with these instruments.

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.
- 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



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each analyte spectrum contains extraneous ions contributed by the co-eluting compound.



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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 Pace National.

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 15% 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.

8.8.6.1 Routinely, Pace National employs a minimum Q value of 85% for tentative identifications and a minimum concentration of 10ppb. Peaks below a Q value of 85% but above 10ppb may be reported as "Unknown". Any identified peaks below 10ppb are removed as these could result from baseline noise or other interferences, not necessarily attributable to the field sample or reliably quantifiable using GCMS technology. Additionally, any peaks that are attributable to instrument contamination (e.g., siloxanes) are also removed.

8.8.6.2 If multiple TICs, with same exact name, exist for a sample, the LIMS will only display the TIC with the highest quality match per sample.

8.8.6.3 TIC names assigned as "Unknown" may initially have the same name as another "Unknown" until parsed and displayed in LIMS where it is given a hyphen and incremental number which then becomes a unique TIC (e.g., Unknown-1).



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8.8.6.4 When reporting “Total TIC” for any client sample, only concentrations per above requirements will be used to sum the Total TIC concentration.

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 Pace National, 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 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.

9.2 Concentration of Target Analytes in Sediment/Soil, Sludge, and Waste

9.2.1 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.

V_t = Volume of total extract (μL) (use 10,000 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 purged (g). The wet weight or dry weight may be used, depending upon the specific applications of the data.



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9.2.2 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 10,000μL 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 purged (g). The wet weight or dry weight may be used, depending upon the specific applications of the data.

9.2.3 Soil Weight determination with Methanol (samples received with MeOH).

$$\text{SoilWeight} = \text{VialTotalWeight(vial, soil, MeOH)} - \text{TareVialWeight} - \text{MeOHWeight}.$$

- 9.3 In order to report results for volatiles analysis of samples prepared in methanol 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 V_t. This total solvent/water volume is calculated as follows:

$$\mu\text{L solvent/water } V_t = \left[\frac{\text{mL of solvent} + (\% \text{ moisture} \times \text{g of sample})}{100} \right] \times 1000 \mu\text{L/mL}$$

9.4 Percent Error (%Error)

$$\%Error = \frac{x_i - x'_i}{x_i} * 100$$

where:

x'_i = Measured amount of analyte at the calibration level *i*, in mass or concentration units

x_i = True amount of analyte at calibration level *i*, in mass or concentration units

- 9.5 Relative Standard Error (%RSE) – As an alternative to using the average response factor when using Method 624.1, the quality of the calibration may be evaluated using the Relative Standard Error (RSE). The acceptance criterion for the RSE is the same as the acceptance criterion for Relative Standard Deviation (RSD), in the method. RSE is calculated as:

$$\%RSE = 100 \times \frac{\sum_{i=1}^n \left[\frac{x'_i - x_i}{x_i} \right]^2}{(n - p)}$$

where:

x'_i = Calculated concentration at level *i*

x_i = Actual concentration of the calibration level *i*

n = number of calibration points

p = Number of terms in the fitting equation (average = 1; linear = 2; quadratic = 3)



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- 9.6 See the current Quality Assurance Manual for other equations associated with common calculations.

10.0 QUALITY CONTROL AND METHOD PERFORMANCE

- 10.1 All analysts must meet the qualifications specified in ENV-SOP-MTJL-0015, *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.

STATE NOTE: IDOCs for South Carolina DHEC compliance need to meet 70-130% for all compounds except the poor purgers. Poor purgers need to recover at 60-140%. For samples analyzed from South Carolina that are not utilized for compliance purposes, in house established acceptance limits are utilized to demonstrate controlled analyses.

METHOD NOTE: IDOCs for SM 6200B should have an average recovery of 80-120% for each target analyte and the RSD of the replicates should be <20%.

10.1.1 Method 624.1 Requirements

- 10.1.1.1 Establish MDLs for the analytes of interest using the MDL procedure at 40 CFR part 136, appendix B. The laboratory's MDLs must be equal to or lower than those listed in Table 1 for those analytes which list MDL values, or lower than one-third the regulatory compliance limit, whichever is greater. For MDLs not listed in Table 1, the laboratory must determine the MDLs using the MDL procedure at 40 CFR part 136, appendix B under the same conditions used to determine the MDLs for the analytes listed in Table 1. All procedures used in the analysis must be included in the DOC.

624.1 Purgeable Analytes	MDL (ug/L)	Limit for s (%)	Range for $\bar{X}$ (%)
Acrolein		30	50-150
Acrylonitrile		30	50-150
Benzene	4.4	33	75-125
Bromodichloromethane	2.2		
Bromoform	4.7		
Bromomethane		90	D-242
Carbon tetrachloride	2.8	26	65-125
Chlorobenzene	6.0	29	82-137
Chloroethane		47	42-202
2-Chloroethylvinyl ether		130	D-252
Chloroform	1.6	32	68-121
Chloromethane		472	D-230
Dibromochloromethane	3.1	30	69-133
1,2-Dichlorobenzene		31	59-174
1,3-Dichlorobenzene		24	75-144
1,4-Dichlorobenzene		31	59-174
1,1-Dichloroethane	4.7	24	71-143

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624.1 Purgeable Analytes	MDL (ug/L)	Limit for s (%)	Range for $\bar{X}$ (%)
1,2-Dichloroethane	2.8	29	72-137
1,1-Dichloroethene	2.8	40	19-212
trans-1,2-Dichloroethene	1.6	27	68-143
1,2-Dichloropropane	6.0	69	19-181
cis-1,3-Dichloropropene	5.0	79	5-195
trans-1,3-Dichloropropene		52	38-162
Ethylbenzene	7.2	34	75-134
Methylene chloride	2.8	192	D-205
1,1,2,2-Tetrachloroethane	6.9	36	68-136
Tetrachloroethene	4.1	23	65-133
Toluene	6.0	22	75-134
1,1,1-Trichloroethane	3.8	21	69-151
1,1,2-Trichloroethane	5.0	27	75-136
Trichloroethene	1.9	29	75-138
Trichlorofluoromethane		50	45-158
Vinyl chloride		100	D-218

- 10.1.1.2 Prepare and analyze four LCSs by adding an appropriate volume of the second source standard (Section 7.5.2.1) to each of four aliquots of reagent water.
- 10.1.1.3 Calculate the average percent recovery ($\bar{X}$) and the standard deviation of the percent recovery (s) for each analyte using the four results.
- 10.1.1.4 For each analyte, compare s and $\bar{X}$ with the corresponding acceptance criteria for precision and recovery in Section 10.1.1.1. For analytes not listed, DOC QC acceptance criteria must be developed by the laboratory. Alternatively, acceptance criteria for analytes not listed may be based on laboratory control charts. If s and $\bar{X}$ for all analytes of interest meet the acceptance criteria, system performance is acceptable and analysis of blanks and samples may begin. If any individual s exceeds the precision limit or any individual $\bar{X}$ falls outside the range for recovery, system performance is unacceptable for that analyte.

NOTE: The large number of analytes present, a substantial probability that one or more will fail at least one of the acceptance criteria when many or all analytes are determined simultaneously. Therefore, the analyst is permitted to conduct a “re-test” as described here.

When one or more of the analytes tested fail at least one of the acceptance criteria, repeat the test for only the analytes that failed. If results for these analytes pass, system performance is acceptable and analysis of samples and blanks may proceed. If one or more of the analytes again fail, system performance is unacceptable for the analytes that failed the acceptance criteria. Correct the problem and repeat the test. To maintain the validity



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of the test and re-test, system maintenance and/or adjustment is not permitted between this pair of tests.

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 (LCS)), 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, and an LCS.

Additional instructions on Batch QC including required frequency and corrective actions can be found in Section 11 while acceptance criteria are found in the LIMS.

STATE NOTE: For samples from FL, AZ, MN and MA, 1 Laboratory Control Sample/Laboratory Control Sample Duplicate pair (LCS/LCSD) is required per batch.

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, and/or 1 Continuing Calibration Verification (CCV) and BFB tune every 12 hours.

- 10.3 Perform BFB tune every 12 hours for 624.1, 8260B, 8260C, and 6200B. BFB tuning for method 8260D is prior to calibration curve. Tuning acceptance criteria are presented in Section 8.2.1. Once the data is acquired, 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. If Autofind is not utilized, select the mass spectrum at the peak apex for evaluation, or use an average mass spectrum across the entire BFB peak. Background subtraction is conducted using a single scan prior to the elution of BFB but no more than 20 scans prior. No part of the BFB peak or any other discrete peak should be subtracted. If a tune cannot be achieved that meets the criteria in Section 8.2.1 retune the mass spectrometer and run a new calibration or maintenance may be necessary.

10.3.1 Method 624.1 Requirements

- 10.3.1.1 Verify calibration after the criteria for BFB are met and prior to analysis of a blank or sample. After verification, analyze a blank to demonstrate freedom from contamination and carry-over at the MDL. Tests for BFB, the CCV, and the blank are outside of the 12-hour shift, and the 12-hour shift includes samples and matrix spikes and matrix spike duplicates. The total time for analysis of BFB, the CCV, the blank, and the 12-hour shift must not exceed 14 hours.

- 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 are presented in Section 8.3.1.



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10.4.1 Method 624.1

If the RSD is less than 35%, the RF can be assumed to be invariant and the average RF can be used for calculations. Alternatively, the results can be used to fit a linear or quadratic regression of response ratios vs. concentration ratios. If used, the regression must be weighted inversely proportional to concentration. The coefficient of determination (R^2) of the weighted regression must be greater than 0.920 (this value roughly corresponds to the RSD limit of 35%). Alternatively, the relative standard error may be used as an acceptance criterion. As with the RSD, the RSE must be less than 35%. If an RSE less than 35% cannot be achieved for a quadratic regression, system performance is unacceptable, and the system must be adjusted and re-calibrated.

STATE NOTE: South Carolina does not allow the use of quadratic regression for compounds that have previously demonstrated linearity.

10.5 Run a mid-point Continuing Calibration Verification (CCV) using the primary source standards every 12 hours before sample analysis and every 12 hours during an analytical sequence for 8260B, 8260C, 8260D, 624.1 and 6200B. See sections 8.3.1 – 8.3.6 for acceptance criteria.

10.6 Retention Time Evaluation

10.6.1 Internal standard retention time - The retention times of the internal standards in the calibration verification standard must be evaluated immediately after or during data acquisition. If the retention time for any internal standard changes by more than 10 seconds from that in the mid-point standard level of the most recent initial calibration sequence, then the chromatographic system must be inspected for malfunctions and corrections must be made, as required. When corrections are made, reanalysis of samples analyzed while the system was malfunctioning is required.

10.6.2 Evaluation of retention times - The relative retention time (RRT) of each target analyte in each calibration standard should agree within 0.06 RRT units. Late-eluting target analytes usually have much better agreement.

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 the method detection limit for any of the method target analytes.

10.8.1 If more than one instrument blank or method blanks are analyzed, evaluate and assess the blank and field samples under the same conditions for possible mid-level standard carryover using the subsequent blank after the mid-level standard on a per analyte basis.

10.8.2 Method 624.1 – If any analyte of interest is found in the blank at a concentration greater than the MDL for the analyte, at a concentration greater than one-third the regulatory compliance limit, or at a concentration greater than one-tenth the



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concentration in a sample analyzed during the 12-hour shift, whichever is greater; analysis of samples must be halted and samples affected by the blank must be re-analyzed. If, however, continued re-testing results in repeated blank contamination, the laboratory must document and report the failures (e.g., as qualifiers on results), unless the failures are not required to be reported as determined by the regulatory/control authority. Results associated with blank contamination for an analyte regulated in a discharge cannot be used to demonstrate regulatory compliance.

- 10.9 **LABORATORY CONTROL SAMPLES** - Assess that the LCS was prepared at the required frequency of 1 per batch of 20. If the same injection is used for the LCS and the CCV, ensure that no more than 20 samples are analyzed in conjunction. Routine LCS Control limits are presented in the LIMS.

STATE NOTE: For all samples analyzed from South Carolina, the LCS/LCSD RPD must be <20%, recoveries must be 70-130% in a soil matrix, and recoveries must be within the following limits in a water matrix:

Benzene	70 – 130%
Bromodichloromethane	70 – 130%
Bromoform	70 – 130%
Bromomethane	70 – 130%
Carbon tetrachloride	70 – 130%
Chlorobenzene	70 – 130%
Chloroethane	70 – 130%
2-Chloroethyl vinyl ether	70 – 130%
Chloroform	70 – 130%
Chloromethane	70 – 130%
Dibromochloromethane	70 – 130%
Dichlorodifluoromethane	70 – 130%
1,2-Dichlorobenzene	70 – 130%
1,3-Dichlorobenzene	70 – 130%
1,4-Dichlorobenzene	70 – 130%
1,1-Dichloroethane	70 – 130%
1,2-Dichloroethane	70 – 130%
1,1-Dichloroethene	70 – 130%
Trans-1,2-Dichloroethene	70 – 130%
1,2-Dichloropropane	70 – 130%
Cis-1,3-Dichloropropene	70 – 130%
Trans-1,3-Dichloropropene	70 – 130%
Ethyl benzene	70 – 130%
Methylene chloride	70 – 130%
Methyl tert-butyl ether	70 – 130%
1,1,2,2-Tetrachloroethane	70 – 130%
Tetrachloroethene	70 – 130%
Toluene	70 – 130%
1,1,1-Trichloroethane	70 – 130%
1,1,2-Trichloroethane	70 – 130%
Trichloroethene	71 – 130%
Trichlorofluoromethane	70 – 130%
Vinyl chloride	70 – 130%
Xylenes, total	70 – 130%



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10.9.1 Method 624.1 Requirements

- 10.9.1.1 Compare the percent recovery (Q) for each analyte with its corresponding QC acceptance criterion in the following table. For analytes of interest not listed in the following table, use QC acceptance criteria developed for the LCS. If the recoveries for all analytes of interest fall within their respective QC acceptance criteria, analysis of blanks and field samples may proceed. If any individual Q falls outside the range, proceed according to Section 8.3.5.1.4.

624.1 Purgeable Analytes	Range for Q (%)
Acrolein	60-140
Acrylonitrile	60-140
Benzene	65-135
Bromodichloromethane	65-135
Bromoform	70-130
Bromomethane	15-185
Carbon tetrachloride	70-130
Chlorobenzene	65-135
Chloroethane	40-160
2-Chloroethylvinyl ether	D-225
Chloroform	70-135
Chloromethane	D-205
Dibromochloromethane	70-135
1,2-Dichlorobenzene	65-135
1,3-Dichlorobenzene	70-130
1,4-Dichlorobenzene	65-135
1,1-Dichloroethane	70-130
1,2-Dichloroethane	70-130
1,1-Dichloroethene	50-150
trans-1,2-Dichloroethene	70-130
1,2-Dichloropropane	35-165
cis-1,3-Dichloropropene	25-175
trans-1,3-Dichloropropene	50-150
Ethylbenzene	60-140
Methylene chloride	60-140
1,1,2,2-Tetrachloroethane	60-140
Tetrachloroethene	70-130
Toluene	70-130
1,1,1-Trichloroethane	70-130
1,1,2-Trichloroethane	70-130
Trichloroethene	65-135
Trichlorofluoromethane	50-150
Vinyl chloride	5-195

- 10.9.1.2 Repeat the test only for those analytes that failed to meet the acceptance criteria (Q). If these analytes now pass, system performance is acceptable and analysis of blanks and samples may proceed. Repeated



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failure, however, will confirm a general problem with the measurement system. If this occurs, repeat the test using a fresh LCS or an LCS prepared with a fresh QC check sample concentrate, or perform and document system repair. Subsequent to repair, repeat the calibration verification/LCS test. If the acceptance criteria for Q cannot be met, re-calibrate the instrument. To maintain the validity of the test and re-test, system maintenance and/or adjustment is not permitted between the pair of tests

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 if volume allows.

- 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 applies, 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 found in the LIMS.

Acceptance criteria are that all %Recovery and/or RPD results must be within the indicated control limits on the appropriate MS control charts. See the LIMS for LCS/LCSD & MS/MSD limits and QC acceptance.

STATE NOTE: For all water samples analyzed from South Carolina, the MS/MSD recoveries must be within the most stringent limits comparing in-house derived recovery limits to those given in Table 3 of Method 608. The following are the current water limits:

Benzene	58.6 - 133%
Bromodichloromethane	69.2 – 127%
Bromoform	66.3 – 140%
Bromomethane	16.6 – 183%
Carbon tetrachloride	70 – 139%
Chlorobenzene	70.1 – 130%
Chloroethane	33.3 – 155%
2-Chloroethyl vinyl ether	5 – 149%
Chloroform	66.1 – 133%
Chloromethane	40.7 – 139%
Dibromochloromethane	69.2 – 127%
Dichlorodifluoromethane	42.2 – 146%
1,2-Dichlorobenzene	77.4 – 127%
1,3-Dichlorobenzene	67.9 – 136%
1,4-Dichlorobenzene	74.4 – 123%
1,1-Dichloroethane	64 – 134%
1,2-Dichloroethane	60.7 – 132%
1,1,-Dichloroethene	48.8 – 144%



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Trans-1,2-Dichloroethene	61 – 132%
1,2-Dichloropropane	69.7 – 132%
Cis-1,3-Dichloropropene	71.1 – 129%
Trans-1,3-Dichloropropene	66.3 – 136%
Ethyl benzene	62.7 – 136%
Methylene chloride	61.5 – 125%
Methyl tert-butyl ether	61.4 – 136%
1,1,2,2-Tetrachloroethane	64.9 – 145%
Tetrachloroethene	64 – 141%
Toluene	67.8 – 124%
1,1,1-Trichloroethane	58.7 – 134%
1,1,2-Trichloroethane	74.1 – 130%
Trichloroethene	71 – 148%
Trichlorofluoromethane	39.9 – 165%
Vinyl chloride	44.3 – 143%
Xylenes, total	65.6 – 133%

10.10.1 Method 624.1 Requirements

- 10.10.1.1 Spike at least 5% of the samples in duplicate from each discharge being monitored to assess accuracy (recovery and precision). If direction cannot be obtained from the data user, the laboratory must spike at least one sample in duplicate per extraction batch of up to 20 samples with the analytes in the table in Section 10.10.1.3. Spiked sample results should be reported only to the data user whose sample was spiked, or as requested or required by a regulatory/control authority, or in a permit.
- 10.10.1.2 If the concentration of a specific analyte will be checked against a regulatory concentration limit, the concentration of the spike should be at that limit; otherwise, the concentration of the spike should be one to five times higher than the background concentration, at or near the mid-point of the calibration range, or at the concentration in the LCS whichever concentration would be larger.
- 10.10.1.3 Compare the percent recoveries (P1 and P2) and the RPD for each analyte in the MS/MSD aliquots with the corresponding QC acceptance criteria in the following table. A laboratory may develop and apply QC acceptance criteria more restrictive than the presented criteria if desired.

624.1 Purgeable Analytes	Range for P1, P2 (%)	Limit for RPD
Acrolein	40-160	60
Acrylonitrile	40-160	60
Benzene	37-151	61
Bromodichloromethane	35-155	56
Bromoform	45-169	42
Bromomethane	D-206	61
Carbon tetrachloride	70-140	41
Chlorobenzene	37-160	53

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624.1 Purgeable Analytes	Range for P1, P2 (%)	Limit for RPD
Chloroethane	14-230	78
2-Chloroethylvinyl ether	D-305	71
Chloroform	51-138	54
Chloromethane	D-273	60
Dibromochloromethane	53-149	50
1,2-Dichlorobenzene	18-190	57
1,3-Dichlorobenzene	59-156	43
1,4-Dichlorobenzene	18-190	57
1,1-Dichloroethane	59-155	40
1,2-Dichloroethane	49-155	49
1,1-Dichloroethene	D-234	32
trans-1,2-Dichloroethene	54-156	45
1,2-Dichloropropane	D-210	55
cis-1,3-Dichloropropene	D-227	58
trans-1,3-Dichloropropene	17-183	86
Ethylbenzene	37-162	63
Methylene chloride	D-221	28
1,1,2,2-Tetrachloroethane	46-157	61
Tetrachloroethene	64-148	39
Toluene	47-150	41
1,1,1-Trichloroethane	52-162	36
1,1,2-Trichloroethane	52-150	45
Trichloroethene	70-157	48
Trichlorofluoromethane	17-181	84
Vinyl chloride	D-251	66

10.10.1.4 If any individual P falls outside the designated range for recovery in either aliquot, or the RPD limit is exceeded, the result for the analyte in the unspiked sample is suspect.

10.10.1.5 If in-house QC limits are developed, at least 80% of the analytes tested in the MS/MSD must have in-house QC acceptance criteria that are tighter than those in Section 10.10.1.3 and the remaining analytes (those other than the analytes included in the 80%) must meet the acceptance criteria in Section 10.10.1.3. If an in-house QC limit for the RPD is greater than the limit in Section 10.10.1.3, then the limit in the table must be used. Similarly, if an in-house lower limit for recovery is below the lower limit in Section 10.10.1.3, then the lower limit in the table must be used, and if an in-house upper limit for recovery is above the upper limit in Section 10.10.1.3, then the upper limit in the table must be used.

10.11 SURROGATE EVALUATION: Check the surrogate calculations for correctness for all samples, blanks, ICV/CCV/SSCV, LCS/LCSD, MS and MSD. Acceptance criteria can be found in the LIMS: The surrogate recoveries for all QC samples must be within established control limits.

10.11.1 Method 624.1 – Spike the surrogates into all samples, blanks, LCSs, and MS/MSDs. Compare surrogate recoveries against limits must be developed by



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the laboratory. In-house QC acceptance criteria must be updated at least every two years. If any recovery fails its criteria, attempt to find and correct the cause of the failure.

- 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. Additional 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 CCV. 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 8.3.5

- 10.12.1 Method 624.1 – The responses of each internal standard in each sample, blank, and MS/MSD must be within 50% to 200% (1/2 to 2×) of its respective response in the mid-point calibration standard. If, as a group, all internal standards are not within this range, perform and document system repair, repeat the calibration verification/LCS test, and re-analyze the affected samples. If a single internal standard is not within the 50% to 200% range, use an alternative internal standard for quantitation of the analyte referenced to the affected internal standard. It may be necessary to use the data system to calculate a new response factor from calibration data for the alternative internal standard/analyte pair. If an internal standard fails the 50–200% criteria and no analytes are detected in the sample, ignore the failure or report it if required by the regulatory/control authority.

- 10.13 **SECOND SOURCE:** The second source calibration verification or initial 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 (ICV) 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.

STATE NOTE: For all samples analyzed from South Carolina, the SSCV must recover $\pm 30\%$ for all target analytes.

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 $\pm 40\%$ of the expected concentration. If these criteria are 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.



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- 10.13.1 Method 624.1 Calibration verification/LCS—The working calibration curve or RF must be verified immediately after calibration and at the beginning of each 12-hour shift by the measurement of an LCS. The LCS must be from a source different from the source used for calibration, but may be the same as the sample prepared for the DOC.
- 10.14 The laboratory participates in semi-annual proficiency testing that meets and/or exceeds the requirements of the Quality Control Sample as listed in the published Standard Method SM 6200B-2011, *Volatile Organic Compounds*, and SM 6020-2011, *Quality Assurance/Quality Control*.
- 10.15 For sample analyzed per the requirements of Method 8000D, the LLOQ (see Section 1.8.2) must be verified at least annually, and whenever significant changes are made to the preparation and/or analytical procedure, to demonstrate quantitation capability at lower analyte concentration levels.
 - 10.15.1 The LLOQ verification (to be performed after the initial calibration) is prepared by spiking a clean control material with the analyte(s) of interest at 0.5-2 times the LLOQ concentration level(s).
 - 10.15.2 The LLOQ check is carried through the same preparation and analytical procedures as environmental samples and other QC samples.
 - 10.15.3 It is recommended to analyze the LLOQ verification on every instrument where data is reported; however, at a minimum, the lab must rotate the verification among similar analytical instruments such that all are included within three (3) years.
 - 10.15.4 Recovery of target analytes in the LLOQ verification must be within established in-house limits or within other such project-specific acceptance limits to demonstrate acceptable method performance at the LLOQ. Until the laboratory has sufficient data to determine acceptance limits, the LCS criteria $\pm 20\%$ (i.e., lower limit minus 20% and upper limit plus 20%) may be used for the LLOQ acceptance criteria.
- 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.
 - 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 Tune Check - A successful BFB tune must be achieved prior to initial calibration or daily calibration verification. If a tune does not meet the acceptance criteria detailed above, 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



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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.

- 11.4 **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 and analysis of a second consecutive (immediate) calibration verification fails to produce results within acceptance criteria, corrective actions shall be performed. The laboratory shall demonstrate acceptable performance after the final round of corrective action with two consecutive calibration verifications, or a new initial instrument calibration shall be performed.

Method 8000D: To determine calibration function acceptability, refit the initial calibration data back to the calibration model and calculate %Error (see Section 9.5). Percent error between the calculated and expected amounts of an analyte must be $\leq 30\%$ for all standards. For some data uses, $\leq 50\%$ may be acceptable for the lowest calibration point.

- 11.5 **METHOD BLANK:** 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.

General guidelines for qualifying sample results with regard to method blank quality are as follows:

- If the method blank concentration is less than the MDL and sample results are greater than the RL, then no qualification is required.
- No qualification is necessary when an analyte is detected in the method blank but not in the associated samples.
- If the concentration in a sample is more than ten times the concentration in the method blank, then no qualification is required.
- If the method blank concentration is greater than the MDL but less than the RL and sample results are greater than the MDL, then qualify associated sample results to indicate that analyte was detected in the method blank.
- If the method blank concentration is greater than the RL, further corrective action and qualification is required. An analyst should consult their supervisor for further instruction.

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.

- 11.5.1 **Method 8000D:** When samples that are extracted together are analyzed on separate instruments or in separate analytical shifts, the method blank associated with those samples (e.g., extracted with the samples) must be analyzed on at least one of those instruments. A solvent blank must be analyzed on all other instruments on which the set of samples was analyzed to demonstrate the instrument is not contributing contaminants to the samples. At



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least one method blank or instrument blank must be analyzed on every instrument after calibration standard(s) and prior to the analysis of any samples.

When sample extracts are subjected to cleanup procedures, the associated method blank must also be subjected to the same cleanup procedures.

Results of the method blank should be less than the LLOQ for the analyte or less than the level of acceptable blank contamination specified in the approved QAPP or other appropriate systematic planning document. Blanks are generally considered to be acceptable if target analyte concentrations are less than one-half the LLOQ or are less than project-specific requirements.

When new reagents or chemicals are received, the lab should monitor the blanks associated with samples for any signs of contamination. It is not necessary to test every new batch of reagents or chemicals prior to sample preparation if the source shows no prior problems. However, if reagents are changed during a preparation batch, separate blanks need to be prepared for each set of reagents.

- 11.6 **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.

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.

STATE NOTE: SOUTH CAROLINA DHEC Compliance LCS: responses must be within 70 – 130% for Method 8260 and within the limits given in Attachment VI for Method 624. Qualifiers cannot be used. (If an LCS standard is above the acceptable QC criteria and all samples being reported are below the reporting limit, the data is acceptable based on a high bias with undetectable levels in the field samples. Any positive samples require reanalysis.) Failures require a batch re-analysis. For samples analyzed from South Carolina that are not utilized for compliance purposes, in house established acceptance limits are utilized to demonstrate controlled analyses.

11.7 **LCS/LCSD & MS/MSD CRITERIA**

11.7.1 **Quality control criteria must be checked for the LCS and LCSD.**

LCS samples that do not pass the acceptable QC criteria must be re-analyzed. LCS failures can meet marginal exceedance criteria below. Normal compound list for 8260/624.1 contains typical 90 analytes; therefore only five analyte can be considered as marginal exceedances. 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 exceedance (ME) limits are established by +/- four times the standard deviation. The number of marginal exceedance is based on the number of analytes in the LCS.

Number of allowable marginal exceedances:

>90 analytes, 5 analytes allowed in the ME limit



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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 exceedances must be random events.

STATE NOTE: For South Carolina DHEC compliance samples, marginal exceedances do not apply. All outliers in QC require corrective action when possible and the data must be flagged when necessary.

- 11.7.2 **Method 8000D:** If, as in compliance monitoring, the concentration of a specific analyte in the sample is being checked against a regulatory concentration limit or action level, the spike should be at or below the limit, or 1 - 5 times the background concentration (if historical data are available), whichever concentration is higher. If historical data are not available, a background sample of the same matrix from the site may be submitted for matrix spiking purposes to ensure that high concentrations of target analytes and/or interferences will not prevent calculation of recoveries. If the background sample concentration is very low or non-detect, a spike of greater than five (5) times the background concentration is still acceptable. To assess data precision with duplicate analyses, it is preferable to use a low concentration field sample to prepare a MS/MSD for organic analyses. This spiking procedure will be performed when project-specific instructions are received from the client.

If the concentration of a specific analyte in a sample is not being checked against a limit specific to that analyte, then the analyst may spike the matrix spike or MS/MSD sample(s) at the same concentration as the reference sample at 20 times the estimated LLOQ in the matrix of interest, or at a concentration near the middle of the calibration range. It is suggested that a background sample of the same matrix from the site be submitted as a sample for matrix spiking purposes.

NOTE: Preparing the spiking solution from the same source as the calibration standards helps minimize additional variability due to differences between sources. Typically, spiking concentrations are near the middle of the calibration range.

To develop precision and bias data for the spiked compounds, the analyst has two choices: analyze the original sample, and an MS/MSD pair; or analyze the original sample, a duplicate sample, and one spiked sample. If samples are not expected to contain the target analytes of concern, then the laboratory may use a MS/MSD pair. If samples are expected to contain the target analytes of concern, then the laboratory may use one matrix spike and a duplicate analysis of an unspiked field sample as an alternative to the MS/MSD pair.

The laboratory should use 70 - 130% as interim acceptance criteria for recoveries of spiked analytes, until in-house LCS limits are developed. Where in-house limits have been developed for matrix spike percent recoveries, the LCS results should be similar to or tighter than those limits, as the LCS is prepared in a clean matrix.

- 11.8 **MATRIX SPIKE ASSESSMENT:** If acceptance criteria are not met, perform the following corrective actions as appropriate.

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- If both LCS 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. Reported data must be flagged. Reasonable attempts must be made to address matrix interference.
- If analyst error appears to be the root cause of the failure (i.e., no spiked) reanalysis of the MS/MSD and parent is required if volume permits.

Acceptance criteria are that all RPD results must be within the current control limits found in the LIMS. If these conditions are not met, perform the following corrective actions as appropriate.

- Re-analyze the sample to verify a matrix effect only if analyst error appears to be the root cause.
- If the duplicate precision is still unacceptable, and LCS precision is acceptable, then a potential matrix effect has been identified.

STATE NOTE: South Carolina DHEC compliance analyses require that all target compounds meet the established MS/MSD criteria. No qualifiers can be applied, except in the circumstance where matrix interference is apparent.

PROJECT SPECIFIC CRITERIA (Non-South Carolina Samples): Acceptance criteria are that all %Recovery and/or RPD results meet project-established goals.

- 11.9 **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 surrogate 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 surrogates fail, data is qualified with J1 (outside upper limit) or J2 (outside lower limit).
- 11.10 **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 8.3.5, 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).



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- 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).
- 11.11 **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.
- 11.12 **SECOND SOURCE (SSCV) or Initial Calibration Verification (ICV):** If the SSCV does not meet acceptance criteria, it can be reanalyzed once. If the failure persists, a new initial calibration curve must be prepared and analyzed.
- 11.13 Data that does not meet acceptable QC criteria may be acceptable for use in certain circumstances.
- 11.13.1 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.
- 11.13.1.1 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.
- 11.13.2 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.
- 11.13.3 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”.
- 11.14 Quantitation and manual integration of all QC samples and client samples must follow the procedures outlined in ENV-SOP-MTJL-0024, *Manual Integration Procedure*. “Before” and “After” quantitation reports must be printed in order to verify that any manual integration is performed properly and consistently.
- 11.15 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.
- 11.15.1 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.
- 11.15.2 All tentatively identified compounds (TICs) are reported with a “J” qualifier for estimated value and an “N” for presumptive evidence of material.
- 11.16 For samples analyzed per the requirements of Method 8000D, reported concentrations of target analytes between the MDL and the LLOQ must be qualified as estimated.

12.0 POLLUTION PREVENTION AND WASTE MANAGEMENT



STANDARD OPERATING PROCEDURE

TITLE: ENV-SOP-MTJL-0100 Volatile Organic Compounds by GC/MS
(EPA 8260B, 8260C, 624, 624.1, and SM6200 B)

ISSUER: Pace National – Mt. Juliet, Tennessee

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- 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 *ENV-SOP-MTJL-0051, Waste Management Plan*.
- 12.2 See ENV-SOP-MTJL-0046, *Environmental Sustainability & Pollution Prevention*.
- 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.
 - 13.3 Heated purge may be used for all samples regardless of matrix or method.
- 14.0 REFERENCES
 - 14.1 *Determinative Chromatographic Separations*, SW-846 Method 8000B, Revision 2, December 1996.
 - 14.2 *Determinative Chromatographic Separations*, SW-846 Method 8000C, Revision 3, March 2003.
 - 14.3 *Determinative Chromatographic Separations*, SW-846 Method 8000D, Revision 4, July 2014.
 - 14.4 *Volatile Organic Compounds by Gas Chromatography/Mass Spectrometry (GC/MS)*, SW-846 Method 8260B, Revision 2, December 1996.
 - 14.5 *Purgeables*, 40 CFR 136, EPA Method 624/
 - 14.6 *Volatile Organic Compounds by the Purge and Trap Capillary-Column Gas Chromatographic/Mass Spectrometric Method*, SM 6200B, 20th edition.
 - 14.7 Policy Document, NELAC Standard, Chapter 2: Proficiency Testing Program Standard and the relevant section of NELAC Standard Chapter 5 National Environmental Laboratory Accreditation Conference
 - 14.8 *Volatile Organic Compounds by Gas Chromatography/Mass Spectrometry (GC/MS)*, SW-846 Method 8260C, Revision 3, August 2006.
 - 14.9 *Volatile Organic Compounds by the Purge and Trap Capillary-Column Gas Chromatographic/Mass Spectrometric Method*, SM 6200B-2011.
 - 14.10 *Purgeables by GC/MS*, EPA Method 624.1, Federal Register, Volume 82, Number 165, August 28, 2017.
 - 14.11 40 Code of Federal Regulations §136.6(b)(4)(j).
 - 14.12 *Volatile Organic Compounds by Gas Chromatography/Mass Spectrometry*, SW-846 Method 8260D, Revision 4, June 2018.

**STANDARD OPERATING PROCEDURE**

TITLE: ENV-SOP-MTJL-0100 Volatile Organic Compounds by GC/MS
(EPA 8260B, 8260C, 624, 624.1, and SM6200 B)

ISSUER: Pace National – Mt. Juliet, Tennessee

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Attachment I: Revision History**Current Version (Pace National):**

Date	Description of Revisions
9/19/2019	Update in response to SC DHEC 8/29/2019 technical review letter. Revised Section 8.2.6.3 as well as added State Notes to Sections 8.2.2, 8.2.6.3, 8.2.9, 10.4.1, and 10.13.

Superseded Versions (ESC Lab Sciences SOP#330363):

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
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.
18	12/11/13	Technical and Quality Review and update. Added state notes in sections 1.0, 10.1, 10.3 and sections 10.14 and 14.7; Revised state notes in sections 1.0, 4.2 and sections 5.5, 6.1, 6.4, 7.1, 7.2, 7.5.1, 7.5.2, 7.6, 7.7, 8.2.4, 8.2.7.3, 8.3.1.4, 8.3.2.2, 8.5.1 and 10.5; Revised Attachments II, IV, VIII; Removed state notes pertaining to Ohio VAP throughout and sections 8.2.3.3 and 11.14.1.
19	2/13/15	Technical and Quality Review and update. Revised all state notes regarding South Carolina sample analyses; Revised sections 7.5.1, 7.5.2 and 8.3.3; Revised Attachments VIII.

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(EPA 8260B, 8260C, 624, 624.1, and SM6200 B)

ISSUER: Pace National – Mt. Juliet, Tennessee

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Version	Date	Description of Revisions
20	9/2/2015	Header and signature block re-formatting. Technical and Quality Review and update. Added Section 9.11 and Attachment IX. Revised Sections 10.2.1, 10.6, 10.6.1, 10.6.2, 10.9, 10.10, 10.11, and 11.7. Deleted Sections 10.6.3 and 10.6.4. Revised footnote in Attachment II. Replaced Attachment VIII.
21	10/29/2015	Technical and quality review and update. Revised Sections 8.3.4, 10.8, and 11.4. Added State Note in Sections 8.2.1 and 10.3.
22	11/17/2015	Technical and quality review and update. Revised Sections 1.3.1, 7.1, 8.3.4, 8.5.9, 10.9, 11.5, and 12.2.
23	10/19/2016	Technical and quality review and update. Header and signature block re-formatting. Revised Sections 1.0, 2.3, 2.4, 4.2, 4.3, 5.3.4, 5.5, 6.1, 6.6.4, 6.8, 7.2, 7.5.1.1, 7.5.2.1, 8.2.3.1, 8.2.4.1, 8.2.7.3, 8.2.8.2, 8.5.5, 8.5.6, 8.5.7.1, 8.5.7.3, 8.6.3, 8.7.2.4, 8.7.2.6, 8.7.2.7, 9.1, 9.2, 9.3, 9.5, 10.2.2, 10.9, 10.10, 11.3, 11.13, 14.1, 14.4, 14.5, 14.6, 14.8, 14.9, Attachment V, Attachment VIII, and Attachment IX Table 2. Added Sections 1.3.2, 9.4, 10.15, 10.15.1, 10.15.2, 10.15.3, 10.15.4, 11.4.1, 11.6.2, 11.15, 13.3, 14.2, and 14.3. Deleted Sections 2.4 through 2.17, 8.5.7.4, 8.5.7.5, 9.1, 9.2, 9.5, 9.6, 9.7, and 9.8.
24	11/28/2017	Technical and quality review and update. Changed ESC logo. Revised Sections 1.0, 3.1, 4.3, 7.5, 7.5.1, 7.5.1.1, 7.5.1.2, 7.5.2, 7.5.2.1, 7.5.2.2, 8.2.3.1, 8.2.3.2, 8.2.4.1, 8.3.1.1, 8.3.1.2, 8.3.2.1, 8.3.2.2, 8.3.3, 8.4.1, 8.4.2, 10.2.1, 10.5, 10.9, 10.10, 10.12, 11.5, and Attachment IX Table 5. Added Sections 8.7.2.8 and 10.8.1.
25	1/2/2018	Technical and quality review and update. Added Method 624.1 to title. Revised Sections 2.1, 8.1.3, and 8.2.2. Added Section 4.4 and all subsections, 7.5.2.3, 8.2.9, 8.3.4, 8.3.5.1 and all subsections, 8.8.6.1, 8.8.6.2, 8.8.6.3, 8.8.6.4, 9.5, 10.1.1 and all subsections, 10.3.1 and all subsections, 10.4.1, 10.7.1, 10.8.2, 10.9.1 and all subsections, 10.10.1 and all subsections, 10.11.1, 10.12.1, 10.13.1, 14.10, and 14.11.

Superseded Versions (Pace National):

Date	Description of Revisions
2/9/2019	Technical and quality review and update. Deleted header, footer and signature block. Revised sections 1.0, 1.1, 1.3, 1.3.1, 2.1, 2.2, 3.1, 4.2, 4.4.1, 4.4.5, 5.3, 5.3.4, 5.5, 6.1, 6.2, 6.5, 6.6, 6.6.1, 6.6.4, 6.8, 7.1, 7.5, 7.5.1, 7.5.1.1, 7.5.1.2, 7.5.2, 7.5.2.1, 7.6, 7.6.1, 7.7, 7.7.1, 7.8, 7.9, 8.0, 8.1.3, 8.2.1, 8.2.2, 8.2.3.2, 8.2.5, 8.2.7.2, 8.2.9, 8.3.1, 8.3.1.4, 8.3.2, 8.3.2.2, 8.3.5, 8.5.1, 8.5.7.3, 8.5.8, 8.5.9, 8.6, 8.6.1, 8.7.1, 8.7.1.2, 8.7.2, 8.7.2.2, 8.7.2.5, 8.7.2.6, 8.8.6, 8.8.6.1, 8.9.2, 10.1, 10.1.1.2, 10.2, 10.3, 10.3.1.3, 10.5, 10.9, 10.10, 10.12, 10.13, 10.15.3, 11.4, 11.5, 11.6.1, 11.6.2, 11.7, 11.8, 11.9, 11.11, 11.13, 12.1, 12.2, 14.1, 14.2, 14.3, 14.5 and 14.11. Section 4.3 was split into 4.3 and 4.4, both sections were revised and remaining sections were renumbered as necessary. Added sections 5.4, 6.5.3, 8.2.9, 8.2.9.1, 8.2.9.2, 11.3 and 14.12 and renumbered as necessary. Deleted sections 5.10, 6.6.2, 7.5.2.3, 8.3.4, 8.3.5.1, 8.3.5.1.1, 8.3.5.1.2, 10.3.1.1, 10.3.1.2 and 10.7.1 and renumbered as necessary. Revised Attachments I, II and VI. Added Attachment VIII and renumbered remaining attachments. Revised Attachment IX(X) sections 4.11, 4.18, 4.19



STANDARD OPERATING PROCEDURE

TITLE: ENV-SOP-MTJL-0100 Volatile Organic Compounds by GC/MS
(EPA 8260B, 8260C, 624, 624.1, and SM6200 B)

ISSUER: Pace National – Mt. Juliet, Tennessee

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Date	Description of Revisions
829/2019	Revised in response to ORELAP audit findings. Added corporate header and footer. Revised Sections 1.0, 1.1, 6.2, 7.2, 7.5.1.1, 7.5.1.2, 7.5.2.1, 7.6, 7.7.1, 7.9.3, 8.2.3.2, 8.2.4, 8.2.5, 8.5.8, 8.7.1.4, and 10.11. Added Section 1.4. Deleted Section 8.2.4 and all subsections. Re-numbered sections as appropriate.

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(EPA 8260B, 8260C, 624, 624.1, and SM6200 B)

ISSUER: Pace National – Mt. Juliet, Tennessee

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Attachment II: 8260/624.1/6200B Reporting Limits and Common Analyte List

Compound	Water		Low Soil		High Soil	
	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	0.002	mg/L	0.002	mg/Kg	0.10	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
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

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(EPA 8260B, 8260C, 624, 624.1, and SM6200 B)

ISSUER: Pace National – Mt. Juliet, Tennessee

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Compound	Water		Low Soil		High Soil	
	RL	Units	RL*	Units	RL	Units
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
Propene	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)	0.010	mg/L	0.010	mg/Kg	0.50	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
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

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ISSUER: Pace National – Mt. Juliet, Tennessee

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Compound	Water		Low Soil		High Soil	
	RL	Units	RL*	Units	RL	Units
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
2-Propanol	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 (ENV-SOP-MTJL-0129) Section 8.2.4.1

High Soil – Using 200uL extract from 5g soil sample to 5mL methanol; see Method 5035 (ENV-SOP-MTJL-0129) Sect. 8.3.1.2

+ 1-4, Dioxane has a RL of .002 when run using the SIM mode.

**STANDARD OPERATING PROCEDURE**

TITLE: ENV-SOP-MTJL-0100 Volatile Organic Compounds by GC/MS
(EPA 8260B, 8260C, 624, 624.1, and SM6200 B)

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**Attachment III: Characteristic Masses (m/z) for Purgeable Organic Compounds as printed from
SW-846 Method 8260B Table 5**

Compound	Primary Characteristic	Secondary Characteristic
	Ion	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
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

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Compound	Primary Characteristic	Secondary Characteristic
	Ion	Ion(s)
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
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

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Compound	Primary Characteristic Ion	Secondary Characteristic Ion(s)
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
Tert-butyl formate	59	57, 41
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	
4-Bromofluorobenzene	95	174, 176
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).



STANDARD OPERATING PROCEDURE

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Attachment IV: Potential Compounds to be Analyzed by this Procedure

Acetone	Dicyclopentadiene
Acetonitrile	1,4-Dioxane
Acrolein	Epichlorohydrin
Acrylonitrile	Ethanol
Allyl alcohol	Ethylbenzene
Allyl chloride	Ethylene oxide
Benzene	Ethyl methacrylate
Benzyl chloride	n-Hexane
Bromoacetone	2-Hexanone
Bromochloromethane (I.S.)	2-Hydroxypropionitrile
Bromodichloromethane	Iodomethane
4-Bromofluorobenzene (Surr.)	Isobutylalcohol
Bromoform	Malononitrile
Bromomethane	Methacrylonitrile
2-Butanone	Methylene chloride
Carbon disulfide	Methyl iodide
Carbon tetrachloride	Methyl methacrylate
Chloral hydrate	4-methyl-2-pentanone
Chlorobenzene	Pentachloroethane
Chlorobenzene d-5 (I.S.)	2-Picoline
Chlorodibromomethane	Propargyl alcohol
2-Propanol	Propene
Chloroethane	B-propiolactone
2-Chloroethanol	Propionitrile
bis-(2-Chloroethyl) sulfide	n-Propylamine
2-Chloroethyl vinyl ether	Pyridine
Chloroform	Styrene
Chloromethane	1,1,1,2-Tetrachloroethane
Chloroprene	1,1,2,2-Tetrachloroethane
3-Chloropropionitrile	Tetrachloroethene
1,2-Dibromo-3-chloropropane	Toluene
1,2-Dibromoethane	1,3-Butadiene
Dibromomethane	1,1,1-Trichloroethane
1,4-Dichloro-2-butene	1,1,2-Trichloroethane
dichlorodifluoromethane	Trichloroethene
1,1-Dichloroethane	Trichlorofluoromethane
1,2-Dichloroethane	1,2,3-Trichloropropane
1,2-Dichloroethane d-4 (surr.)	Vinyl acetate
1,1-Dichloroethene	Vinyl chloride
Trans-1,2-dichloroethene	Xylene (total)
Cis-1,2-dichloroethene	1,2,3,4-Diepoxybutane
1,2-dichloropropane	4-Ethyltoluene



STANDARD OPERATING PROCEDURE

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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.

**STANDARD OPERATING PROCEDURE**

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Attachment VI: EPA 624.1 CCC Criteria:

Table 7 – LCS (Q), DOC (s and $\bar{X}$), and MS/MSD (P and RPD) Acceptance Criteria ¹					
Analyte	Range for Q (%)	Limit for s (%)	Range for $\bar{X}$ (%)	Range for P ₁ P ₂ (%)	Limit for RPD
Acrolein	60-140	30	50-150	40-160	60
Acrylonitrile	60-140	30	50-150	40-160	60
Benzene	65-135	33	75-125	37-151	61
Benzene-d ₆					
Bromodichloromethane	65-135	34	50-140	35-155	56
Bromoform	70-130	25	57-156	45-169	42
Bromomethane	15-185	90	D-206	D-242	61
2-Butanone-d ₆					
Carbon tetrachloride	70-130	26	65-125	70-140	41
Chlorobenzene	65-135	29	82-137	37-160	53
Chloroethane	40-160	47	42-202	14-230	78
Chloroethane-d ₃					
2-Chloroethylvinyl ether	D-225	130	D-252	D-305	71
Chloroform	70-135	32	68-121	51-138	54
Chloroform- ¹³ C					
Chloromethane	D-205	472	D-230	D-273	60
Dibromochloromethane	70-135	30	69-133	53-149	50
1,2-Dichlorobenzene	65-135	31	59-174	18-190	57
1,2-Dichlorobenzene-d ₄					
1,3-Dichlorobenzene	70-130	24	75-144	59-156	43
1,4-Dichlorobenzene	65-135	31	59-174	18-190	57
1,1-Dichloroethane	70-130	24	71-143	59-155	40
1,2-Dichloroethane	70-130	29	72-137	49-155	49
1,2-Dichloroethane-d ₄					
1,1-Dichloroethene	50-150	40	19-212	D-234	32
1,1-Dichloroethene-d ₂					
trans-1,2-Dichloroethene	70-130	27	68-143	54-156	45
1,2-Dichloropropane	35-165	69	19-181	D-210	55
1,2-Dichloropropane-d ₆					
cis-1,3-Dichloropropene	25-175	79	5-195	D-227	58
trans-1,3-Dichloropropene	50-150	52	38-162	17-183	86
trans-1,3-Dichloropropene-d ₄					
Ethyl benzene	60-140	34	75-134	37-162	63
2-Hexanone-d ₈					
Methylene chloride	60-140	192	D-205	D-221	28
1,1,2,2-Tetrachloroethane	60-140	36	68-136	46-157	61
1,1,2,2-Tetrachloroethane-d ₂					
Tetrachloroethene	70-130	23	65-133	64-148	39
Toluene	70-130	22	75-134	47-150	41
Toluene-d ₈					
1,1,1-Trichloroethane	70-130	21	69-151	52-162	36
1,1,2-Trichloroethane	70-130	27	75-136	52-150	45
Trichloroethene	65-135	29	75-138	70-157	48
Trichlorofluoromethane	50-150	50	45-158	17-181	84
Vinyl chloride	5-195	100	D-218	D-251	66
Vinyl chloride-d ₃					

¹ Criteria were calculated using an LCS concentration of 20 µg/L

**STANDARD OPERATING PROCEDURE**

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Attachment VII: EPA 8260C Minimum Relative Response Factor Criteria for Initial and Continuing Calibration Verification:

Volatile Compound	Minimum Response Factor (RF)
Dichlorodifluoromethane	0.100
Chloromethane	0.100
Vinyl chloride	0.100
Bromomethane	0.100
Chloroethane	0.100
Trichlorofluoromethane	0.100
1,1-Dichloroethene	0.100
1,1,2-Trichloro-1,2,2-trifluoroethane	0.100
Acetone	0.100
Carbon disulfide	0.100
Methyl Acetate	0.100
Methylene chloride	0.100
trans-1,2-Dichloroethene	0.100
cis-1,2-Dichloroethene	0.100
Methyl tert-Butyl Ether	0.100
1,1-Dichloroethane	0.200
2-Butanone	0.100
Chloroform	0.200
1,1,1-Trichloroethane	0.100
Cyclohexane	0.100
Carbon tetrachloride	0.100
Benzene	0.500
1,2-Dichloroethane	0.100
Trichloroethene	0.200
Methylcyclohexane	0.100

Volatile Compound	Minimum Response Factor (RF)
1,2-Dichloropropane	0.100
Bromodichloromethane	0.200
cis-1,3-Dichloropropene	0.200
trans-1,3-Dichloropropene	0.100
4-Methyl-2-pentanone	0.100
Toluene	0.400
1,1,2-Trichloroethane	0.100
Tetrachloroethene	0.200
2-Hexanone	0.100
Dibromochloromethane	0.100
1,2-Dibromoethane	0.100
Chlorobenzene	0.500
Ethylbenzene	0.100
meta-/para-Xylene	0.100
ortho-Xylene	0.300
Styrene	0.300
Bromoform	0.100
Isopropylbenzene	0.100
1,1,2,2-Tetrachloroethane	0.300
1,3-Dichlorobenzene	0.600
1,4-Dichlorobenzene	0.500
1,2-Dichlorobenzene	0.400
1,2-Dibromo-3-chloropropane	0.050
1,2,4-Triichlorobenzene	0.200

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Attachment VIII: EPA 8260D (Table 4) Guidance Response Factors Criteria From EPA Contract Laboratory Program (Min RF):

Analyte	RF
Acetone	0.01
Benzene	0.2
Bromochloromethane	0.1
Bromodichloromethane	0.3
Bromoform	0.1
Bromomethane	0.01
2-Butanone	0.01
Carbon disulfide	0.1
Carbon tetrachloride	0.1
Chlorobenzene	0.4
Chloroethane	0.01
Chloroform	0.3
Chloromethane	0.01
Cyclohexane	0.01
Dibromochloromethane	0.2
1,2-Dibromo-3-chloropropane	0.01
1,2-Dibromoethane (EDB, Ethylene dibromide)	0.2
1,2-Dichlorobenzene	0.6
1,3-Dichlorobenzene	0.5
1,4-Dichlorobenzene	0.6
Dichlorodifluoromethane	0.01
1,1-Dichloroethane	0.3
1,2-Dichloroethane	0.07
1,1-Dichloroethene (Vinylidene chloride)	0.06
cis-1,2-Dichloroethene	0.2
trans-1,2-Dichloroethene	0.1
1,2-Dichloropropane	0.2
cis-1,3-Dichloropropene	0.3
trans-1,3-Dichloropropene	0.3
Ethylbenzene	0.4
2-Hexanone	0.01
Isopropylbenzene	0.4
Methyl acetate	0.01
4-Methyl-2-pentanone	0.03
Methyl tert-butyl ether (MTBE)	0.1
Methylcyclohexane	0.05
Methylene chloride	0.01
Styrene	0.2
1,1,2,2-Tetrachloroethane	0.2
Tetrachloroethene	0.1
Toluene	0.3
1,2,3-Trichlorobenzene	0.4
1,2,4-Trichlorobenzene	0.4
1,1,1-Trichloroethane	0.05
1,1,2-Trichloroethane	0.2
1,1,2-Trichlorotrifluoroethane	0.05
Trichloroethene (Trichloroethylene)	0.2
Trichlorofluoromethane	0.01
Vinyl chloride	0.01
m,p-Xylene	0.2
o-Xylene	0.2

Values in this table are referenced from the CLP Statement of Work SOM 02.4. These response factors are provided as guidance only and are not intended to be a requirement. See Appendix B for additional information.

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**Attachment IX: Laboratory Control Standard and Matrix Spike
Typically Spiked Compounds**

STANDARD ANALYTE LIST

1,1,1,2-TETRACHLOROETHANE	CHLORODIBROMOMETHANE
1,1,1-TRICHLOROETHANE	CHLOROETHANE
1,1,2,2-TETRACHLOROETHANE	CHLOROFORM
1,1,2-TRICHLOROETHANE	CHLOROMETHANE
1,1,2-TRICHLOROTRIFLUOROETHANE	CIS-1,2-DICHLOROETHENE
1,1-DICHLOROETHANE	CIS-1,3-DICHLOROPROPENE
1,1-DICHLOROETHENE	DIBROMOMETHANE
1,1-DICHLOROPROPENE	DICHLORODIFLUOROMETHANE
1,2,3-TRICHLOROBENZENE	DICHLOROFLUOROMETHANE
1,2,3-TRICHLOROPROPANE	DICYCLOPENTADIENE
1,2,3-TRIMETHYLBENZENE	DI-ISOPROPYL ETHER
1,2,4-TRICHLOROBENZENE	ETHYL ETHER
1,2,4-TRIMETHYLBENZENE	ETHYLBENZENE
1,2-DIBROMO-3-CHLOROPROPANE	HEXACHLORO-1,3-BUTADIENE
1,2-DIBROMOETHANE	IODOMETHANE
1,2-DICHLOROBENZENE	ISOPROPYLBENZENE
1,2-DICHLOROETHANE	M&P-XYLENE
1,2-DICHLOROPROPANE	METHYL TERT-BUTYL ETHER
1,3,5-TRIMETHYLBENZENE	METHYLENE CHLORIDE
1,3-BUTADIENE	NAPHTHALENE
1,3-DICHLOROBENZENE	N-BUTYLBENZENE
1,3-DICHLOROPROPANE	N-HEXANE
1,4-DICHLOROBENZENE	N-PROPYLBENZENE
1-METHYLNAPHTHALENE	O-XYLENE
2,2,4-TRIMETHYLPENTANE	P-ISOPROPYLTOLUENE
2,2-DICHLOROPROPANE	PROPENE
2-BUTANONE (MEK)	SEC-BUTYLBENZENE
2-CHLOROETHYL VINYL ETHER	STYRENE
2-CHLOROTOLUENE	TERT-BUTYLBENZENE
2-HEXANONE	TETRACHLOROETHENE
2-METHYLNAPHTHALENE	TETRAHYDROFURAN
4-CHLOROTOLUENE	TOLUENE
4-ETHYLTOLUENE	TPH (GC/MS) LOW FRACTION
4-METHYL-2-PENTANONE (MIBK)	TRANS-1,2-DICHLOROETHENE
ACETONE	TRANS-1,3-DICHLOROPROPENE
ACROLEIN*	TRANS-1,4-DICHLORO-2-BUTENE

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STANDARD ANALYTE LIST

ACRYLONITRILE*

BENZENE

BROMOBENZENE

BROMOCHLOROMETHANE

BROMODICHLOROMETHANE

BROMOFORM

BROMOMETHANE

CARBON DISULFIDE

CARBON TETRACHLORIDE

CHLOROBENZENE

TRICHLOROETHENE

TRICHLOROFLUOROMETHANE

VINYL ACETATE

VINYL CHLORIDE

XYLENES, TOTAL

SURROGATE LIMITS

4-BROMOFLUOROBENZENE

A,A,A-TRIFLUOROTOLUENE

DIBROMOFLUOROMETHANE

TOLUENE-D8



STANDARD OPERATING PROCEDURE

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Attachment X: DoD Requirements

1.0 Equipment/Instrument Maintenance

Instrument maintenance must be performed routinely to optimize instrument performance and improve chromatography. Commonly performed maintenance includes baking traps and columns, changing injection port liners, changing pump oil, etc. A new calibration curve must be analyzed following any major maintenance performed on the analytical system.

2.0 Computer Hardware and Software

Software name and version: HP Chemstation G1701CA Version C.00.00 or equivalent

3.0 Troubleshooting

Table 1. GCMS Troubleshooting Guide		
Problem	Cause	Treatment
Peaks broaden and tail	Poor column installation causing dead volume in the injector	Reinstall column in injector. Check seal at ferrule. Check insertion depth. Ensure a good column cut.
	Solvent flashing in hot injector	Reduce injection speed on hot injectors and if possible reduce injector temperature
	Injector not being purged properly after splitless injection	For splitless injection, the vent flow should be 70 ml/min, and the injector should be switched to the split mode 0.5_1.5 min after injection.
Tailing sample peaks for active components	Active sites in the injector insert or liner	Change or clean the injector insert
	Active sites or degraded phase in column	Remove the front 15 cm of the column and reinstall. If retention times are changing or cutting the column does not help, replace the column.
	Injector not hot enough for higher boiling compounds	Increase the injector temperature and lower the injection speed. Check that the graphite ferrule is free of cracks and the septum support is tight.
Low response and tailing of high boiling point compounds	Injector is not hot enough to vaporize high boilers	Increase injector temperature
	Interface/ion source not getting to adequate temperature	Change the manifold heater
Leading sample peaks	Column overload due to excess amount of component injected	Dilute the sample or do split injection
	Degradation of stationary phase	Change the column
	Carrier gas velocity too low	Increase carrier gas flow rate
Poor chromatographic resolution	Column temperature or program not optimized	Modify method by changing temperature ramp segment slopes

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Table 1. GCMS Troubleshooting Guide

Problem	Cause	Treatment
	Carrier gas flow rate not optimized	Decrease carrier gas linear velocity
	Stationary phase has degraded	Replace the column
Peak splitting, especially low boilers	Sample is flashing in the injector simulating two injections	Lower injector temperature
Retention times shift in chromatogram	Unstable carrier gas flow controller/regulator	Check pneumatics for leaks. Replace flow controller/ regulator if necessary.
	Column contamination or degradation	Condition or replace column
	Leaks at septum or column to injector connection	Replace septum regularly and check that the septum nut and the capillary column nut are tight
Cannot reach operating vacuum	Analyzer contaminated by diffusion pump oil	Shut down and clean mass spec
	Major air leak around column fitting into interface	Replace column ferrule and reseal compression fitting
No tune peaks	Cal gas valve not open	Open cal gas valve
	PFTBA solenoid valve stuck open. All PFTBA has evaporated.	Have solenoid replaced. Put fresh PFTBA in the cal gas vial.
Analysis sensitivity has decreased	Background has increased	Check column bleed, septum bleed, pump oil, and ion source contamination
	Detector needs replacement	Replace detector
	Defective syringe	Try a new or proven syringe
	“Blown” septum or other massive leaks at the inlet or with carrier gas flow. Poor peak shapes usually result from bad leaks.	Find and fix leaks and adjust gas flow.
	Purge flow or split ratio too high	Adjust gas flow rates

4.0 Other Requirements

- 4.1 All hardcopy laboratory notebooks must be reviewed by the Supervisor, or their designee, on a monthly basis.
- 4.2 If not self-explanatory (e.g., a typo or transposed number), corrections to technical and quality records shall also include a justification for the change.
- 4.3 A storage blank must be stored with all volatile organic samples, regardless of suspected concentration levels.
- 4.4 A person performing a manual integration must sign and date each manually integrated chromatogram and record the rationale for performing manual integration. Electronic signatures are acceptable.

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- 4.5 The results of calibration and verification of support equipment must be within the specifications required of the application for which this equipment is used or the equipment must be removed from service until repaired. Calibration and verification records, including those of established correction factors, must be maintained. In the absence of method-specific requirements, the minimum requirements are as follows:

Table 2. Support Equipment Checks

Performance Check	Frequency	Acceptance Criteria
Balance calibration check [Using two standard weights that bracket the expected mass]	Daily prior to use	Top-loading balance: $\pm 2\%$ or $\pm 0.02\text{g}$, whichever is greater Analytical balance: $\pm 0.1\%$ or $\pm 0.5\text{mg}$, whichever is greater
Verification of standard mass [Using weights traceable to the International System of Units (SI) through a NMI]	Every 5 years	Certificate of Calibration from ISO/IEC 17025 accredited calibration laboratory
Monitoring of refrigerator/freezer temperatures	Daily (i.e. 7 days per week) [use MIN/MAX thermometers or data loggers equipped with notification of out of control event capabilities if personnel not available to record daily]	Refrigerators: 0°C to 6°C Freezers: $\leq -10^{\circ}\text{C}$
Thermometer verification check [Using a thermometer traceable to the SI through an NMI] [Performed at two temperatures that bracket the target temperature(s). Assume linearity between the two bracketing temperatures.] [If only a single temperature is used, at the temperature of use]	Liquid in glass: Before first use and annually Electronic: Before first use and quarterly	Apply correction factors or replace thermometer
Volumetric labware	Class B: By lot before first use Class A and B: Upon evidence of deterioration	Bias: Mean within $\pm 2\%$ of nominal volume Precision: $\text{RSD} \leq 1\%$ of nominal volume (based on 10 replicate measurements)
Non-volumetric labware [Applicable only when used for measuring initial sample volume and final extract/ digestates volume]	By lot before first use or upon evidence of deterioration	Bias: Mean within $\pm 3\%$ of nominal volume Precision: $\text{RSD} \leq 3\%$ of nominal volume (based on 10 replicate measurements)
Mechanical volumetric pipette	Quarterly	Bias: Mean within $\pm 2\%$ of nominal volume Precision: $\text{RSD} \leq 1\%$ of nominal volume (based on minimum of 3 replicate measurements) [Note: for variable volume pipettes, the nominal volume is the volume of use]

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Table 2. Support Equipment Checks

Performance Check	Frequency	Acceptance Criteria
Glass microliter syringe	Upon receipt and upon evidence of deterioration	General Certificate of Bias & Precision upon receipt Replace if deterioration is evident
Drying oven temperature check	Daily prior to and after use	Within $\pm 5\%$ of set temperature
Water purification system	Daily prior to use	See method blank criteria given in Section 4.20 of this addendum

- 4.6 The expiration date of the prepared standard shall not exceed the expiration date of the primary standard. All containers must bear a preparation date.
- 4.7 To avoid preparing non-representative samples, the laboratory shall not “target” within a relatively small mass range (e.g., $1.00 \pm 0.01\text{g}$) because such targeting will produce non-representative subsamples if the sample has high heterogeneity. The laboratory shall not manipulate the sample material so the sample aliquot weighs exactly $1.00\text{g} \pm 0.01\text{g}$, as an example.
- 4.8 In the absence of project-specific requirements, the minimum standard data qualifiers to be used are:
- U Analyte was not detected and is reported as less than the LOD or as defined by the customer. The LOD has been adjusted for any dilution or concentration of the sample.
 - J The reported result is an estimated value (e.g., matrix interference was observed or the analyte was detected at a concentration outside the quantitation range).
 - B Blank contamination. The recorded result is associated with a contaminated blank.
 - N Non-target analyte. The analyte is a tentatively identified compound using mass spectrometry or any non-customer requested compounds that are tentatively identified.
 - Q One or more quality control criteria failed (e.g., LCS recovery, surrogate spike recovery, or CCV recovery).
- Additional data qualifiers may be used, or different letters or symbols to denote the qualifiers listed above, as long as they are appropriately defined and their use is consistent with project-specific requirements (e.g., QSM 5.0, the contract, and project-planning documents).
- 4.9 If the time of the sample collection is not provided, assume the most conservative time of day. For the purpose of batch processing, the start and stop dates and times of the batch preparation shall be recorded.
- 4.10 Each preparation method listed on the scope of accreditation must have quarterly LOD/LOQ verifications. However, not all possible combinations of preparation and cleanup techniques are required to have LOD/LOQ verifications. If LOD/LOQ verifications are not performed on all combinations, the laboratory must base the LOD/LOQ verifications on the worst case basis (preparation method with all applicable cleanup steps).



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- 4.11 After each MDL determination, the laboratory must establish the LOD by spiking a quality system matrix at a concentration of at least two (2) times but no greater than four times the MDL. This spike concentration establishes the LOD and the concentration at which the LOD shall be verified. It is specific to each suite of analyte, matrix, and method (including sample preparation). The following requirements apply to the initial LOD establishment and to the LOD verifications:
- The apparent signal to noise (S/N) ratio at the LOD must be at least three and the results must meet all method requirements for analyte identification (e.g., ion abundance, second column confirmation, or pattern recognition). For data systems that do not provide a measure of noise, the signal produced by the verification sample must produce a result that is at least three standard deviations greater than the mean method blank concentration. This is initially estimated based on a minimum of four method blank analyses and later established with a minimum of 20 method blank results.
 - If the LOD verification fails, then the laboratory must repeat the MDL determination and LOD verification or perform and pass two consecutive LOD verifications at a higher spike concentration and set the LOD at the higher concentration.
 - The laboratory shall maintain documentation for all MDL determinations and LOD verifications.
 - The DL and LOD must be reported for all analyte-matrix-methods suites, unless it is not applicable to the test or specifically excluded by project requirements.
- 4.12 The LOD shall be verified quarterly. In situations where methods are setup and used on an infrequent basis, the laboratory may choose to perform LOD verifications on a one per batch basis. All verification data will be in compliance, reported, and available for review.
- 4.13 For DoD, at a minimum, the LOQ shall be verified quarterly. In situations where methods are setup and used on an infrequent basis, the laboratory may choose to perform LOQ verifications on a one per batch basis.
- 4.14 All initial instrument calibrations must be verified with a standard obtained from a second manufacturer prior to analyzing any samples. The use of a standard from a second lot obtained from the same manufacturer (independently prepared from different source materials) is acceptable for use as a second source standard. The concentration of the second source standard shall be at or near the midpoint of the calibration range. The acceptance criteria for the initial calibration verification must be at least as stringent as those for the continuing calibration verification.
- 4.15 Exclusion of calibration points without documented scientifically valid technical justification is not permitted.
- 4.16 The concentration of the CCV standard shall be greater than the low calibration standard and less than or equal to the midpoint of the calibration range.
- 4.17 All CCVs analyzed must be evaluated and reported. If a CCV fails, reanalysis or corrective actions must be taken.
- If a CCV fails, the laboratory can immediately analyze two additional consecutive CCVs (immediately is defined as starting a consecutive pair within one hour; no samples can be run between the failed CCV and the two additional CCVs). This approach allows for spurious failures of analytes to be reported without reanalysis of samples. Any corrective actions that change the dynamics of the system (e.g., clip



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- column, clean injection port, run blanks) requires that all samples since the last acceptable CCV be reanalyzed.
- Both of these CCVs must meet acceptance criteria in order for the samples to be reported without reanalysis.
 - If either of these two CCVs fail or if the laboratory cannot immediately analyze two CCVs, the associated samples cannot be reported and must be reanalyzed.
 - Corrective action(s) and recalibration must occur if the above scenario fails. All affected samples since the last acceptable CCV must be reanalyzed.
 - Flagging of data for a failed CCV is only appropriate when the affected samples cannot be reanalyzed. The laboratory must notify the client prior to reporting data associated with a failed CCV.
- 4.18 The results of all MS/MSDs must be evaluated using the same acceptance criteria used for the DoD LCS limits (see Tables 3 and 4) or project limits, if specified. If the specific analyte(s) are not available in the Tables 3 and 4, the laboratory shall use their LCS in-house limits (see the LIMS) as a means of evaluating MS/MSDs. The MS and MSD must be spiked with all reported analytes.
- 4.19 Surrogate spike results shall be compared with DoD LCS limits (see Tables 3 and 4) or acceptance criteria specified by the client. If these criteria are not available, the laboratory shall compare the results with its in-house statistically established LCS criteria (see the LIMS).
- 4.20 The method blank shall be considered to be contaminated if:
- The concentration of any target analyte (chemical of concern) in the blank exceeds 1/2 the LOQ and is greater than 1/10th the amount measured in any associated sample, or 1/10th the regulatory limit, whichever is greater;
 - The concentration of any common laboratory contaminant in the blank exceeds the LOQ;
 - If a method blank is contaminated as described above, then the laboratory shall reprocess affected samples in a subsequent preparation batch, except when sample results are below the LOD. If insufficient sample volume remains for reprocessing, the results shall be reported with appropriate data qualifiers.
- 4.21 Sporadic Marginal Exceedances are not allowed for target analytes (chemicals of concern as identified by a project) without project-specific approval. Target analytes are considered those few analytes that are critical for the success of a project (such as risk drivers) where sporadic marginal exceedances cannot be allowed. Laboratories should consult with clients whenever long lists of analytes are requested for analysis to determine if marginal exceedances will not be allowed.
- 4.22 DoD considers the same analyte exceeding the LCS control limit two (2) out of three (3) consecutive LCS to be indicative of non-random behavior, which requires corrective action and reanalysis of the LCS.

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Table 3. LCS Control Limits – Method 8260 Solid Matrix

CAS ID	Analyte	N Records	Mean	Standard Deviation	Lower Control Limit	Upper Control Limit
630-20-6	1,1,1,2-Tetrachloroethane	11115	101.1	7.8	78	125
71-55-6	1,1,1-Trichloroethane	12156	101.6	9.4	73	130
79-34-5	1,1,2,2-Tetrachloroethane	11670	97	8.9	70	124
79-00-5	1,1,2-Trichloroethane	11772	99.7	7.2	78	121
76-13-1	1,1,2-Trifluoro-1,2,2-trichloroethane [Freon-113]	9760	100.8	11.7	66	136
75-34-3	1,1-Dichloroethane	11856	100.4	8.1	76	125
75-35-4	1,1-Dichloroethene	12352	100.3	10.1	70	131
563-58-6	1,1-Dichloropropene	10793	100.5	8.3	76	125
87-61-6	1,2,3-Trichlorobenzene	10572	97.8	10.6	66	130
96-18-4	1,2,3-Trichloropropane	10925	99.1	8.8	73	125
526-73-8	1,2,3-Trimethylbenzene	1948	99.8	6	82	118
120-82-1	1,2,4-Trichlorobenzene	10980	98	10.4	67	129
95-63-6	1,2,4-Trimethylbenzene	11085	98.7	7.9	75	123
96-12-8	1,2-Dibromo-3-chloropropane	11380	96.6	11.7	61	132
106-93-4	1,2-Dibromoethane	11408	100.1	7.3	78	122
95-50-1	1,2-Dichlorobenzene	11785	99.1	7.2	78	121
107-06-2	1,2-Dichloroethane	12328	100.5	9.2	73	128
17060-07-0	1,2-Dichloroethane-d4	5951	103.1	10.8	71	136
540-59-0	1,2-Dichloroethene	7748	99.9	7.3	78	122
78-87-5	1,2-Dichloropropane	12145	99.5	7.8	76	123
354-23-4	1,2-Dichlorotrifluoroethane [Freon 123a]	1269	97.8	11.3	64	132
108-70-3	1,3,5-Trichlorobenzene	4723	99.4	9.6	71	128
108-67-8	1,3,5-Trimethylbenzene	11080	98.4	8.4	73	124
541-73-1	1,3-Dichlorobenzene	11619	98.9	7.4	77	121
142-28-9	1,3-Dichloropropane	10713	99.1	7.3	77	121
542-75-6	1,3-Dichloropropene	3714	101.6	8.1	77	126
106-46-7	1,4-Dichlorobenzene	11848	97.5	7.6	75	120
105-05-5	1,4-Diethylbenzene	1896	96.6	5.9	79	114
123-91-1	1,4-Dioxane	7698	96.4	13.7	55	138
544-10-5	1-Chlorohexane	2543	100.4	9.8	71	130
594-20-7	2,2-Dichloropropane	10703	99.7	11.1	67	133
78-93-3	2-Butanone [MEK]	11514	99.6	16.3	51	148
126-99-8	2-Chloro-1,3-butadiene	6667	99	11.3	65	133
110-75-8	2-Chloroethyl vinyl ether	6957	96.1	17.6	43	149
95-49-8	2-Chlorotoluene	10838	98.5	7.9	75	122
591-78-6	2-Hexanone	11004	99.1	15.4	53	145
79-46-9	2-Nitropropane	4969	98.3	17.1	47	150
67-63-0	2-Propanol [Isopropyl alcohol]	1696	99.8	13.4	60	140
460-00-4	4-Bromofluorobenzene	6267	98.9	6.8	79	119
106-43-4	4-Chlorotoluene	10785	98.3	8.6	72	124
108-10-1	4-Methyl-2-pentanone [MIBK]	11364	99.6	11.6	65	135
67-64-1	Acetone	11089	99.6	21.4	36	164
75-05-8	Acetonitrile	5697	98.5	14.8	54	143
107-02-8	Acrolein [Propenal]	7528	101.1	18	47	155

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Table 3. LCS Control Limits – Method 8260 Solid Matrix

CAS ID	Analyte	N Records	Mean	Standard Deviation	Lower Control Limit	Upper Control Limit
107-13-1	Acrylonitrile	8293	99.7	11.4	65	134
107-05-1	Allyl chloride	6908	101.1	11.2	68	135
71-43-2	Benzene	12853	99.2	7.4	77	121
100-44-7	Benzyl chloride	2743	92.1	9.4	64	120
108-86-1	Bromobenzene	10974	99.3	7.3	78	121
74-97-5	Bromochloromethane	11023	101.4	7.8	78	125
75-27-4	Bromodichloromethane	11850	101	8.5	75	127
75-25-2	Bromoform	11890	99.1	10.8	67	132
74-83-9	Bromomethane	11416	98.3	15	53	143
75-15-0	Carbon disulfide	11132	97.9	11.5	63	132
56-23-5	Carbon tetrachloride	12090	102.3	10.7	70	135
108-90-7	Chlorobenzene	12382	99.7	6.9	79	120
124-48-1	Chlorodibromomethane	11852	100.2	8.7	74	126
75-00-3	Chloroethane	11444	98.8	13.3	59	139
67-66-3	Chloroform	12344	100.3	7.6	78	123
74-87-3	Chloromethane	11876	93.3	14.3	50	136
156-59-2	cis-1,2-Dichloroethene	11645	99.9	7.6	77	123
10061-01-5	cis-1,3-Dichloropropene	11805	99.8	8.7	74	126
1476-11-5	cis-1,4-Dichloro-2-butene	977	106	12.4	69	143
110-82-7	Cyclohexane	8827	98.9	10.6	67	131
108-94-1	Cyclohexanone	3764	93.2	20.9	30	156
1868-53-7	Dibromofluoromethane	2142	98.1	6.8	78	119
74-95-3	Dibromomethane	10913	101.1	7.9	78	125
75-71-8	Dichlorodifluoromethane [Freon-12]	11467	88.9	20.1	29	149
75-05-8	Acetonitrile	5697	98.5	14.8	54	143
107-02-8	Acrolein [Propenal]	7528	101.1	18	47	155
107-13-1	Acrylonitrile	8293	99.7	11.4	65	134
107-05-1	Allyl chloride	6908	101.1	11.2	68	135
71-43-2	Benzene	12853	99.2	7.4	77	121
100-44-7	Benzyl chloride	2743	92.1	9.4	64	120
108-86-1	Bromobenzene	10974	99.3	7.3	78	121
74-97-5	Bromochloromethane	11023	101.4	7.8	78	125
75-27-4	Bromodichloromethane	11850	101	8.5	75	127
75-25-2	Bromoform	11890	99.1	10.8	67	132
74-83-9	Bromomethane	11416	98.3	15	53	143
75-15-0	Carbon disulfide	11132	97.9	11.5	63	132
56-23-5	Carbon tetrachloride	12090	102.3	10.7	70	135
108-90-7	Chlorobenzene	12382	99.7	6.9	79	120
124-48-1	Chlorodibromomethane	11852	100.2	8.7	74	126
75-00-3	Chloroethane	11444	98.8	13.3	59	139
67-66-3	Chloroform	12344	100.3	7.6	78	123
74-87-3	Chloromethane	11876	93.3	14.3	50	136
156-59-2	cis-1,2-Dichloroethene	11645	99.9	7.6	77	123
10061-01-5	cis-1,3-Dichloropropene	11805	99.8	8.7	74	126
1476-11-5	cis-1,4-Dichloro-2-butene	977	106	12.4	69	143

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Table 3. LCS Control Limits – Method 8260 Solid Matrix

CAS ID	Analyte	N Records	Mean	Standard Deviation	Lower Control Limit	Upper Control Limit
110-82-7	Cyclohexane	8827	98.9	10.6	67	131
108-94-1	Cyclohexanone	3764	93.2	20.9	30	156
1868-53-7	Dibromofluoromethane	2142	98.1	6.8	78	119
74-95-3	Dibromomethane	10913	101.1	7.9	78	125
75-71-8	Dichlorodifluoromethane [Freon-12]	11467	88.9	20.1	29	149
75-43-4	Dichlorofluoromethane	717	100.8	18	47	155
60-29-7	Diethyl ether	6283	99.6	9.6	71	129
108-20-3	Diisopropyl ether	8542	98.3	9.7	69	127
64-17-5	Ethanol	3958	102.2	18.9	45	159
141-78-6	Ethyl acetate	4516	95.4	14.5	52	139
97-63-2	Ethyl methacrylate	7075	98.9	9.9	69	129
637-92-3	Ethyl tert-butyl ether	7514	98.9	9.1	72	126
100-41-4	Ethylbenzene	12427	99.1	7.7	76	122
462-06-6	Fluorobenzene	689	97.3	5.4	81	114
142-82-5	Heptane	5420	93.4	14.9	49	138
87-68-3	Hexachlorobutadiene	10264	98.1	12.4	61	135
67-72-1	Hexachloroethane	3265	102.5	10.1	72	133
110-54-3	Hexane	7116	93.6	16.1	45	142
74-88-4	Iodomethane	9457	100.9	10.1	71	131
78-83-1	Isobutyl alcohol	6162	97.5	12.6	60	135
108-21-4	Isopropyl acetate [Acetic acid]	2885	94.2	12.2	58	131
98-82-8	Isopropylbenzene	11596	100.8	11.1	68	134
179601-23-1	m/p-Xylene [3/4-Xylene]	10612	100.4	7.7	77	124
126-98-7	Methacrylonitrile	6736	99.2	11.1	66	132
79-20-9	Methyl acetate	8320	98.7	15.2	53	144
80-62-6	Methyl methacrylate	7050	98.4	11.9	63	134
1634-04-4	Methyl tert-butyl ether [MTBE]	11253	98.9	8.7	73	125
108-87-2	Methylcyclohexane	8565	99.4	11.2	66	133
75-09-2	Methylene chloride	12024	98.9	9.7	70	128
123-86-4	n-Butyl acetate	2981	95.1	11	62	128
71-36-3	n-Butyl alcohol	4800	92.9	12.6	55	131
104-51-8	n-Butylbenzene	10921	98.7	9.7	70	128
103-65-1	n-Propylbenzene	10947	98.9	8.8	73	125
91-20-3	Naphthalene	10602	95.6	11.2	62	129
95-47-6	o-Xylene	11940	100	7.7	77	123
99-87-6	p-Isopropyltoluene [p-Cymene]	10953	100.3	9	73	127
76-01-7	Pentachloroethane	5957	102	11.1	69	135
107-12-0	Propionitrile [Ethyl cyanide]	6734	101	11.1	68	134
135-98-8	sec-Butylbenzene	10960	99	8.8	73	126
100-42-5	Styrene	11809	100.2	8	76	124
994-05-8	tert-Amyl methyl ether [TAME]	7153	99.8	8.9	73	126
75-65-0	tert-Butyl alcohol	7492	100.5	10.7	68	133
98-06-6	tert-Butylbenzene	10974	98.8	8.6	73	125

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**STANDARD OPERATING PROCEDURE**

TITLE: ENV-SOP-MTJL-0100 Volatile Organic Compounds by GC/MS
(EPA 8260B, 8260C, 624, 624.1, and SM6200 B)

ISSUER: Pace National – Mt. Juliet, Tennessee

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Table 3. LCS Control Limits – Method 8260 Solid Matrix

CAS ID	Analyte	N Records	Mean	Standard Deviation	Lower Control Limit	Upper Control Limit
127-18-4	Tetrachloroethene	12091	100.5	9.2	73	128
109-99-9	Tetrahydrofuran	8039	98	12.4	61	135
108-88-3	Toluene	12499	99.3	7.3	77	121
2037-26-5	Toluene-d8	6232	100.7	5.2	85	116
156-60-5	trans-1,2-Dichloroethene	11849	99.2	8.6	74	125
10061-02-6	trans-1,3-Dichloropropene	11805	100.9	9.8	71	130
110-57-6	trans-1,4-Dichloro-2-butene	8307	98.6	12.3	62	136
79-01-6	Trichloroethene	12440	100.2	7.6	77	123
75-69-4	Trichlorofluoromethane [Freon-11]	11530	101	13.1	62	140
108-05-4	Vinyl acetate	7260	100.3	16.9	50	151
75-01-4	Vinyl chloride	12129	95.6	13.2	56	135
1330-20-7	Xylenes [total]	8623	100.7	7.7	78	124
104-51-8	n-Butylbenzene	10921	98.7	9.7	70	128
103-65-1	n-Propylbenzene	10947	98.9	8.8	73	125
91-20-3	Naphthalene	10602	95.6	11.2	62	129
95-47-6	o-Xylene	11940	100	7.7	77	123
99-87-6	p-Isopropyltoluene [p-Cymene]	10953	100.3	9	73	127
76-01-7	Pentachloroethane	5957	102	11.1	69	135
107-12-0	Propionitrile [Ethyl cyanide]	6734	101	11.1	68	134
135-98-8	sec-Butylbenzene	10960	99	8.8	73	126
100-42-5	Styrene	11809	100.2	8	76	124
994-05-8	tert-Amyl methyl ether [TAME]	7153	99.8	8.9	73	126
75-65-0	tert-Butyl alcohol	7492	100.5	10.7	68	133
98-06-6	tert-Butylbenzene	10974	98.8	8.6	73	125
127-18-4	Tetrachloroethene	12091	100.5	9.2	73	128
109-99-9	Tetrahydrofuran	8039	98	12.4	61	135
108-88-3	Toluene	12499	99.3	7.3	77	121
2037-26-5	Toluene-d8	6232	100.7	5.2	85	116
156-60-5	trans-1,2-Dichloroethene	11849	99.2	8.6	74	125
10061-02-6	trans-1,3-Dichloropropene	11805	100.9	9.8	71	130
110-57-6	trans-1,4-Dichloro-2-butene	8307	98.6	12.3	62	136
79-01-6	Trichloroethene	12440	100.2	7.6	77	123
75-69-4	Trichlorofluoromethane [Freon-11]	11530	101	13.1	62	140
108-05-4	Vinyl acetate	7260	100.3	16.9	50	151
75-01-4	Vinyl chloride	12129	95.6	13.2	56	135
1330-20-7	Xylenes [total]	8623	100.7	7.7	78	124

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**STANDARD OPERATING PROCEDURE**

TITLE: ENV-SOP-MTJL-0100 Volatile Organic Compounds by GC/MS
(EPA 8260B, 8260C, 624, 624.1, and SM6200 B)

ISSUER: Pace National – Mt. Juliet, Tennessee

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Table 4. LCS Control Limits – Method 8260 Water Matrix

CAS ID	Analyte	N Records	Mean	Standard Deviation	Lower Control Limit	Upper Control Limit
630-20-6	1,1,1,2-Tetrachloroethane	24511	101.1	7.6	78	124
71-55-6	1,1,1-Trichloroethane	28223	102.7	9.6	74	131
79-34-5	1,1,2,2-Tetrachloroethane	27450	96.4	8.3	71	121
79-00-5	1,1,2-Trichloroethane	27338	99.5	6.5	80	119
76-13-1	1,1,2-Trifluoro-1,2,2-trichloroethane [Freon-113]	21122	103	11.1	70	136
75-34-3	1,1-Dichloroethane	28154	101.3	8	77	125
75-35-4	1,1-Dichloroethene	29436	101	10	71	131
563-58-6	1,1-Dichloropropene	23631	102	7.8	79	125
87-61-6	1,2,3-Trichlorobenzene	24271	98.7	10.1	69	129
96-18-4	1,2,3-Trichloropropane	24525	97.5	8	73	122
526-73-8	1,2,3-Trimethylbenzene	2965	100.9	6.2	82	120
120-82-1	1,2,4-Trichlorobenzene	25290	99.8	10.1	69	130
95-63-6	1,2,4-Trimethylbenzene	27917	99.6	8	76	124
96-12-8	1,2-Dibromo-3-chloropropane	24955	94.9	11.1	62	128
106-93-4	1,2-Dibromoethane	29096	99	7.2	77	121
95-50-1	1,2-Dichlorobenzene	27583	99.4	6.5	80	119
107-06-2	1,2-Dichloroethane	32965	100.3	9.2	73	128
17060-07-0	1,2-Dichloroethane-d4	8673	99.5	6.1	81	118
540-59-0	1,2-Dichloroethene	18667	100.2	7.1	79	121
78-87-5	1,2-Dichloropropane	27787	100.1	7.2	78	122
354-23-4	1,2-Dichlorotrifluoroethane [Freon 123a]	3144	103.1	10.9	70	136
108-70-3	1,3,5-Trichlorobenzene	10037	102.1	9.2	75	130
108-67-8	1,3,5-Trimethylbenzene	27820	99.5	8.1	75	124
106-99-0	1,3-Butadiene	1202	100.6	19.2	43	158
541-73-1	1,3-Dichlorobenzene	26951	99.7	6.5	80	119
142-28-9	1,3-Dichloropropane	23811	99.1	6.5	80	119
542-75-6	1,3-Dichloropropene	9784	99.9	7.6	77	123
106-46-7	1,4-Dichlorobenzene	27715	98.3	6.5	79	118
105-05-5	1,4-Diethylbenzene	1980	98.4	6.4	79	118
123-91-1	1,4-Dioxane	17866	99	13.4	59	139
544-10-5	1-Chlorohexane	5790	99.6	8	76	124
540-84-1	2,2,4-Trimethylpentane [Isooctane]	5432	95.2	12.3	58	132
594-20-7	2,2-Dichloropropane	23775	99.7	13.2	60	139
75-85-4	2-Butanol	4332	92.7	9.1	66	120
78-93-3	2-Butanone [MEK]	26659	99.6	14.6	56	143
126-99-8	2-Chloro-1,3-butadiene	15673	100	11.7	65	135
110-75-8	2-Chloroethyl vinyl ether	18225	94.7	14.7	51	139
95-49-8	2-Chlorotoluene	23750	100	7.2	79	122
591-78-6	2-Hexanone	25368	97.9	13.5	57	139
91-57-6	2-Methylnaphthalene	3754	79.4	20.9	17	142
79-46-9	2-Nitropropane	10213	92.6	14.5	49	136
67-63-0	2-Propanol [Isopropyl alcohol]	2034	98.8	14.4	56	142
624-95-3	3,3-Dimethyl-1-butanol	6491	90.9	13.9	49	133

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**STANDARD OPERATING PROCEDURE**

TITLE: ENV-SOP-MTJL-0100 Volatile Organic Compounds by GC/MS
(EPA 8260B, 8260C, 624, 624.1, and SM6200 B)

ISSUER: Pace National – Mt. Juliet, Tennessee

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Table 4. LCS Control Limits – Method 8260 Water Matrix

CAS ID	Analyte	N Records	Mean	Standard Deviation	Lower Control Limit	Upper Control Limit
460-00-4	4-Bromofluorobenzene	9971	99.7	4.9	85	114
106-43-4	4-Chlorotoluene	23616	99.9	7.4	78	122
108-10-1	4-Methyl-2-pentanone [MIBK]	25796	98.5	10.6	67	130
67-64-1	Acetone	25006	99.5	20.1	39	160
75-05-8	Acetonitrile	13308	95.8	15.2	50	142
106-99-0	1,3-Butadiene	1202	100.6	19.2	43	158
541-73-1	1,3-Dichlorobenzene	26951	99.7	6.5	80	119
142-28-9	1,3-Dichloropropane	23811	99.1	6.5	80	119
542-75-6	1,3-Dichloropropene	9784	99.9	7.6	77	123
106-46-7	1,4-Dichlorobenzene	27715	98.3	6.5	79	118
105-05-5	1,4-Diethylbenzene	1980	98.4	6.4	79	118
123-91-1	1,4-Dioxane	17866	99	13.4	59	139
544-10-5	1-Chlorohexane	5790	99.6	8	76	124
540-84-1	2,2,4-Trimethylpentane [Isooctane]	5432	95.2	12.3	58	132
594-20-7	2,2-Dichloropropane	23775	99.7	13.2	60	139
75-85-4	2-Butanol	4332	92.7	9.1	66	120
78-93-3	2-Butanone [MEK]	26659	99.6	14.6	56	143
126-99-8	2-Chloro-1,3-butadiene	15673	100	11.7	65	135
110-75-8	2-Chloroethyl vinyl ether	18225	94.7	14.7	51	139
95-49-8	2-Chlorotoluene	23750	100	7.2	79	122
591-78-6	2-Hexanone	25368	97.9	13.5	57	139
91-57-6	2-Methylnaphthalene	3754	79.4	20.9	17	142
79-46-9	2-Nitropropane	10213	92.6	14.5	49	136
67-63-0	2-Propanol [Isopropyl alcohol]	2034	98.8	14.4	56	142
624-95-3	3,3-Dimethyl-1-butanol	6491	90.9	13.9	49	133
460-00-4	4-Bromofluorobenzene	9971	99.7	4.9	85	114
106-43-4	4-Chlorotoluene	23616	99.9	7.4	78	122
108-10-1	4-Methyl-2-pentanone [MIBK]	25796	98.5	10.6	67	130
67-64-1	Acetone	25006	99.5	20.1	39	160
75-05-8	Acetonitrile	13308	95.8	15.2	50	142
107-02-8	Acrolein [Propenal]	16380	96.8	19.3	39	155
107-13-1	Acrylonitrile	20173	99	11.9	63	135
107-05-1	Allyl chloride	15758	99	10.4	68	130
71-43-2	Benzene	34376	99.4	6.9	79	120
100-44-7	Benzyl chloride	10675	90.1	15.9	42	138
108-86-1	Bromobenzene	23762	99.7	6.7	80	120
74-97-5	Bromochloromethane	24356	100.8	7.5	78	123
75-27-4	Bromodichloromethane	26888	101.8	7.8	79	125
75-25-2	Bromoform	27675	97.8	10.8	66	130
74-83-9	Bromomethane	26717	97	14.7	53	141
75-15-0	Carbon disulfide	25719	98.8	11.5	64	133
56-23-5	Carbon tetrachloride	28870	103.8	10.7	72	136
108-90-7	Chlorobenzene	29802	100	6.1	82	118
124-48-1	Chlorodibromomethane	27424	100	8.5	74	126
75-45-6	Chlorodifluoromethane	7197	84.4	14.9	40	129

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**STANDARD OPERATING PROCEDURE**

TITLE: ENV-SOP-MTJL-0100 Volatile Organic Compounds by GC/MS
(EPA 8260B, 8260C, 624, 624.1, and SM6200 B)

ISSUER: Pace National – Mt. Juliet, Tennessee

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Table 4. LCS Control Limits – Method 8260 Water Matrix

CAS ID	Analyte	N Records	Mean	Standard Deviation	Lower Control Limit	Upper Control Limit
75-00-3	Chloroethane	27069	99	13	60	138
67-66-3	Chloroform	29373	101.1	7.5	79	124
74-87-3	Chloromethane	27697	94.5	15	50	139
156-59-2	cis-1,2-Dichloroethene	27935	100.1	7.5	78	123
10061-01-5	cis-1,3-Dichloropropene	27197	99.5	8	75	124
1476-11-5	cis-1,4-Dichloro-2-butene	1524	101.5	14.9	57	146
110-82-7	Cyclohexane	20438	100.4	10	71	130
1868-53-7	Dibromofluoromethane	5702	99.1	6.5	80	119
74-95-3	Dibromomethane	24473	101.1	7.3	79	123
75-71-8	Dichlorodifluoromethane [Freon-12]	25410	92	20.1	32	152
107-02-8	Acrolein [Propenal]	16380	96.8	19.3	39	155
107-13-1	Acrylonitrile	20173	99	11.9	63	135
107-05-1	Allyl chloride	15758	99	10.4	68	130
71-43-2	Benzene	34376	99.4	6.9	79	120
100-44-7	Benzyl chloride	10675	90.1	15.9	42	138
108-86-1	Bromobenzene	23762	99.7	6.7	80	120
74-97-5	Bromochloromethane	24356	100.8	7.5	78	123
75-27-4	Bromodichloromethane	26888	101.8	7.8	79	125
75-25-2	Bromoform	27675	97.8	10.8	66	130
74-83-9	Bromomethane	26717	97	14.7	53	141
75-15-0	Carbon disulfide	25719	98.8	11.5	64	133
56-23-5	Carbon tetrachloride	28870	103.8	10.7	72	136
108-90-7	Chlorobenzene	29802	100	6.1	82	118
124-48-1	Chlorodibromomethane	27424	100	8.5	74	126
75-45-6	Chlorodifluoromethane	7197	84.4	14.9	40	129
75-00-3	Chloroethane	27069	99	13	60	138
67-66-3	Chloroform	29373	101.1	7.5	79	124
74-87-3	Chloromethane	27697	94.5	15	50	139
156-59-2	cis-1,2-Dichloroethene	27935	100.1	7.5	78	123
10061-01-5	cis-1,3-Dichloropropene	27197	99.5	8	75	124
1476-11-5	cis-1,4-Dichloro-2-butene	1524	101.5	14.9	57	146
110-82-7	Cyclohexane	20438	100.4	10	71	130
1868-53-7	Dibromofluoromethane	5702	99.1	6.5	80	119
74-95-3	Dibromomethane	24473	101.1	7.3	79	123
75-71-8	Dichlorodifluoromethane [Freon-12]	25410	92	20.1	32	152
75-43-4	Dichlorofluoromethane	1504	101.5	9.8	72	131
60-29-7	Diethyl ether	17189	98.6	10.2	68	129
108-20-3	Diisopropyl ether	22989	97.5	10.3	67	128
64-17-5	Ethanol	9543	99.2	17.1	48	151
141-78-6	Ethyl acetate	9208	96.8	13.9	55	138
97-63-2	Ethyl methacrylate	16674	98.7	9	72	126
637-92-3	Ethyl tert-butyl ether	19841	98.3	9.4	70	127
100-41-4	Ethylbenzene	33325	99.8	7	79	121
462-06-6	Fluorobenzene	1373	97.9	6.1	80	116

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**STANDARD OPERATING PROCEDURE**

TITLE: ENV-SOP-MTJL-0100 Volatile Organic Compounds by GC/MS
(EPA 8260B, 8260C, 624, 624.1, and SM6200 B)

ISSUER: Pace National – Mt. Juliet, Tennessee

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Table 4. LCS Control Limits – Method 8260 Water Matrix

CAS ID	Analyte	N Records	Mean	Standard Deviation	Lower Control Limit	Upper Control Limit
142-82-5	Heptane	11878	94.4	15	49	140
87-68-3	Hexachlorobutadiene	23535	100.1	11.3	66	134
67-72-1	Hexachloroethane	8718	102.9	10.3	72	134
110-54-3	Hexane	15545	95.5	15.9	48	143
74-88-4	Iodomethane	20229	100	10.4	69	131
78-83-1	Isobutyl alcohol	14123	97.7	11.7	63	133
108-21-4	Isopropyl acetate [Acetic acid]	7216	97.8	11.6	63	133
98-82-8	Isopropylbenzene	28636	101.5	9.9	72	131
179601-23-1	m/p-Xylene [3/4-Xylene]	28168	100.5	6.9	80	121
126-98-7	Methacrylonitrile	15982	97.9	11.6	63	133
79-20-9	Methyl acetate	19698	96	13.2	56	136
80-62-6	Methyl methacrylate	16524	97.7	10.2	67	128
1634-04-4	Methyl tert-butyl ether [MTBE]	29660	97.3	8.8	71	124
108-87-2	Methylcyclohexane	20025	101.8	10.1	72	132
75-09-2	Methylene chloride	27659	99.4	8.3	74	124
123-86-4	n-Butyl acetate	7247	96.8	9.4	69	125
75-43-4	Dichlorofluoromethane	1504	101.5	9.8	72	131
60-29-7	Diethyl ether	17189	98.6	10.2	68	129
108-20-3	Diisopropyl ether	22989	97.5	10.3	67	128
64-17-5	Ethanol	9543	99.2	17.1	48	151
141-78-6	Ethyl acetate	9208	96.8	13.9	55	138
97-63-2	Ethyl methacrylate	16674	98.7	9	72	126
637-92-3	Ethyl tert-butyl ether	19841	98.3	9.4	70	127
100-41-4	Ethylbenzene	33325	99.8	7	79	121
462-06-6	Fluorobenzene	1373	97.9	6.1	80	116
142-82-5	Heptane	11878	94.4	15	49	140
87-68-3	Hexachlorobutadiene	23535	100.1	11.3	66	134
67-72-1	Hexachloroethane	8718	102.9	10.3	72	134
110-54-3	Hexane	15545	95.5	15.9	48	143
74-88-4	Iodomethane	20229	100	10.4	69	131
78-83-1	Isobutyl alcohol	14123	97.7	11.7	63	133
108-21-4	Isopropyl acetate [Acetic acid]	7216	97.8	11.6	63	133
98-82-8	Isopropylbenzene	28636	101.5	9.9	72	131
179601-23-1	m/p-Xylene [3/4-Xylene]	28168	100.5	6.9	80	121
126-98-7	Methacrylonitrile	15982	97.9	11.6	63	133
79-20-9	Methyl acetate	19698	96	13.2	56	136
80-62-6	Methyl methacrylate	16524	97.7	10.2	67	128
1634-04-4	Methyl tert-butyl ether [MTBE]	29660	97.3	8.8	71	124
108-87-2	Methylcyclohexane	20025	101.8	10.1	72	132
75-09-2	Methylene chloride	27659	99.4	8.3	74	124
123-86-4	n-Butyl acetate	7247	96.8	9.4	69	125
71-36-3	n-Butyl alcohol	10122	95.1	12	59	131
104-51-8	n-Butylbenzene	24088	101.1	8.8	75	128
109-60-4	n-Propyl acetate	602	100.8	8.3	76	126
103-65-1	n-Propylbenzene	24419	101	8.5	76	126
91-20-3	Naphthalene	27847	94.6	11.3	61	128

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(EPA 8260B, 8260C, 624, 624.1, and SM6200 B)

ISSUER: Pace National – Mt. Juliet, Tennessee

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Table 4. LCS Control Limits – Method 8260 Water Matrix

CAS ID	Analyte	N Records	Mean	Standard Deviation	Lower Control Limit	Upper Control Limit
95-47-6	o-Xylene	31776	100	7.2	78	122
99-87-6	p-Isopropyltoluene [p-Cymene]	24335	102	8.5	77	127
76-01-7	Pentachloroethane	11688	101.1	10.7	69	133
109-66-0	Pentane	3915	74.8	19.7	16	134
107-12-0	Propionitrile [Ethyl cyanide]	15701	99.9	12	64	136
135-98-8	sec-Butylbenzene	24191	101.1	8.1	77	126
100-42-5	Styrene	26985	100.5	7.6	78	123
994-05-8	tert-Amyl methyl ether [TAME]	19726	98.1	10.1	68	128
75-65-0	tert-Butyl alcohol	21112	98.6	10.1	68	129
762-75-4	tert-Butyl formate	6651	98.1	11.1	65	132
98-06-6	tert-Butylbenzene	23919	101	7.7	78	124
127-18-4	Tetrachloroethene	29017	101.3	9.3	74	129
109-99-9	Tetrahydrofuran	18021	95	12.8	57	133
108-88-3	Toluene	33510	100.1	6.8	80	121
2037-26-5	Toluene-d8	9809	100.4	3.8	89	112
156-60-5	trans-1,2-Dichloroethene	27663	99.5	8.2	75	124
10061-02-6	trans-1,3-Dichloropropene	27134	100	8.9	73	127
110-57-6	trans-1,4-Dichloro-2-butene	19320	91.5	16.1	43	140
79-01-6	Trichloroethene	30150	101.1	7.3	79	123
75-69-4	Trichlorofluoromethane	26108	103	12.8	65	141
71-36-3	n-Butyl alcohol	10122	95.1	12	59	131
104-51-8	n-Butylbenzene	24088	101.1	8.8	75	128
109-60-4	n-Propyl acetate	602	100.8	8.3	76	126
103-65-1	n-Propylbenzene	24419	101	8.5	76	126
91-20-3	Naphthalene	27847	94.6	11.3	61	128
95-47-6	o-Xylene	31776	100	7.2	78	122
99-87-6	p-Isopropyltoluene [p-Cymene]	24335	102	8.5	77	127
76-01-7	Pentachloroethane	11688	101.1	10.7	69	133
109-66-0	Pentane	3915	74.8	19.7	16	134
107-12-0	Propionitrile [Ethyl cyanide]	15701	99.9	12	64	136
135-98-8	sec-Butylbenzene	24191	101.1	8.1	77	126
100-42-5	Styrene	26985	100.5	7.6	78	123
994-05-8	tert-Amyl methyl ether [TAME]	19726	98.1	10.1	68	128
75-65-0	tert-Butyl alcohol	21112	98.6	10.1	68	129
762-75-4	tert-Butyl formate	6651	98.1	11.1	65	132
98-06-6	tert-Butylbenzene	23919	101	7.7	78	124
127-18-4	Tetrachloroethene	29017	101.3	9.3	74	129
109-99-9	Tetrahydrofuran	18021	95	12.8	57	133
108-88-3	Toluene	33510	100.1	6.8	80	121
2037-26-5	Toluene-d8	9809	100.4	3.8	89	112
156-60-5	trans-1,2-Dichloroethene	27663	99.5	8.2	75	124
10061-02-6	trans-1,3-Dichloropropene	27134	100	8.9	73	127

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STANDARD OPERATING PROCEDURE
TITLE: ENV-SOP-MTJL-0100 Volatile Organic Compounds by GC/MS
(EPA 8260B, 8260C, 624, 624.1, and SM6200 B)

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Table 4. LCS Control Limits – Method 8260 Water Matrix

CAS ID	Analyte	N Records	Mean	Standard Deviation	Lower Control Limit	Upper Control Limit
110-57-6	trans-1,4-Dichloro-2-butene	19320	91.5	16.1	43	140
79-01-6	Trichloroethene	30150	101.1	7.3	79	123
75-69-4	Trichlorofluoromethane [Freon-11]	26108	103	12.8	65	141
108-05-4	Vinyl acetate	18941	100.2	15.3	54	146
75-01-4	Vinyl chloride	29472	97.4	13.2	58	137
1330-20-7	Xylenes [total]	23426	100.1	7	79	121

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Table 5. Quality Control Requirements – Organic Analysis by Gas Chromatography/Mass Spectrometry

QC Check	Minimum Frequency	Acceptance Criteria	Corrective Action	Flagging Criteria	Comments
Tune Check	Prior to ICAL and prior to each 12-hour period of sample analysis.	Specific ion abundance criteria of BFB from method.	Retune instrument and verify.	Flagging is not appropriate.	No samples shall be analyzed without a valid tune.
Initial calibration (ICAL) for all analytes (including surrogates)	At instrument set-up, prior to sample analysis	Each analyte must meet one of the three options below: Option 1: RSD for each analyte $\leq 15\%$; Option 2: linear least squares regression for each analyte: $r^2 \geq 0.99$; Option 3: non-linear least squares regression (quadratic) for each analyte: $r^2 \geq 0.99$.	Correct problem then repeat ICAL.	Flagging is not appropriate.	Minimum 5 levels for linear and 6 levels for quadratic. No samples shall be analyzed until ICAL has passed. If the specific version of a method requires additional evaluation (e.g., RFs or low calibration standard analysis and recovery criteria) these additional requirements must also be met.
Retention Time window position establishment	Once per ICAL and at the beginning of the analytical sequence.	Position shall be set using the midpoint standard of the ICAL curve when ICAL is performed. On days when ICAL is not performed, the initial CCV is used.	NA.	NA.	Calculated for each analyte and surrogate.
Evaluation of Relative Retention Times(RRT)	With each sample.	RRT of each reported analyte within ± 0.06 RRT units.	Correct problem, then rerun ICAL.	NA	After maintenance is performed which may affect retention times, RRTs may be updated based on the daily CCV. RRTs shall be compared with the most recently updated RRTs.

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Table 5. Quality Control Requirements – Organic Analysis by Gas Chromatography/Mass Spectrometry

QC Check	Minimum Frequency	Acceptance Criteria	Corrective Action	Flagging Criteria	Comments
Initial Calibration Verification (ICV)	Once after each ICAL, analysis of a second source standard prior to sample analysis	All reported analytes within $\pm 20\%$ of true value.	Correct problem. Rerun ICV. If that fails, repeat ICAL.	Flagging is not appropriate.	No samples shall be analyzed until calibration has been verified with a second source.
Continuing Calibration Verification (CCV)	Daily before sample analysis; after every 12 hours of analysis time; and at the end of the analytical batch run.	All reported analytes and surrogates within $\pm 20\%$ of true value. All reported analytes and surrogates within $\pm 50\%$ for end of analytical batch CCV.	Immediately analyze two additional consecutive CCVs. If both pass, samples may be reported without reanalysis. If either fails or if two consecutive CCVs cannot be run, perform corrective action(s) and repeat the CCV and all associated samples since the last successful CCV. Alternately, Recalibrate if necessary; then reanalyze all associated samples since the last acceptable CCV	If reanalysis cannot be performed, data must be qualified and explained in the case narrative. Apply Q-flag to all results for the specific analyte(s) in all samples since last acceptable calibration verification.	Results may not be reported without a valid CCV. Flagging is only appropriate in cases where the samples cannot be reanalyzed. If the specific version of a method requires additional evaluation (e.g., average RFs) these additional requirements must also be met.
Internal standards (IS)	Every field sample, standard and QC sample.	Retention time within ± 10 seconds from retention time of the midpoint standard in the ICAL; EICP area within - 50% to +100% of ICAL midpoint standard. On days when ICAL is not performed, the daily initial CCV can be used.	Inspect mass spectrometer and GC for malfunctions and correct problem. Reanalysis of samples analyzed while system was malfunctioning is mandatory.	If corrective action fails in field samples, data must be qualified and explained in the case narrative. Apply Q-flag to analytes associated with the non-compliant IS. Flagging is not appropriate for failed standards.	

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Table 5. Quality Control Requirements – Organic Analysis by Gas Chromatography/Mass Spectrometry

QC Check	Minimum Frequency	Acceptance Criteria	Corrective Action	Flagging Criteria	Comments
Method Blank (MB)	One per preparatory batch.	No analytes detected > ½ LOQ or > 1/10 the amount measured in any sample or 1/10 the regulatory limit, whichever is greater. Common contaminants must not be detected > LOQ.	Correct problem. If required, reprep and reanalyze MB and all samples processed with the contaminated blank.	If reanalysis cannot be performed, data must be qualified and explained in the case narrative. Apply B-flag to all results for the specific analyte(s) in all samples in the associated preparatory batch.	Results may not be reported without a valid method blank. Flagging is only appropriate in cases where the samples cannot be reanalyzed.
Laboratory Control Sample (LCS)	One per preparatory batch.	A laboratory must use the limits in Tables 3 and 4 for batch control if project limits are not specified. If the analyte(s) are not listed, use in-house LCS limits (see the LIMS) if project limits are not specified.	Correct problem, then reprep and reanalyze the LCS and all samples in the associated preparatory batch for failed analytes, if sufficient sample material is available	If reanalysis cannot be performed, data must be qualified and explained in the case narrative. Apply Q-flag to specific analyte(s) in all samples in the associated preparatory batch.	Must contain all surrogates and all analytes to be reported. Results may not be reported without a valid LCS. Flagging is only appropriate in cases where the samples cannot be reanalyzed.
Matrix Spike (MS)	One per preparatory batch.	A laboratory must use the limits in Tables 3 and 4 for batch control if project limits are not specified. If the analyte(s) are not listed, use in-house LCS limits (see the LIMS) if project limits are not specified.	Examine the project specific requirements. Contact the client as to additional measures to be taken.	For the specific analyte(s) in the parent sample, apply J-flag if acceptance criteria are not met and explain in the case narrative.	Must contain all surrogates and all analytes to be reported. For matrix evaluation only. If MS results are outside the limits, the data shall be evaluated to determine the source(s) of difference, i.e., matrix effect or analytical error.

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Table 5. Quality Control Requirements – Organic Analysis by Gas Chromatography/Mass Spectrometry

QC Check	Minimum Frequency	Acceptance Criteria	Corrective Action	Flagging Criteria	Comments
Matrix Spike Duplicate (MSD) or Matrix Duplicate (MD)	One per preparatory batch.	A laboratory must use the limits in Tables 3 and 4 for batch control if project limits are not specified. If the analyte(s) are not listed, use in-house LCS limits (see the LIMS) if project limits are not specified. MSD or MD: RPD of all analytes $\leq 20\%$ (between MS and MSD or sample and MD).	Examine the project specific requirements. Contact the client as to additional measures to be taken.	For the specific analyte(s) in the parent sample, apply J-flag if acceptance criteria are not met and explain in the case narrative.	MSD: Must contain all surrogates and all analytes to be reported. The data shall be evaluated to determine the source of difference. For Sample/MD: RPD criteria only apply to analytes whose concentration in the sample is greater than or equal to the LOQ.
Surrogate Spike	All field and QC samples.	QC acceptance criteria specified by the project, if available; otherwise use limits in Tables 3 and 4 or in-house LCS limits (see the LIMS) if analyte(s) are not listed.	Correct problem, then prep and reanalyze all failed samples for all surrogates in the associated preparatory batch, if sufficient sample material is available. If obvious chromatographic interference is present, reanalysis may not be necessary, but the client must be notified prior to reporting data and the failures must be discussed in the case narrative.	Apply Q-flag to all associated analytes if acceptance criteria are not met and explain in the case narrative.	Alternative surrogates are recommended when there is obvious chromatographic interference.



Document Information

Document Number: ENV-SOP-MTJL-0215	Revision: 06
Document Title: Determination Metals and Trace Elements in Various Matrices by ICP-AES (EPA Methods 6010B, 6010C, 6010D [ICP-OES], and 200.7) Including Hardness (EPA Methods 200.7 and 6010B/C/D and SM 2340B)	
Department(s): Metals	

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All Dates and Times are listed in: Central Time Zone

Signature Manifest

Document Number: ENV-SOP-MTJL-0215**Revision:** 06**Title:** Determination Metals and Trace Elements in Various Matrices by ICP-AES (EPA Methods 6010B, 6010C, 6010D [ICP-OES], and 200.7) Including Hardness (EPA Methods 200.7 and 6010B/C/D and SM 2340B)

All dates and times are in Central Time Zone.

ENV-SOP-MTJL-0215

QM Approval

Name/Signature	Title	Date	Meaning/Reason
Regina Stone (006490)	Quality Analyst 3	05 Feb 2020, 10:03:45 AM	Approved

Management Approval

Name/Signature	Title	Date	Meaning/Reason
Steven Miller (006597)	Manager - Quality	05 Feb 2020, 10:02:52 AM	Approved



STANDARD OPERATING PROCEDURE

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1.0 SCOPE AND APPLICATION

STATE NOTE: For samples analyzed in conjunction with the Ohio Voluntary Action Program (VAP) please utilize ENV-SOP-MTJL-0214.

- 1.1 Inductively coupled plasma-atomic emission spectrometry (ICP-AES) determines trace elements, including metals and some non-metals in solution. This procedure follows the guidelines established in EPA method 200.7 and SW-846 Method 6010B, 6010C, and 6010D for drinking water, waste water, ground water, TCLP, SPLP, and STLC leachates, soils, sludge, sediments, solid wastes, oils, and other digestates after appropriate preparatory procedure is performed.

This procedure is also applicable to reporting calculated values for Calcium, Magnesium, and Total Hardness from values determined using EPA methods 200.7 or 6010B/C/D from groundwater, wastewater and drinking waters. Reporting limits for Hardness are derived from the annual MDL studies for Calcium and Magnesium of the appropriate determinative EPA method. The routine reporting limits for each category of hardness are listed in Table 1.2b.

- 1.2 This method is applicable for the analytes listed in Table 1.2a and b. Detection limits, sensitivity, and the optimum and linear concentration ranges of the elements can vary with the wavelength, spectrometer, matrix, and instrument operating conditions. Table 1.2 also lists the Reporting Limits (RLs), used routinely by Pace Analytical National Center for Testing & Innovation (Pace National).

Table 1.2a: Environmental Analytes and Reporting Limits (Subject to change, see section 13.1)

Analyte	Aqueous				Sediment		
	Ground Water/ Wastewater 6010B/C/D/200 .7	Drinking Water 200.7*	RL	Units	Solids 6010B/C/ D	RL	Units
Aluminum	✓	✓	00.200	mg/L	✓	2.00	mg/Kg
Antimony	✓	✓	0.010	mg/L	✓	1.00	mg/Kg
Arsenic	✓	✓	00.010	mg/L	✓	1.00	mg/Kg
Barium	✓	✓	0.005	mg/L	✓	0.50	mg/Kg
Beryllium	✓	✓	0.002	mg/L	✓	0.20	mg/Kg
Boron	✓	✓	0.050	mg/L	✓	5.0	mg/Kg
Cadmium	✓	✓	0.002	mg/L	✓	0.20	mg/Kg
Calcium	✓	✓	1.000	mg/L	✓	100	mg/Kg
Chromium	✓	✓	0.010	mg/L	✓	1.00	mg/Kg
Cobalt	✓	✓	0.010	mg/L	✓	1.00	mg/Kg
Copper	✓	✓	0.010	mg/L	✓	1.00	mg/Kg
Iron	✓	✓	0.100	mg/L	✓	10.0	mg/Kg
Lead	✓	✓	0.005	mg/L	✓	0.50	mg/Kg
Lithium	✓		0.015	mg/L	✓	1.50	mg/Kg
Magnesium	✓	✓	1.000	mg/L	✓	100	mg/Kg
Manganese	✓	✓	0.010	mg/L	✓	1.00	mg/Kg
Molybdenum	✓	✓	0.005	mg/L	✓	0.50	mg/Kg

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Analyte	Aqueous				Sediment		
	Ground Water/ Wastewater 6010B/C/D/200.7	Drinking Water 200.7*	RL	Units	Solids 6010B/C/D	RL	Units
Nickel	✓	✓	0.010	mg/L	✓	1.00	mg/Kg
Potassium	✓	✓	1.000	mg/L	✓	100	mg/Kg
Selenium	✓	✓	0.010	mg/L	✓	1.00	mg/Kg
Silicon	✓	✓	0.050	mg/L	✓	5.00	mg/Kg
Silver	✓	✓	0.005	mg/L	✓	5.00	mg/Kg
Sulfur	✓	✓	1.0	mg/L	✓		mg/Kg
Sodium	✓	✓	1.000	mg/L	✓	100	mg/Kg
Strontium	✓	✓	0.010	mg/L	✓	1.00	mg/Kg
Thallium	✓	✓	0.010	mg/L	✓	1.00	mg/Kg
Tin	✓	✓	0.050	mg/L	✓	5.00	mg/Kg
Titanium	✓	✓	0.050	mg/L	✓	5.00	mg/Kg
Vanadium	✓	✓	0.010	mg/L	✓	1.00	mg/Kg
Zinc	✓	✓	0.050	mg/L	✓	5.00	mg/Kg

*May not meet required Drinking Water Maximum Contamination Levels (MCLs) using this methodology.

Table 1.2b: Hardness Categories and Reporting Limits

(Subject to change, see section 13.1)

Hardness:	RL (mg/L)
Calcium Hardness	1.25
Magnesium Hardness	0.41
Total Hardness	1.6

- 1.3 For the determination of total recoverable analytes in aqueous and solid samples, an acid digestion process is required. Environmental samples for analysis by Method 6010B, 6010C, or 6010D including, TCLP or EP leachates, soils, sludge, sediments, and other solid wastes require an acid digestion prior to analysis. Samples are digested by SW-846 methods 3005 (Acid Digestion of Waters for Total Recoverable Metals), 3010 (Acid Digestion of Aqueous Samples), 3015 (Microwave Digestion of Aqueous Samples), 3050 (Acid Digestion of Sediments, Sludge, Soil, and Oils) and 3051 (Microwave Assisted Digestion of Sediments, Sludge, Soil, and Oils). Digestion methods are found in ENV-SOP-MTJL-0217 and ENV-SOP-MTJL-0219.
- 1.4 The Clean Water Act has approved EPA Method 200.7 for demonstrating compliance on discharge monitoring for NPDES (National Pollution Discharge Elimination System) permits. 40 CFR136.3 has Guidelines for Establishing Test Procedures for Analysis of Pollutants. The National Primary Drinking Water Regulations for inorganic chemical sampling and analytical requirements can be found in 40 CFR141.23. Updates to these regulations can be found in the current Code of the Federal Register.



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- 1.5 To determine dissolved analytes in aqueous samples, a 0.45µm filtration method is employed then the filtered samples are acidified. To reduce potential interferences, dissolved solids must be <0.2% (w/v).
- 1.6 Analysis without acid digestion can be used for drinking water samples if the samples have been properly preserved with acid and have turbidity of <1 NTU at the time of analysis. These samples must be acidified to match the acid matrix of the calibration standards and analyzed directly. This total recoverable determination procedure is referred to as "direct analysis". Silver concentration cannot be determined from direct analysis when chloride ions are present as a silver chloride precipitate may be formed. The sample must be acid digested to form a soluble silver chloride complex. Some primary drinking water metal contaminants may require sample concentration to meet regulatory drinking water reporting limits criteria^{14,2}.

Method 6010D – Samples that are not digested necessitate the use of either an internal standard or should be matrix-matched with the standards. If using the former option, the instrument software should be programmed to correct for the intensity differences of the internal standard between samples and standards. **NOTE:** All samples analyzed by Method 6010 are typically digested.

- 1.7 When determining boron and silicon in aqueous samples, only plastic, PTFE (Teflon™) sample containers and laboratory glassware must be used. For accurate determination of boron in solid samples, only quartz or PTFE tubes must be used during acid digestion with immediate transfer of an aliquot of the final volume of digestate to a plastic centrifuge tube^{14,2}.
- 1.8 For the determination of titanium, white plastic and white printed containers must be avoided as titanium dioxide is used as a white pigment.
- 1.9 The total recoverable sample digestion procedure dissolves and maintains in solution only minimal concentrations of barium in the presence of free sulfate. For the analysis of barium in samples having varying and unknown concentrations of sulfate, analysis must be completed as soon as possible following sample preparation^{14,2}.
- 1.10 Detection limits and linear ranges for the elements vary with the wavelength selected, the spectrometer, and the matrix. Table 1.11 provides a list of routinely used wavelengths and the type of spectrometer view used.

Method 6010D – IDLs are necessarily instrument-specific. Therefore, if needed, an IDL must be determined through a separate experimental study for each instrument. IDLs should be established, at a minimum, on an annual basis for each matrix and for each preparatory/determinative method combination used.

TABLE 1.10: WAVELENGTHS
(exact wavelengths vary slightly depending on the instrument)

Analyte	Wavelength (nm)	Type of View
Aluminum	308.215	Radial
Antimony	206.836	Axial
Arsenic	188.979	Axial



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Analyte	Wavelength (nm)	Type of View
Barium	233.527	Axial
Beryllium	313.107	Radial
Boron	249.772	Radial
Cadmium	214.440	Axial
Calcium	317.933 373.690	Radial
Chromium	205.560	Axial
Cobalt	228.616	Axial
Copper	324.752	Radial
Iron	259.940 271.441	Radial
Lead	220.353	Axial
Lithium	670.784	Radial
Magnesium	279.077	Radial
Manganese	257.610	Axial
Molybdenum	202.031	Axial
Nickel	232.003	Axial
Potassium	766.490	Radial
Phosphorus	177.495	Axial
Selenium	196.026	Axial
Silicon	251.611	Axial
Silver	328.068	Axial
Sodium	589.592 818.326	Radial
Strontium	407.771	Radial
Sulfur	181.972	Axial
Thallium	190.801	Axial
Tin	189.927	Axial
Titanium	334.940	Radial
Vanadium	292.402	Radial
Zinc	213.857	Axial

- 1.11 Users of the data generated using this method must state the data-quality objectives (DQOs) prior to analysis.
- 1.12 Any deviations from this SOP must be documented. Deviations are reflected in a case narrative and the method is reported as modified. Per customer requirement, the procedure and QC criteria described in this SOP can be changed/modified. Authorization from the Operations Manager and Project Manager is required for each modification and Regulatory Affairs approval must also be secured for any deviation.

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- 1.13 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 ENV-SOP-MTJL-0016. Updated MDL records are filed and stored in a central location within the department.
 - 1.13.1 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 ENV-SOP-MTJL-0016, *Method Detection Limits (MDL)*, *Limits of Detection (LOD)*, and *Limits of Quantitation (LOQ)*. Should the procedure be utilized for DoD support; then the frequency of these studies must meet the requirements of the current DoD QSM.
- 1.14 Linear Dynamic Range (LDR) and Inter-element correction factor (IEC) studies must be analyzed semi-annually for each analytical instrument or when there are major changes/repairs to the instrument^{14.5, 14.1}. Instrument Detection Limit studies must be analyzed at least quarterly for each analytical instrument^{14.5}.

2.0 METHOD SUMMARY AND DEFINITIONS

- 2.1 The analysis described in this method involves multi-elemental determinations by ICP-AES using sequential or simultaneous instruments. The instrument measures characteristic atomic-line emission spectra by optical spectrometry. Samples are aspirated into the nebulizer and the resulting aerosol is transported to the plasma torch. The emission spectra are dispersed by a grating spectrometer separating the light emitted into the distinct wavelengths generated by each element in the sample. A photosensitive device monitors the intensities of each wavelength line in the spectra. The intensity of light on the photosensitive device produces a signal that is measured and processed by a computer system. Due to the many possible wavelengths of light generated by each element and possible overlapping of high intensity peaks, a background correction technique is required for trace element determination. Background intensities must be measured adjacent to the analyte spectra lines during analysis. The position selected for background intensity measurement can be selected on either or both sides of the analyte wavelength line and must be determined by the complexity of the spectrum adjacent to the analyte line. The position used for background correction must be as free from spectral interference as possible and must reflect the same change in background intensity as occurs at the analyte wavelength. Background correction is not required in cases of line broadening where the background correction measurement would actually degrade the analytical result. The possibility of additional interferences should also be recognized and appropriate corrections made.
- 2.2 Dissolved Analyte - The concentration of analyte in an aqueous sample that has been passed through a 0.45µm membrane filter assembly prior to sample acidification and digestion.
- 2.3 Total (Total Recoverable) Analyte – The concentration of analyte determined either by “direct analysis” of an unfiltered acid preserved drinking water sample with turbidity of <1 NTU or by analysis of the solution extract of a solid sample or an unfiltered aqueous sample following digestion by refluxing with hot dilute mineral acid(s) as specified in the method

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- 2.4 Instrument Detection Limit (IDL) - The concentration equivalent to the analyte signal which is equal to three times the standard deviation of a series of 10 replicate measurements of the calibration blank signal at the same wavelength. The IDL assures with 99% certainty that a value is above the instrument noise level.
- Note:** An IDL is a statistical determination without analytes present used to assess background correction protocols and an MDL is determined with low levels of analytes present to determine instrument sensitivity for each analyte.
- 2.5 Linear Dynamic Range (LDR) - The range over which the instrument response to analyte concentration remains linear.
- 2.6 Plasma Solution - A solution that is used to determine the optimum torch height relative to the radio frequency (RF) coil for viewing the spectrum.
- 2.7 Interference Check Sample (ICS) – A series of two solutions (ICSA & ICSAB) to verify that inter-element interferences are correctly compensated. The ICSA and ICSAB provide an adequate on-going test of inter-element correction (IEC) factors. These standards are referred to the Spectra Interference Check (SIC) in EPA Method 200.7
- 2.7.1 ICSA – A solution containing only the interfering analytes at high concentrations.
- 2.7.2 ICSAB – A solution containing interferents plus other method analytes at the level of concern, which corresponds to the project specific action limits.
- 2.8 Water Sample - For the purpose of this method, a sample taken from one of the following sources: drinking water, surface water, ground water, storm water, industrial or domestic wastewater.
- 2.9 Preparation Batch - For method 6010B/C/D/ EPA 200.7 (WW only): A group of samples (not to exceed twenty) of a similar matrix, which have been digested at the same time using the same digestion process and have all necessary QC associated with them. For method 200.7 (DW only): A group of samples (not to exceed ten) of similar matrix, which have been digested at the same time using the same digestion process and have all necessary associated QC.
- 2.10 Analytical batch - A group of samples that are analyzed in the same sequence with all appropriate preparation and analytical QC.
- 2.11 Inter-element correction (IEC) coefficient - analyte concentration equivalent arising from a given interferent's concentration.
- 2.12 Serial Dilution - a dilution and reanalysis of a field sample that is performed once per batch of samples. One sample is diluted 5X and reanalyzed.
- 2.13 Post Spike – A second aliquot of a field sample that is spiked with known concentrations of target analytes and analyzed to assess recovery of the spike. A post spike must be analyzed when the MS and/or MSD fail due to a suspected matrix effect. One sample is spiked after digestion and analyzed per batch.
- 2.14 Lower Limit of Quantitation (LLOQ) - A term associated with analysis per the requirements of Method 6010D; the lowest point of quantitation which, in most cases, is the lowest concentration in the calibration curve.

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- 2.15 See the current Quality Assurance Manual for other definitions associated with terms found in this document.

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. A reference file of safety data sheets (SDSs) are made available on Pace National's intranet to all personnel. Use hazardous 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 protocols.
- 3.2 The acidification of samples containing reactive materials may result in the release of toxic gases, such as cyanides or sulfides. Acidification of samples should be done in a fume hood.
- 3.3 All personnel handling environmental samples known to contain or to have been in contact with human waste should be immunized against known disease causative agents.
- 3.4 All ICP instruments provide protection from ultraviolet light emission. These shielding screens cannot be removed.

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 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 per the ENV-SOP-MTJL-0014, *Data Handling and Reporting*.
- 4.3 Prior to the collection of an aqueous sample, consideration must be given to the type of data required, (i.e., dissolved or total recoverable), so that appropriate preservation and pre-treatment steps can be taken. The pH of all aqueous samples must be assessed immediately prior to sample digestion or "direct analysis" to ensure the sample has been properly preserved. If the field sample is properly preserved, the sample can be held up to 6 months prior to analysis.
- 4.4 For the determination of dissolved elements, the sample must be filtered through a 0.45µm pore diameter membrane filter to remove the suspended elements or particles. This filtration must take place at the time of collection or as soon thereafter as practically possible. Glass or plastic filtering apparatus are recommended to avoid possible contamination. Only plastic apparatus must be used when the determinations of boron and silica are critical. Use a portion of the filtered sample to rinse the filter flask, discard this portion and collect the required volume of filtrate. Acidify the filtrate with (1:1) nitric acid: water immediately following filtration to pH <2.

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- 4.5 For the determination of total recoverable elements in aqueous samples, samples must not be filtered, but acidified with (1:1) nitric acid: water to pH <2. Preservation may be done at the time of collection; however, to avoid the hazards of strong acid use in the field, possible transport restrictions, or possible contamination, it is recommended that the samples be returned to the laboratory within two weeks of collection and acid preserved upon receipt in the laboratory. Following acidification, the sample must be mixed and equilibrated for 24 hours. The pH is verified at <2 prior to withdrawing an aliquot for acid digestion or "direct analysis". If, for reasons such as high alkalinity, the sample pH is verified to be >2, more acid must be added and the sample equilibrated for another sixteen hours until verified to be pH <2.
- 4.6 Solid samples require no preservation prior to analysis. Solid samples can be held up to six months from the time of sample collection until preparation and analysis.
- 4.7 For aqueous samples, a field blank must be prepared and analyzed as required by the data user. Use the same container and preservative as is used in field sample collection. The sample holding time is six (6) months from the date and time of collection until analysis. Samples are preserved to pH <2 with nitric acid.

5.0 INTERFERENCES

- 5.1 Spectral interferences are caused by background emission from continuous or recombination phenomena, stray light from the line emission of high concentration elements, overlap of a spectral line from another element, or unresolved overlap of molecular band spectra.
 - 5.1.1 Subtracting the background emission determined by measurement(s) adjacent to the analyte wavelength peak can usually compensate for background emission and stray light. The location(s) selected for the measurement of background intensity is determined by the complexity of the spectrum adjacent to the wavelength peak. The location(s) used for routine measurement must be free of off-line spectral interference (inter-element or molecular) or adequately corrected to reflect the same change in background intensity as occurs at the wavelength peak. Changes in background correction must be saved in the instrument method. Background correction can be established by scanning the following three solutions: 1) blank (same as calibration blank); 2) solution, containing analytes at significant concentration to raise a signal above background signal (CCV solution may be used) at mid-range of the curve; 3) solution(s) containing most common interfering elements at high concentration and other interferents as well (ICSAB solution may be used).
 - 5.1.2 Spectral overlaps can be compensated for by equations that correct for inter-element contributions, which involve measuring the interfering elements. When operative and uncorrected, these interferences produce false-positive determinations and are reported as analyte concentrations. Users may apply inter-element correction factors determined on their instruments within tested concentration ranges to compensate (offline or online) for the effects of interfering elements. Consult the method for specific identified interferences.



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- 5.1.3 When inter-element corrections are applied, there is a need to verify their accuracy by analyzing spectral interference check solutions. The IEC's are established by analyzing a solution of the interfering element at a high concentration within the LDR limit, measuring the analyte concentration equivalents arising from the interfering element, calculating the interference factor as analyte reading in mg/L, then dividing by the interfering element concentration. The IEC's are changed in the stored ICP instrument method. Inter-element corrections vary for the same emission line among instruments because of differences in resolution, as determined by the grating plus the entrance and exit slit widths, and by the order of dispersion. Inter-element corrections also vary depending upon the choice of background correction points. Selecting a background correction point where an interfering emission line may appear should be avoided. Inter-element corrections that constitute a major portion of an emission signal may not yield accurate data. Users must not forget that some samples might contain uncommon elements that could contribute spectral interferences.
- 5.1.4 Interference effects must be evaluated for each individual instrument. For each instrument, intensities vary not only with optical resolution but also with operating conditions (such as power, viewing height and argon flow rate). To determine the appropriate location for offline background correction, the user must scan the area on either side of the peak adjacent to the wavelength and record the apparent emission intensity from all other method analytes. The location selected for background correction must be either free from offline inter-element spectral interference or a computer routine must be used for their automatic correction on all determinations.
- 5.2 Physical interferences are effects associated with the sample nebulization and transport processes. Changes in viscosity and surface tension can cause significant inaccuracies, especially in samples containing high dissolved solids or high acid concentrations. If physical interferences are present, they must be reduced by such means as using a high-solids nebulizer, diluting the sample, using a peristaltic pump, or using an appropriate internal standard element. Another problem that can occur with high dissolved solids is salt buildup at the tip of the nebulizer, which affects aerosol flow rate and causes instrumental drift. This can be controlled using a high-solids nebulizer, wetting the argon prior to nebulization, using a tip washer, or diluting the sample. Also, it has been reported that better control of the argon flow rates, especially for the nebulizer, improves instrument stability and precision. This is accomplished with the use of mass flow controllers.
- 5.3 Chemical interferences include molecular-compound formation, ionization effects, and solute-vaporization effects. Normally, these effects are not significant with the ICP-AES technique. If observed, they can be minimized by careful selection of operating conditions (such as incident power and observation height), by buffering of the sample, by matrix matching and by standards addition procedures. Chemical interferences are highly dependent on matrix type.



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- 5.4 Memory interferences result when analytes in a previous sample contribute to the signals measured in a new sample. Memory effects can result from sample deposition on the uptake tubing to the nebulizer and from the buildup of sample material in the plasma torch and spray chamber. The site where these effects occur is dependent on the element and can be minimized by flushing the system with a rinse blank between samples. The possibility of memory interferences must be recognized within an analytical run and suitable rinse times must be used to reduce them.
 - 5.5 Linear Dynamic Range (LDR) study is performed by analyzing a solution of each element at maximal concentration unless the result falls outside 10% RPD. The highest calibration standard for each analyte cannot be greater than the LDR for that analyte. If an interferent is found greater than the LDR and an IEC factor is established between the interferent and analyte of interest, the sample must be diluted for proper correction of inter-element interferences. Instrument methods with different calibration standard concentrations require separate LDR studies.
 - 5.6 Background correction is performed as needed and LDR and IEC studies are completed as required by each published analytical method and whenever significant changes to instrumentation are made. Background, or blank matrix, subtraction is not performed for environmental samples.
- 6.0 EQUIPMENT AND SUPPLIES
- 6.1 Inductively coupled plasma emission spectrometer:
 - 6.1.1 Perkin Elmer Model 5300 or Thermo Model 7000 series ICP, or equivalent, with background correction and computer control
 - 6.1.2 Cetac Autosampler or ESI autosampler
 - 6.1.3 Argon gas supply - High purity grade (99.99%). When analyses are conducted frequently, liquid argon is more economical and requires less frequent replacement of tanks than compressed argon in conventional cylinders.
 - 6.2 Narrow-mouth storage bottles, FEP (fluorinated ethylene propylene) with screw closure, 125mL to 1L capacities
 - 6.3 One-piece stem FEP wash bottle with screw closure, 125mL capacity
 - 6.4 Adjustable pipettes (Eppendorf or equivalent), ranges from 2µL to 5000µL
 - 6.5 Class A volumetric flasks for standards preparations
 - 6.6 Polypropylene (PP) conical tubes
 - 6.7 Peristaltic pump



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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 ENV-SOP-MTJL-0041, *Standard Logger – Tree Operation*. 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 six (6) months or sooner if a problem is detected unless otherwise noted.
- 7.2 Hydrochloric acid, concentrated (sp. gr. 1.19) - HCl
- 7.2.1 Hydrochloric acid (1+1) - Add 500mL concentrated HCl to 400mL reagent water and dilute to 1L with reagent water.
- 7.3 Nitric acid, concentrated (sp. gr. 1.41) - HNO₃
- 7.3.1 Nitric acid (1+1) - Add 500mL concentrated HNO₃ to 400mL reagent water and dilute to 1L with reagent water.
- 7.4 Laboratory Reagent water
- 7.5 ICP Standard List

Calibration	Stock Std Cat #	Working Standard Preparation
RL Standard #1	HP7013-500	50mL of Stock LL Std, 10% rinse to volume 1000mL
0.5ppm Standard #2	HP6928-1L	50mL of 2.0ppm Std, 10% rinse to volume 150mL
1ppm Standard #3		100mL of 2.0ppm, 10% rinse to volume 100mL
2ppm Standard #4		Direct pour
10ppm Standard #5	HP6409-500	Direct pour
250ppm Standard #6	HP4526-1L	500mL of 500ppm Std, 10% rinse to volume 1000mL
500ppm Standard #7		Direct pour
La Standard #8	VHG-PLAN-100	2.5mL of Stock La Std, 10% rinse to volume 250mL

Standards	Stock Std Cat #	Working Standard Preparation
ICV	ESC-8	5mL of Stock ICV Std, 10% rinse to volume 500mL
CCV	HP6929-1L	50mL of Stock CCV Std, 10% rinse to volume 1000mL
ICVLL	HP7013-500	50mL of Stock LL Std, 10% rinse to volume 1000mL
CCVLL		50mL of Stock LL Std, 10% rinse to volume 1000mL

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Standards	Stock Std Cat #	Working Standard Preparation
ICSA	ICL500-6	50mL of Stock A Std, 10% rinse to volume 500mL
ICSAB	ICL500-6,HP2739-500	50mL of Stock A Std, 5ml of Stock AB Std, 10% rinse to volume 500mL
CE	HP100010-1	5mL of Stock Ce Std, 10% rinse to volume 500mL
Internal Std	HP10M67-1,HP10M24-1	1mL of Stock Y, 3mL of Stock In, 10% rinse to volume 1000mL
10% Blank and Rinse	A200C-212	Fill container to 90% with DI water, add HNO ₃ to volume

7.6 Blanks - Three types of blanks are required for ICP-AES analysis

7.6.1 The calibration blank is used to establish the baseline for the instrument prior to the analysis of the analytical curve. The calibration blank is prepared by acidifying reagent water to the same acid concentration as used for the standards (10% HNO₃).

NOTE: The calibration blank must be stored in a FEP bottle to minimize leaching from other container materials that can cause an elevation in the target analytes leached causing an inherent bias in the calibration and quantitation of field samples when baselines are established prior to calibration of the ICP-AES.

7.6.1.1 Following calibration, the Initial Calibration Blank (ICB) is analyzed prior to field sample analyses. A Continuing Calibration Blank (CCB) is analyzed following the CCV after every ten samples and at the end of the analytical sequence to verify on-going acceptable instrument conditions.

7.6.2 The method blank is used to assess possible contamination from the sample preparation procedure. The method blank must contain all the reagents in the same volumes as used in sample preparation. The method blank must be prepared in the same manner as the samples including sample digestion, when applicable.

7.6.3 The rinse blank is prepared by acidifying reagent water to the same concentrations as the acids as used in the calibration blank (10% HNO₃). This solution is stored in a convenient manner. The rinse blank is used for equipment “wash out” to flush the sample delivery system and eliminate memory effects (carryover) from previous samples or standards.

7.7 Mixed Calibration Standard Solutions are used to make the following calibration solutions. All standards are prepared in Class A volumetric flasks using adjustable pipettes. The final acid concentration is matrix matched to digested field sample concentrations. See section 7.5 above for preparation of these standards.

Concentration of Target Analytes in Calibration Standards in mg/L

Analyte	STD 1	STD 2	STD 3	STD 4	STD 5	STD 6	STD 7	STD 8
Silver	0.005	0.5	1.0	2.0				

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Analyte	STD 1	STD 2	STD 3	STD 4	STD 5	STD 6	STD 7	STD 8
Aluminum	0.2				10	250	500	
Arsenic	0.01	0.5	1.0	2.0				
Boron	0.2		1.0	2.0				
Barium	0.005	0.5	1.0	2.0	10			
Beryllium	0.002	0.5	1.0	2.0				
Calcium	1.0		1.0	2.0	10	250	500	
Cadmium	0.002	0.5	1.0	2.0				
Cerium						10		
Cobalt	0.01	0.5	1.0	2.0				
Chromium	0.01	0.5	1.0	2.0				
Copper	0.01	0.5	1.0	2.0				
Iron	0.10	0.5	1.0	2.0	10	100	200	
Potassium	1.0			2.0	10	50	100	
Phosphorus	0.1	0.5	1.0	2.0				
Lanthanum								10
Lithium	0.015	0.5	1.0					
Magnesium	1.0				10	250	500	
Manganese	0.01	0.5	1.0	2.0				
Molybdenum	0.005	0.5	1.0	2.0				
Sodium	1.0		1.0	2.0	10	250	500	
Nickel	0.01	0.5	1.0	2.0				
Lead	0.005	0.5	1.0	2.0				
Antimony	0.01	0.5	1.0	2.0				
Selenium	0.01	0.5	1.0	2.0				
Silicon	0.2	0.5	1.0	2.0	10			
Strontium	0.01	0.5	1.0	2.0				
Sulfur	1.0		1.0	2.0	10	50	100	
Tin	0.05	0.5	1.0	2.0				
Thallium	0.01	0.5	1.0	2.0				
Vanadium	0.02	0.5	1.0	2.0				
Zinc	0.05	0.5	1.0	2.0				
Titanium	0.05	0.5	1.0	2.0				

- 7.8 Initial Calibration Verification (ICV) – The ICV is an analytical standard solution from a second source different from the calibration and CCV standards. The ICV is prepared at a mid-range concentration within the linear working range of the instrument. The ICV must have the same acid matrix as the Calibration Standards. See Section 7.5 above for the preparation of this standard.

All analytes are present in the mid-level ICV solution at the following concentrations (mg/L):

Analyte	Concentration
Silver	1.0
Aluminum	10.0
Arsenic	1.0
Boron	1.0
Barium	1.0

Analyte	Concentration
Manganese	1.0
Molybdenum	1.0
Sodium	10.0
Nickel	1.0
Lead	1.0

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Analyte	Concentration
Beryllium	1.0
Calcium	10.0
Cadmium	1.0
Cobalt	1.0
Chromium	1.0
Copper	1.0
Iron	10.0
Potassium	10.0
Lithium	1.0
Magnesium	10.0
Phosphorus	1.0

Analyte	Concentration
Antimony	1.0
Selenium	1.0
Silicon	1.0
Strontium	0.4
Tin	1.0
Thallium	1.0
Vanadium	1.0
Zinc	1.0
Titanium	1.0
Sulfur	10.0

- 7.9 Continuing Calibration Verification (CCV) – The CCV is the mid-range calibration standard prepared from the same source as the initial calibration curve. The CCV is used to verify the regression of the initial calibration of the instrument and must be repeated following every ten samples and at the conclusion of the sequence. EPA Method 200.7 refers to this standard as the Instrument Performance Check (IPC) standard. See Section 7.5 above for the preparation of this standard.

All analytes are present in the mid-level CCV solution at the following concentrations (mg/L):

Analyte	Concentration
Silver	0.5
Aluminum	10.0
Arsenic	1.0
Boron	1.0
Barium	0.50
Beryllium	0.20
Calcium	50.0
Cadmium	0.50
Cobalt	1.0
Chromium	1.0
Copper	1.0
Iron	10.0
Potassium	50.0
Lithium	1.0
Magnesium	10.0
Phosphorus	1.0

Analyte	Concentration
Manganese	1.0
Molybdenum	0.25
Sodium	50.0
Nickel	1.0
Lead	0.50
Antimony	0.5
Selenium	1.0
Silicon	2.0
Strontium	1.0
Tin	0.5
Thallium	1.0
Vanadium	1.0
Zinc	1.0
Titanium	1.0
Sulfur	5.0

- 7.10 Low Level Initial/Continuing Calibration Verification for EPA 6010C (ICVLL/CCVLL) –The ICVLL/CCVLL is prepared at a low concentration within the linear working range of the instrument and defines the lowest level of quantitation/reporting. The ICVLL/CCVLL must have the same acid matrix as the calibration standards. See Section 7.5 above for the preparation of this standard.

The concentration of the low-level ICVLL/CCVLL solution is listed in the table below:



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Analyte	Concentration (mg/L)
Silver	0.005
Aluminum	0.2
Arsenic	0.01
Boron	0.2
Barium	0.005
Beryllium	0.002
Calcium	1
Cadmium	0.002
Cobalt	0.01
Chromium	0.01
Copper	0.01
Iron	0.10
Potassium	1
Lithium	0.015
Magnesium	1
Phosphorus	0.1

Analyte	Concentration (mg/L)
Manganese	0.01
Molybdenum	0.005
Sodium	1
Nickel	0.01
Lead	0.005
Antimony	0.01
Selenium	0.01
Silicon	0.2
Strontium	0.01
Tin	0.05
Thallium	0.01
Vanadium	0.02
Zinc	0.05
Titanium	0.05
Sulfur	1.00

- 7.11 Interference Check Solutions (ICSA and ICSAB) – The ICSA and ICSAB are prepared to contain known concentrations of interfering elements that provides a test of the correction factors. The ICSA solution contains the interfering elements at a high concentration and the ICSAB contains both the interfering analytes at a high concentration and the analytes of interest at 0.5 to 1.0mg/L. EPA Method 200.7 and 6010D refers to this standard as the Spectral Interference Check (SIC) standard. See Section 7.5 above for the preparation of these standards.
- 7.11.1 The ICSA solution contains 5000mg/L of each Al, Ca, Mg and 2000mg/L Fe.
- 7.11.2 The ICSAB solution contains all the components at the same concentrations of the ICSA and other target analytes of interest spiked. In the working ICSAB solution, silver, boron, cadmium, nickel, lead, silica, and zinc are present at 1.0mg/L. All other analytes (arsenic, barium, beryllium, cobalt, chromium, copper, manganese, molybdenum, antimony, selenium, tin, thallium, vanadium and titanium) are present at 0.5mg/L.
- 7.12 Internal Standards – The internal standard response is used to measure the relative responses of other method analytes in each sample. Yttrium and Indium are used as internal standards. See Section 7.5 above for the preparation of this standard.
- 7.13 For the preparation of Laboratory Control Samples (LCSs) and Matrix Spikes (MSs) see the applicable sample preparation SOPs.



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8.0 PROCEDURE

8.1 Sample Analysis

8.1.1 Initializing the Instrument: Prior to daily calibration of the instrument, inspect the sample introduction system including the nebulizer, torch, injector tube for salt deposits, dirt, and debris that would restrict solution flow and affect instrument performance.

8.1.1.1 Replace the uptake tubing daily.

8.1.1.2 If any of the sample introduction parts appear soiled, first remove the part from the instrument by following the maintenance procedure in the instrument manual. Once removed, attempt to clean the part with a dilute solution of 5% nitric acid. Cleaning may be performed using a cotton swab or by submersing the part in the acid solution for no longer than five (5) minutes. If cleaning is successful, dry the part using compressed air or argon and replace it in the instrument. If cleaning does not adequately remove the residue, the part must be replaced with a new one in accordance with the manufacturer's directions. Replacement parts are kept in the cabinet in the instrument lab.

8.1.2 Instrument Stability: The instrument must be allowed to become thermally stable before calibration and analyses. This usually requires at least 30 minutes of operation.

8.1.3 Instrument Calibration: For initial and daily operation, calibrate the instrument according to the instrument manufacturer's recommended procedures using mixed calibration standard solutions and the calibration blank. A peristaltic pump is used to introduce all solutions, samples, and the internal standard to the nebulizer. To allow adequate time for equilibrium to be reached in the plasma, aspirate all solutions for at least 30 seconds after the solution reaches the plasma before obtaining the sample analyte response.

8.1.3.1 Use the average value from three replicate analyte responses per sample to be correlated to the overall analyte concentration in the solution being sampled. Flush the system with the rinse blank for a minimum of 60 seconds between each standard.

8.1.3.2 The calibration regression is generated using first order linear regression of a calibration blank and three calibration standards where each element is present. The blank is included as a point in the calibration curve to determine the baseline correction needed for the instrument to effectively quantitate target analyte concentrations.

8.1.3.3 Calibration acceptance criteria are described in section 10.4.

8.1.4 Internal Standard: All standards/samples/QC etc. contain yttrium and indium as the internal standards. The instrument adds the internal standards automatically. The instrument injects a constant volume into each solution being analyzed (i.e. standard, blank, field sample, LCS/LCSD/MS/MSD/DUP) and monitors the intensity at the sample level. An internal standard is the chosen alternative to the



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method of standard additions (MSA). If signal variation results from the sample introduction system (samples of different viscosity, matrix constitution), all the elements are corrected in the same way by an internal standard. If variation results from a variation of the energy transfer, the internal standard most accurately corrects elements of similar energy. Internal standard acceptance criteria are described in section 10.14.

- 8.1.5 **Calibration Accuracy:** Verify the acceptable initial calibration of the instrument using a standard source that is either an independent lot or entirely different manufacturer to ensure calibration accuracy. After calibrating and rinsing the instrument, analyze the ICV and, if analyzing samples using EPA 6010C, analyze the ICVLL standards. These standards are prepared as directed in section 7.7 and 7.9. Acceptance criteria are described in section 10.5.
- 8.1.6 **On-going Calibration Stability:** Verify the acceptable on-going instrument calibration by analyzing appropriate check standards during the sequence. Instrument calibration acceptability is demonstrated after every 10 samples using the CCV and CCB and at the end of the analytical run using the CCV, CCVLL (if analyzing EPA 6010C samples), and CCB that must meet the criteria described in sections 10.6 & 10.14.
- 8.1.7 **Accurate Background Corrections:** The interference check standards (ICSA and ICSAB) are used to verify the inter-element and background correction factors at the beginning and end of an analytical sequence or twice during every 8-hour work shift, whichever is more frequent. The interference check standards must meet the criteria found in section 10.10.
- 8.1.8 An Initial Calibration Blank (Section 7.5.1) is analyzed before sample analysis is initiated to verify the cleanliness of the analytical system. Acceptance criteria are described in section 10.14.
- 8.1.9 **Field Sample Analysis:** After completion of the above calibration requirements, samples must be analyzed in the same operational manner used in the calibration routine with the rinse blank also being used between all sample solutions, method blanks, Laboratory Control Standards, matrix spike, matrix spike duplicates, and check solutions.
- 8.1.10 **Dilutions:** If a sample analyte concentration is quantitated within 90% or greater of the upper limit of the analyte's determined Linear Dynamic Range (LDR), see section 10.4.2 for further guidance.

9.0 DATA ANALYSIS AND CALCULATIONS

- 9.1 Sample data should be reported in units of mg/L for aqueous samples and mg/kg dry weight corrected for solid samples.
- 9.2 For dissolved aqueous analytes, report the data generated directly from the instrument with compensation for sample dilution. Never report analyte concentrations below the MDL and if reporting between the MDL and the routine RL, results should be qualified with the appropriate indicator for estimated target analyte concentrations.

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- 9.3 For total recoverable aqueous analytes, multiply solution analyte concentrations by the dilution factor 0.5 when a 100mL aliquot is used to produce the 50mL final digestate volume, and report data. If a different aliquot volume other than 100mL is used for sample preparation, adjust the dilution factor accordingly. Account for any additional dilution of the prepared sample digestate required to complete the determination of any analytes exceeding 90% or greater of the LDR upper limit. Never report analyte concentrations below the MDL and if reporting between the MDL and the routine RL, results should be qualified with the appropriate indicator for estimated target analyte concentration. Routine reporting limits are adjusted for any dilution required by the sample analysis.
- 9.4 Results are reported to Three significant figures by the laboratory LIMS. Analyte concentrations for solids data should be rounded in a similar manner following dry weight corrections.
- 9.5 For total recoverable analytes in solid samples, calculate the target analyte concentration using the equation below and do not report analyte data below the estimated solids RL or an adjusted RL based on additional dilutions required to complete the analysis:

$$\text{Sample Conc. (mg/Kg) = } \frac{C \times V \times D}{W}$$

dry-weight basis

where: C = Concentration in extract (mg/L)
V = Volume of extract (L, 100mL = 0. 1L)
D = Dilution factor (undiluted = 1)
W = Weight in Kg of sample aliquot extracted (g x 0.001 = Kg)

- 9.6 Soil samples are routinely reported on a dry weight basis. Soil samples must be processed using the ENV-SOP-MTJL-0065, *Total Solids*. After a dry weight for each sample has been obtained, the calculations are performed automatically by the laboratory LIMS as follows:

$$\% \text{ solids (S)} = \frac{DW}{WW} \times 100$$

where: DW = Sample weight (g) dried
WW = Sample weight (g) before drying

- 9.7 Hardness calculations:
- Total Hardness, mg equivalent CaCO_3/L = 2.497 [Ca, mg/L] + 4.118 [Mg, mg/L]
Calcium Hardness = 2.497 [Ca, mg/L]
Magnesium Hardness = 4.118 [Mg, mg/L]
- 9.8 To calculate the silica concentration from silicon analysis:
- Silica (mg/L) = 2.14*[Silicon, mg/L]



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- 9.9 Formula needed to calculate dilution of stock standards of known concentration to a known final volume, using the basic chemistry formula, $C_1 \cdot V_1 = C_2 \cdot V_2$:

$$V_{\text{stock}} = V_{\text{std}} \cdot C_{\text{std}} / C_{\text{stock}}$$

where: V_{stock} = volume of stock standard required (mL)
 V_{std} = final volume of diluted standard required (mL)
 C_{stock} = concentration of stock standard required ($\mu\text{g/mL}$ or $\mu\text{g/L}$)
 C_{std} = final concentration of diluted standard required ($\mu\text{g/mL}$ or $\mu\text{g/L}$)

NOTE: Be sure to maintain consistent units for both concentration and volume during the use of the calculation and keep in mind that ($1\mu\text{g/mL} = 1\text{mg/L} = 1000\mu\text{g/L}$, $1\text{L} = 1000\text{mL} = 1000000\mu\text{L}$, and $1\text{mL} = 1000\mu\text{L}$)

- 9.10 Percent Relative Intensity (%RI) for internal standard assessment (ISTD):

$$\%RI = \text{Intensity of ISTD}_{\text{sample}} / \text{Intensity of ISTD}_{\text{CalBlk}} \cdot 100\%$$

- 9.11 Relative Standard Error (RSE – expressed as a percentage)

$$RSE = 100 \times \sqrt{\frac{\sum_{i=1}^n \left[\frac{x'_i - x_i}{x_i} \right]^2}{(n - p)}}$$

where:

x'_i = Measured amount of analyte at the calibration level i , in mass or concentration units
 x_i = True amount of analyte at calibration level i , in mass or concentration units
 p = Number of terms in the fitting equation (average – 1, linear = 2, quadratic = 3)

- 9.12 See the current Quality Assurance Manual for other equations associated with common calculations.

10.0 QUALITY CONTROL AND METHOD PERFORMANCE

- 10.1 All analysts must meet the qualifications specified in ENV-SOP-MTJL-0015, *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.

- 10.1.1 Prior to using Method 6010D for quantitation of samples, an initial demonstration of performance packet must be completed. This packet must document:

- The selection criteria for background correction points
- Analytical dynamic ranges including the applicable equations and upper limits of ranges
- IDLs and Method LLOQs



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- The determination and verification of interelement correction equations or other routines for correcting spectral interferences. These data must be generated using the same instrument, operating conditions, and calibration routine to be used for sample analysis. The data must be kept on file and available for review by the data user or auditor.
- 10.2 Use Prep Data to record batch order and standards/reagents used during analysis. See ENV-SOP-MTJL-0014, *Data Handling and Reporting*.
- 10.3 Batch Analyses
 - 10.3.1 Environmental Preparation Batches: Preparation batches are defined as sets of 1-20 samples as defined in Chapter 1 of SW-846 and in section 9.3.1 of EPA 200.7. Preparation batch analysis must include the following: 1 Method Blank, 1 Laboratory Control Sample (LCS), 1 Laboratory Control Sample Duplicate (LCSD), 1 Serial Dilution, 1 Sample Post Digestion Spike, 1 Matrix Spike/Spike Duplicate (MS/MSD) pair. All batch information is maintained in Prep Data computer program.
 - 10.3.2 Analytical Batches: Analytical batches are defined as a sequence of samples analyzed concurrently using the same calibrated instrument. Analytical batches include the QC samples produced in the Preparation Batches, in addition to: 1 Serial Dilution, 1 Sample Post Digestion Spike, 1 Initial Calibration Verification (ICV) following initial calibration, 1 Initial Calibration Verification-Low Level (ICVLL) following initial calibration (when analyzing EPA 6010C, 6010D and DOD only) 1 Initial Calibration Blank following the ICVLL, 1 Continuing Calibration Verification (CCV) following each 10 samples and at the conclusion of the sequence, 1 Continuing Calibration Verification-Low Level (CCVLL) at the conclusion of the sequence (when analyzing EPA 6010C only), 1 Continuing Calibration Blank (CCB) following each CCV, 1 Interference Check Sample A (ICSA) and 1 Interference Check Sample AB (ICSAB) following each initial calibration and at the end of the sequence or at least twice per each eight (8) hour shift. All batch information is maintained in Prep Data computer program.
- 10.4 Supporting Analytical Studies
 - 10.4.1 Instrument Detection Limits (IDL) Studies - IDLs in µg/L can be determined as the mean of the calibration blank results plus three times the standard deviation of 10 replicate analyses of the solution. Use zero for the mean if the mean is determined to be a negative value.

IDLs must be verified quarterly^{14,13} or when major instrumentation change occurs.
 - 10.4.2 Linear Dynamic Range (LDR) Studies – Linear dynamic ranges are established for each instrument to allow for quantitation above the highest level of calibration without qualification. ICP instruments are known to remain linear at high levels, but each upper limit of linearity is based on the target analyte being measured and the routine instrument operating conditions.

To perform a linear dynamic range study, the instrument must be calibrated normally as used with client field samples. The LDR is determined by the analysis of a minimum of three, but preferably five, different increasing



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concentrations of standards containing each target analyte across a range. One concentration should be near the expected upper linear range for each analyte. The highest concentration, where the instrument calibration remains linear, is determined when the observed concentration of the increasing standards is no more than 10% below the expected concentration of the analyte. If more than a 10% deviation exists, the instrument is proven to no longer be linear at that value for that analyte. The upper linear range is therefore the next lower concentration of standards used in the determination. Samples quantitated above that upper determined LDR require dilution to quantitate within the proven linear range of the instrument.

LDR studies must be verified semi-annually^{14.1} or when major instrumentation change occurs.

Method 6010D - LDR standards must be ran daily within ten percent of true value, or dilute all samples above the high standard in the curve.

STATE NOTE: For work performed in support of the NC Department of Natural Resources (15A NCAC 02H.0805(a)(7)(I)) for target analytes quantitated by ICP or ICPMS, a series of at least three standards must be analyzed along with each group of samples. The concentrations of these standards must bracket the concentration of the analytes in the field samples analyzed. Samples with target analyte concentrations above the highest level of calibration must be diluted to quantitate analytes within the calibration range. The use of the dynamic linear range studies to validate analyte/instrument calibration linearity must not be used for NC sample analysis.

- 10.4.3 Method Detection Limits – See also ENV-SOP-MTJL-0016, *Method Detection Limits (MDL), Limits of Detection (LOD) and Limits of Quantitation (LOQ)*.

MDL studies are required annually or when instrumentation change occurs. Method detection limit studies are performed on blank matrices most closely matching field sample matrices.

- 10.4.4 Inter-element Correction Factors – All inter-element spectral correction factors must be verified and updated every six months or when major instrumentation change occurs.^{14.1,14.5,}

Criteria for determining an inter-element spectral interference is an apparent positive or negative concentration of an analyte that is outside the 3-sigma control limits of the calibration blank for the analyte. See Attachment II for a listing of potential interfering analytes and their contributions from SW-846 method EPA 6010B. Testing is performed using 100mg/L single element solutions; however, for analytes such as iron that may be found at high concentration, a more appropriate test would be to use a concentration near the upper analytical range limit.

Suggested analytes that are known to commonly interfere include: Ag, Al, As, B, Ba, Be, Ca, Cd, Ce, Co, Cr, Cu, Fe, K, Li, Mg, Mn, Mo, Na, Ni, P, Pb, Sb, Se, Si, Sn, Sr, Ti, Tl, V, and Zn.



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10.4.5 Proficiency Testing (PT) – See also ENV-SOP-MTJL-0022, *Proficiency Testing Program*. Proficiency testing is performed in the metals department in support of both environmental and industrial hygiene analyses. Environmental PTs are performed semi-annually for Water Supply (Safe Drinking Water Act), Water Pollution (Clean Water Act), and soils (RCRA) testing.

10.5 Initial Calibration - Run a calibration curve on a daily basis that employs a minimum of a calibration blank and three standards for each target analyte. If the correlation coefficient does not meet the acceptance criteria, see the corrective action guidance listed in Section 11.1.

NOTE: For EPA Methods 200.7 & 6010B/D, the linear regression correlation coefficient for the each analyte in the calibration curve lines must be ≥ 0.995 .

NOTE: For Method 6010D - If a multipoint calibration is used the low standard must be at or below the LLOQ.

NOTE: For EPA Method 6010C, the regression correlation coefficient must be ≥ 0.998 .

10.6 Initial Calibration Verification (ICV/ICVLL) - Verify the accuracy of the initial instrument standardization by analyzing appropriate check standards following calibration. The routine mid-level ICV must be prepared from a source that is independent of the stock standard used for the preparation of the initial calibration curve. For EPA Method 6010C, a low-level ICV (ICVLL) is also performed as required; however the low level ICV is not required to be from a second source. It may be made from the same stock standard as the calibration standards as long as the initial calibration is verified by a second source in the mid-level ICV.

10.6.1 **EPA Method 6010B/D** - The routine ICV standard recovery results must be $\pm 10\%$ of the true value for EPA method 6010B/D. The RSD must be $< 5\%$ for the triplicate passes of the spectrometer. If the RSD exceeds 5% and/or the recovery exceeds 10%, locate and correct the cause of the problem and do not proceed until this criterion is met. Corrective actions for failures can be found in section 11.2.

10.6.2 **EPA Method 200.7** - The routine ICV standard recovery results must be $\pm 5\%$ of the true value for EPA method 200.7. The RSD must be within 3% for the four replicate passes of the spectrometer. If the RSD exceeds 3% and/or the recovery exceeds 5%, locate and correct the cause of the problem and do not proceed until this criterion is met. Corrective actions for failures can be found in section 11.2.

10.6.3 **EPA Method 6010C** - The routine ICV standard recovery results must be $\pm 10\%$ of the true value for EPA methods 6010C. The RSD must be $< 5\%$ for the triplicate passes of the spectrometer. The ICVLL standard recovery results should be within 70-130%. If the recovery does not meet the criteria for either level of ICV and/or the %RSD is exceeded, locate and correct the cause of the problem and do not proceed until this criterion is met. Corrective actions for failures can be found in section 11.2.

10.6.4 **EPA Method 6010D** Low-level Readback or Verification - For a multi-point calibration, the low level standard should quantitate to within 80-120% of the true value. For a single point calibration, a standard from the same source as the



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calibration standard and at the LLOQ is analyzed and should recover within 80-120% of the true value.

- 10.6.5 **EPA Method 6010D** Mid-level Readback or Verification - For a multi-point calibration, the midlevel standard should quantitate to within 90-110% of the true value. For a single point calibration, a standard from the same source as the calibration standard and at the midpoint of the linear range is analyzed and should recover within 90-110% of the true value.

- 10.7 Continuing Calibration Verification (CCV/CCVLL) - Verify the on-going instrument standardization by analyzing appropriate check standards during the sequence. Verification is achieved by analyzing both a CCV standard and a CCB (instrument blank). Continuing calibration verification standards can be created from either primary or secondary source standards from those used in instrument calibration. Continuing instrument calibration acceptability is demonstrated after every 10 samples and at the end of the analytical run using the CCV that must meet the following criteria per the method being analyzed:

- 10.7.1 **For SW-846 Method 6010B/D** – Continuing calibration verification (CCV) analyzed after every 10 samples and at the conclusion of the sequence must have a recovery within $\pm 10\%$. The RSD must be within 5% for the triplicate passes of the spectrometer. The CCVLL recovery should be within $\pm 50\%$.

- 10.7.2 **For EPA Method 200.7** - Continuing calibration verification (CCV) analyzed after every 10 samples and at the conclusion of the sequence must have a recovery within $\pm 10\%$. The RSD must be within 3% for the triplicate passes of the spectrometer.

- 10.7.3 **For SW-846 Method 6010C** - Continuing calibration verification (CCV) analyzed after every 10 samples and at the conclusion of the sequence must have a recovery within $\pm 10\%$. The RSD must be within 5% for the triplicate passes of the spectrometer. The CCVLL recovery should be within $\pm 30\%$. ICVLL are analyzed at the beginning of the analytical run and CCVLL are analyzed at the end of the analytical run for 6010C.

- 10.8 Method/Calibration/Rinse Blanks

- 10.8.1 Method Blank

- 10.8.1.1 A method blank is generated for each analytical batch during sample preparation to determine if any contamination is introduced during sample processing. A method blank is routinely a volume of reagent water that is carried through the entire digestion and analysis procedure with the samples.

- 10.8.1.2 The method blank must not contain analytes >MDL or, in the case of common laboratory contaminants, the concentrations should not exceed the RL. Common laboratory contaminants include: Calcium, Potassium, Magnesium, Zinc, Iron and Sodium. If target analytes are present in the method blank, corrective action must be taken. See section 11.5 for corrective actions.

NOTE: Per DoD QSM, version 5.0, Section 1.7.4.1, DoD/DOE require that method blanks be evaluated to $\frac{1}{2}$ RL (LOQ) for target



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analytes and RL (LOQ) for common laboratory contaminants. If contaminants are present in the blank above this level, samples must be re-prepared and re-analyzed or reported with appropriate qualification.

NOTE: Method 6010D – The method blank is considered to be acceptable if target analyte concentrations are less than $\frac{1}{2}$ the LLOQ or are less than project-specific requirements.

State Note: For Wisconsin samples, the method blank must not contain analytes more negative than the MDL value. If target analytes are more negative than the MDL, the instrument must be recalibrated or a new LOD study performed.

10.8.2 Calibration Blank

- 10.8.2.1 An initial calibration blank is generated for each analytical sequence using acidified reagent water. The CALBLK analyzed prior to the initial calibration standards is used to establish a baseline for the instrument prior to calibration. Great care is required during this analysis to ensure that the baseline is correctly established prior to the calibration of the instrument and the analysis of field samples. Inaccurate baselines established with contaminated calibration blanks degrade precision and accuracy of the analyses performed by creating biases in target analyte calibration.

If the initial calibration blank is grossly negative for a target analyte, then the quantitation of that target analyte in the calibration standards will be biased high due to over compensation by the instrument. This will lead to low recovery issues with the CCV, ICV, field samples, and batch QC samples. If target analytes are present in the CALBLK leading the instrument to make an over correction of the baseline for these targets, then the calibration curve will be biased high yielding a low bias for those target analytes in the CCV, ICV, field samples, and batch QC.

- 10.8.2.2 Continuing calibration blank (ICB/CCB) is also analyzed following each initial and continuing calibration verification standard within an instrument sequence to verify instrument stability and system cleanliness. This does not baseline correct the instrument for possible contaminants in the background and must be evaluated to ensure that background corrections are appropriate and consistently applied throughout the sequence.

The ICB must not contain analytes $> \frac{1}{2}$ RL, for CCB must not contain analytes $> RL$ for all 6010 methods or, in the case of common laboratory contaminants, the concentrations should not exceed the RL. Common laboratory contaminants include: Calcium, Potassium, Magnesium, Zinc, Iron and Sodium.

NOTE: West Virginia requires all blank results to be less than the MDL. If the concentration of the target analyte is above the MDL, then corrective action and reanalysis is required, or the data must be qualified as including a potential high bias.

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10.8.3 Rinse Blank

10.8.3.1 A rinse blank is utilized by the ICP to cleanse the system following the intake of each digestate analyzed. No data is obtained during this rinse and no applicable controls are required for this type of blank. This is merely cleansing the lines throughout the analytical system.

- 10.9 Matrix Spike/Matrix Spike Duplicate (MS/MSD) - A matrix/matrix spike duplicate must be prepared for each matrix for each batch of 10 samples for method 200.7 or 20 samples for method 6010B/C/D, where sufficient sample volume was submitted by the client. Matrix spike and matrix spike duplicate are prepared from a sample aliquot spiked with the known concentration of analytes.

The matrix spike recoveries must meet the criteria in the table below unless the analyte concentration in the sample is at least four (4) times greater than the spike concentration.

Method	Acceptance Criteria	
	Water	Soil
6010B, C, and D	75 – 125%	75 – 125%
200.7	70-130%	NA

Assess that the matrix spike duplicate precision (%RPD) results meet project-established goals acceptance criteria. If no project goals are specified, then results for the RPD must be less than 20%.

- 10.10 Laboratory Control Sample/Laboratory Control Sample Duplicate (LCS/LCSD) - An LCS/LCSD pair must be digested and analyzed with each batch of 20 samples.

10.10.1 **For SW-846 Method 6010B, 6010C, and 6010D** – The LCS recovery must be 80-120%. The RPD must be less than 20%. When using a certified solid reference material for paint chip analysis, the manufacturer's established limits are used for control limits

10.10.2 **For EPA Method 200.7** – The LCS recovery must be 85-115%. The RPD must be less than 20%.

- 10.11 Interference Check Standards (ICSA/ICSAB) – The ICSA and ICSAB must be analyzed at the beginning and ending of each sequence or twice within each eight (8) hour shift, whichever is more frequent. The recovery of the ICSA and ICSAB elements must be 80-120%. If the results are unsatisfactory, see section 11.10 for further guidance. Do not proceed until this criterion is met.

NOTE: The unspiked elements in the ICSA are not evaluated by the data capture software. The instrument analyst evaluates whether the unspiked elements are $\pm$ LLOQ for the ICSA.

10.11.1 For Method 6010D two types of SIC checks are used. Individual element SIC checks are performed when the instrument is initially setup, and periodically (at least once every six (6) months) thereafter. The mixed element SIC solution is used daily to check that the instrument is free from interference from elements

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typically observed in high concentration and to check that and interference corrections applied are still valid.

- 10.11.1.1 Single element interference checks - At a minimum, single element SIC checks must be performed for the following elements: Aluminum 500mg/L; Boron 50mg/L; Barium, 50mg/L; Calcium 500mg/L; Copper 20mg/L; Iron 200mg/L; Magnesium 500mg/L; Manganese 10mg/L; Molybdenum 20mg/L; Sodium 1000mg/L; Nickel 20mg/L; Selenium 20mg/L; Silicon 100mg/L; Tin 20mg/L; Vanadium 20mg/L; Zinc 10mg/L.

The absolute value of the concentration observed for any unspiked analyte in the single element SIC checks must be $\pm$ LLOQ. The concentration of the SIC checks are suggested, but become the highest concentration allowed in a sample analysis, and cannot be higher than the highest established linear range. Samples with concentrations of elements higher than the SIC check must be diluted until the concentration is less than the SIC check solution. Note that reanalysis of a diluted sample is required even if the high concentration element is not required to be reported for the specific sample, since the function of the SIC check is to evaluate spectral interferences on other elements.

The single element SIC checks are performed when the instrument is setup and periodically (at least once every six months) thereafter.

- 10.11.1.2 Mixed element interference check - The mixed element SIC solution (see section 7.10.3.2) is analyzed at least once per day, immediately after the initial calibration. The concentration measured for any target analytes must be less than $\pm$ LLOQ. If this criterion is not met then sample analysis may not proceed until the problem is corrected, or alternatively the LLOQ may be raised to the concentration observed in the SIC solution. The only exceptions are those elements that have been demonstrated to be contaminants in the SIC solutions (see Section 7.10.3.1). These may be present up to the concentration documented plus the LLOQ.

- 10.12 Serial Dilution (SD) - If the analyte concentration is sufficiently high (minimally 10X the IDL), an analysis of a 1:4 dilution must agree within 10% of the original determination. If not, see section 11.11 for further guidance.

For Method 6010D - If the analyte concentration is within the linear range of the instrument and sufficiently high (minimally, a factor of 25 times greater than the LLOQ), an analysis of a 1:5 dilution should agree to within $\pm$ 20% of the original determination.

- 10.13 Post digestion Spike (PS) - An analyte spike added to a portion of a prepared sample, or its dilution and must be recovered to within 75% to 125% of the known value. The spike addition should produce a minimum level of 10 times and a maximum of 100 times the IDL. If the spike is not recovered within the specified limits, see section 11.11 for further guidance.

For Methods 6010C and 6010D – The Post Digestion Spike must recover within $\pm$ 20% of the known value.

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- 10.14 Internal Standard (ISTD) – Verify the internal standard responses. The intensity of the internal standard response in a sample is monitored and compared to the intensity of the response for that internal standard in the calibration blank. The Percent Relative Intensity (%RI) in the sample must fall within 60-140% of the response in the calibration blank. If the %RI of the response in the sample falls outside of these limits, see sections 9.15 & 11.9 for further guidance.
- 10.15 Sample Dilution - If a sample analyte concentration is quantitated within 90% or greater of the upper limit of the analyte Linear Dynamic Range (LDR), the sample must be diluted with acidified reagent water and re-analyzed.
- 10.16 Lower Limit of Quantitation (LLOQ) – When analyzing samples according to Method 6010D, the LLOQ is initially verified by the analysis of at least seven (7) replicate samples, spiked at the LLOQ and processed through all preparation and analysis steps of the method. The mean recovery and relative standard deviation of these samples provide an initial statement of precision and accuracy at the LLOQ. In most cases the mean recovery should be +/- 35% of the true value and RSD should be < 20%. In-house limits may be calculated when sufficient data points exist. Monitoring recovery of LLOQ over time is useful for assessing precision and bias.
 - 10.16.1 Ongoing LLOQ verification, at a minimum, is on a quarterly basis to validate quantitation capability at low analyte concentration levels. This verification may be accomplished either with clean control material (e.g., reagent water, method blanks, Ottawa sand, diatomaceous earth, etc.) or a representative sample matrix (free of target compounds). Optimally, the LLOQ should be less than the desired regulatory action levels based on the stated project-specific requirements.

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.
- 11.2 All data must then undergo a second analyst review. This review must be performed according to ENV-SOP-MTJL-0014, *Data Handling and Reporting* and ENV-SOP-MTJL-0038, *Data Review*.
- 11.3 Initial Calibration – After analyzing the calibration standards, the curve is reviewed to ensure the acceptance criteria described in section 10.5 are met. If analytes do not meet this requirement, corrective action must be taken. Corrective actions may include re-calibrating the instrument, replacing the tubing on the peristaltic pump, examining blanks and standards for degradation/contamination, or performing instrument maintenance. If the internal standard responses in the calibration standards do not meet the criteria in section 10.14, re-calibrate the instrument.

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- 11.4 Initial Calibration Verification (ICV) – If the criteria described in section 10.6 are not met for a target analyte, re-analyze the ICV/ICVLL. If this fails a second time, corrective action must be taken. Re-calibrate and re-analyze the ICV/ICVLL using the same standard. If acceptance criteria are still not met, re-check standard curve and ICV/ICVLL preparation and/or perform routine instrument maintenance. If still not acceptable, refer to manufacturer's instruction or call service representative.
- 11.5 Continuing Calibration Verification (CCV) – The continuing calibration verification standard must agree with the criteria in section 10.7 or the CCV must be re-analyzed. If the recovery fails a second time, corrective action must be taken. The corrective action may require re-calibrate the instrument and re-analyze the last 10 samples, using the same CCV standard. If acceptance criteria are still not met, re-check the standard curve and CCV preparation and/or perform instrument maintenance. If the CCV still does not pass, refer to the manufacturer's instruction or call a service representative.
- 11.6 Blanks – Evaluate the blanks. The analyst must confirm that both the method blanks and the continuing calibration blanks were analyzed at the required frequency. Other items to check are as follows:
- 11.6.1 The instrument blank or continuing calibration blank (ICB/CCB) must meet the criteria in section 10.8. Corrective actions for method blank contamination include re-prepping the entire batch of samples, or if the site-specific requirements can be met, an elevated detection limit may be used, or the sample data qualified with a "B" qualifier and footnoted.
- NOTE:** Method 6010D - If the method blank fails to meet the necessary acceptance criteria, it should be reanalyzed once. If still unacceptable, then all samples associated with the method blank must be re-prepared and re-analyzed along with all other appropriate analysis batch QC samples. If the method blank results do not meet the acceptance criteria and reanalysis is not practical, then the laboratory should report the sample results along with the method blank results and provide a discussion of the potential impact of the contamination on the sample results. However, if an analyte of interest is found in a sample in the batch near its concentration confirmed in the blank, the presence and/or concentration of that analyte should be considered suspect and may require qualification.
- 11.7 Laboratory Control Sample (LCS)/Laboratory Control Sample Duplicate (LCSD) – Assess that LCS pairs were prepared at the required frequency. If all target analyte recoveries are not within the criteria described in section 10.10, rinse the instrument and re-analyze. If the LCS/LCSD fails for a second time, re-prepared all samples prepared in conjunction with the failing LCS. The affected samples must be re-digested and re-analyzed along with a new LCS. If there is insufficient volume submitted to re-prepare the field samples, notify the project manager to contact the client for further instruction. Reporting with a qualifier may be performed if acceptable to the client.
- 11.8 Matrix Spike (MS)/Matrix Spike Duplicate (MSD) – Assess that MS pairs were prepared at the required frequency. If all target analyte recoveries are not within the criteria described in section 10.9, review the post digestion spike results for similar failures. If the post spike confirms the matrix interferent, qualify the data using an O1 and J4. If the post spike does not confirm the interferent, rinse the instrument and re-analyze. If the

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MS/MSD fails for a second time, review the data to see if similar results are also present in the LCS/LCSD. If the LCS/LCSD are acceptable, the MS/MSD failures can be attributed to matrix interferences and the data can be qualified with a J4 and reported. If similar results are seen in the LCS, then re-prepared all samples prepared in conjunction with the failing MS/MSD. In this case, the affected samples must be re-digested and re-analyzed along with a new MS/MSD pair. If there is insufficient volume submitted to re-prepare the field samples, notify the project manager to contact the client for further instruction. Reporting with a qualifier may be performed if acceptable to the client. If insufficient field sample remains for re-analysis, report results with a J3 and an L3.

NOTE: If the sample concentration for an analyte is greater than four times (4x) the spike concentration, a “V” qualifier is used. The “V” qualifier indicates that the high concentration of analyte in the sample interfered with the ability to make an accurate spike recovery determination.

- 11.9 ISTD – The intensity of the Yttrium internal standard response in each sample is monitored and compared to the intensity of the response for that internal standard in the calibration blank. The Percent Relative Intensity (%RI) in each sample must meet the criteria in section 10.14. If the %RI of the response in the sample falls outside of these limits, the laboratory must immediately re-analyze the calibration blank and monitor the internal standard intensities. If the %RI for that calibration blank is within the limits, the laboratory must re-analyze the original sample at a two-fold dilution due to a possible interference from the matrix on the ISTD. If the %RI for the re-analyzed calibration blank is outside the limits, the analysis must be terminated, the problem corrected, the instrument recalibrated, the new calibration verified, and the samples reanalyzed.
- 11.10 Interference Check Standards (ICSA/ICSAB) - Evaluate the ICSA and ICSAB. The analyst must verify that the ICS and ICSAB have been analyzed at the required frequency. If the criteria in section 10.11 are not met, check the background correction protocols currently in place for appropriateness. If this is the initial ICS and/or ICSAB run after daily calibration, re-analyze the CALBLANK and re-calibrate the instrument. If the ICSA and/or ICSAB did not agree at the end of an 8-hour shift, re-analyze the ICSA and ICSAB. If failure persists, perform instrument maintenance as needed, recalibrate and re-analyze any samples in the previous run that may have been affected.
- 11.11 Serial Dilution/Post-digestion Spike – The analyst must verify that the SD and PS have been analyzed at the required frequency. If either of these tests fails to meet the required criteria in sections 10.12 & 10.13, the possibility of a matrix interferent should be suspected. An O1 qualifier is used when either sample type fails due to matrix interferences.
- 11.11.1 Serial Dilution - An analysis of a 1:4 dilution must agree within 10% of the original determination. If not, a chemical or physical interference effect is suspected.
- 11.11.2 Post-digestion Spike - The spike addition should produce a minimum level of 10 times and a maximum of 100 times the instrumental detection limit. If the spike is not recovered within the specified limits, a matrix effect is suspected.

CAUTION: If spectral overlap is suspected, use of computerized compensation, an alternate wavelength, or comparison with an alternate method is recommended.



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- 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, 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.
 - 11.12.2 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.
 - 11.12.3 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.
 - 11.12.4 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 J+ qualifier indicating the high bias with no impact on the field sample analysis due to the bias present.
 - 11.12.5 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.
 - 11.12.6 Sample results can be qualified and possible bias is narrated per the ENV-SOP-MTJL-0014, *Data Handling and Reporting*.
 - 11.12.7 Samples with multiple elements above the LDR must be diluted to verify interferences are not present

STATE NOTE: Drinking water samples analyzed using this procedure for compliance cannot be qualified.

12.0 POLLUTION PREVENTION AND WASTE MANAGEMENT

- 12.1 The EPA requires that a laboratory waste management practice 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 ENV-SOP-MTJL-0051, *Waste Management Plan*.
- 12.2 See ENV-SOP-MTJL-0046, *Environmental Sustainability & Pollution Prevention*.

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.



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13.3 Superscripts are provided where necessary to indicate the reference in Section 14.0 where the requirement/information can be found. Subscripts noted identify the most frequent/restrictive cases, but requirements may also be included at different frequencies/conditions in other references noted in section 14.0.

13.4 In the May 2012 Methods Update Rule, the EPA revised the previous interpretation of EPA 200.7 to include the use of axial torch orientation in the published method. Either axial or radial orientation is acceptable.

14.0 REFERENCES

- 14.1 *Inductively Coupled Plasma-Atomic Emission Spectrometry*, SW-846 Method 6010B, Revision 2, December 1996.
- 14.2 *Determination of Metals and Trace Elements in Water and Wastes by Inductively Coupled Plasma-Atomic Emission Spectrometry*, EPA Method 200.7, Revision 4.4, May 1994.
- 14.3 *Identification of Test Procedures*, 40 CFR §136.3.
- 14.4 *Inorganic Chemical Sampling and Analytical Requirements*, 40 CFR §141.23.
- 14.5 *Inductively Coupled Plasma-Atomic Emission Spectrometry*, SW-846 Method 6010C, Revision 3, February, 2007.
- 14.6 *Hardness by Calculation*, Standard Methods 2340B, 20th Edition.
- 14.7 *Hardness by Calculation*, Standard Methods 2340B, 2011.
- 14.8 *Hardness by Calculation*, Standard Methods 2340B, 1997.
- 14.9 *Inorganic Analytes*, SW-846 Chapter 3, Revision 4, February, 2007.

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Attachment I: Revision History**Current Version (Pace National):**

Date	Description of Revisions
2/4/2020	West Virginia audit response. Revised Section 10.8.2.2.

Superseded Versions (ESC Lab Sciences SOP#340386):

Version	Date	Description of Revisions
0	5/1/95	Origination
1	7/25/95	
2	3/11/97	
3	8/18/99	
4	2/11/00	
5	8/21/00	
6	3/28/01	
7	12/14/01	
8	4/11/03	
9	1/26/04	
10	8/2/04	
11	10/15/05	Corrected CCV criteria for EPA 200.7
12	10/29/08	Technical and Quality Review and update. Corrected acceptance criteria in Section 10.6. Updated format and re-organized sections 8.0, 10.0 and 11.0 based on new format.
13	1/23/09	Technical and Quality Review and update.
14	2/2/09	Technical and Quality Review and update. Clarification of holding times, Inclusion of cross-references. Inclusion of section 13.1 and section 7.1.
15	4/15/11	Technical and Quality Review and update. Added state notes where applicable; Added Tables 1.2b & 1.2c; Revised Table 1.2a and Sections 1.1, 1.3, 1.6, 1.11, 2.18, 2.22, 5.6, 7.1, 7.6.2, 7.9, 8.1.3, 8.1.5, through 8.1.10, 9.1, 9.7 through 9.12, 10.3, 10.0 & 11.0, 12.1; Added Sections 2.13.1, 2.14.1, 2.31 through 2.35, 3.1.1, 4.1, 4.7, 7.17, 13.2, 13.3, 14.5 through 14.10.
16	6/27/14	Complete Rewrite and update.
17	12/7/2015	Technical and quality review and update. Header and signature block re-formatting. Revised Sections 1.13.1, 2.13.1, 2.23, 5.1.3, 6.1.1, 6.1.2, 7.1, 7.6, 7.7, 7.8, 7.9, 7.10.1, 7.10.2, 7.11, 7.12, 7.13, 7.14, 7.16, 8.1.3, 8.1.5, 8.1.11, 9.4, 10.2, 10.3.1, 10.3.2, 10.4.2, 10.4.4, 10.9, 10.10, 10.14, 11.12.6, and 12.2. Revised Tables 1.2a, and 1.10, Deleted Sections 2.22 and 7.12. Added Attachment III.

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Version	Date	Description of Revisions
18	10/28/2016	Technical and quality review and update. Update per South Carolina DHEC correspondence of 6/24/16. Header and signature block re-formatting. Revised SOP title. Revised Sections 1.1, Table 1.2a, 1.3, 1.6, 1.10, 2.12, 2.15, 5.6, 7.5.2, 7.10.1, 9.1, 10.2, 10.3.1, 10.4.2, 10.4.3, 10.4.5, 10.5, 10.6.1, 10.6.2, 10.7.1, 10.8.1.2, 10.8.2.2, 10.10.1, 10.16, and Attachment III Table 2. Deleted Table 1.2b. Deleted Sections 2.2, 2.12, 2.13, 2.14, 2.15, 2.17, 2.20, through 2.33, 3.5, 4.8, 7.14, 7.16, 8.1.3, 9.7 through 9.10, 9.13, 10.4.5.1, 10.4.5.2, 10.4.5.3, 10.10.3, 13.5, 14.7, 14.8, 14.9, and 14.10. Added Sections 2.14, 7.10.3 and all subsections, 9.11, 9.12, 10.1.1, 10.6.4, 10.6.5, 10.8.1.2, 10.9.1 and all subsections, 10.11.1 and all subsections, and 11.6.1.
19	11/30/2017	Update in response to A2LA audit finding CAR2872. Changed ESC logo. Updated Sections 1.5, 3.1, 7.9, 10.11, 14.1, 14.2, 14.3, 14.4, 14.5, 14.6, 14.7, 14.8, 14.9, and Attachment III Table 5.
20	7/27/18	Update in response to WI and AZ audit findings. Changed logo and references to “ESC” to “Pace National”. Revised Section 7.14, 10.4.4, and 10.7.3. Added State Note to Sections 10.8.1.2 and 10.11. Also added 6010C note to Section 10.13
21	9/10/18	Update in response AZ audit finding CAR3296. Revised Post-Spike acceptance criteria to $\pm 20\%$ in Section 10.9.1.2

Superseded Versions (Pace National):

Date	Description of Revisions
1/27/2019	Technical and quality review and update. Deleted header, footer and signature. Revised sections 1.0, 1.3, 1.5, 1.6, 1.7, 1.13, 1.13.1, 2.8.1(2.7.1), 2.8.2(2.7.1), 4.2, 4.4, 4.7, 6.2, 6.3, 6.4, 6.5, 6.6, 7.1, 7.2, 7.3, 7.4, 7.5.1, 7.5.3, 7.6, 7.7, 7.8, 7.9, 7.10, 7.10.1, 7.10.2, 7.13, 9.5, 9.6, 9.9, 10.1, 10.2, 10.3, 10.4, 10.4.3, 10.4.5, 10.5, 10.6.3, 10.6.4, 10.6.5, 10.7.3, 10.8, 10.8.1, 10.8.1.2, 10.8.2, 10.8.3, 10.10.1, 10.11, 10.11.1.2, 10.12, 10.13, 10.16, 11.1, 11.2, 11.12.6, 12.1, 12.2, 14.2, 14.6, 14.7 and 14.8. Revised Table 1.10. Added section 7.5 and renumbered as necessary. Deleted sections 7.10.3, 7.10.3.1, 7.10.3.2, 7.11, 7.12, 7.14, 10.9.1, 10.9.1.1 and 10.9.1.2. Revised Attachment I. Revised Attachment II sections 4.10, 4.17 and 4.18.
5/7/2019	Technical and quality review and update. Revised sections 1.1, 1.2, 2.14, 8.1.1.2, 10.3.2, 10.9, 10.11, 10.11.1, 10.11.1.1, 10.11.1.2, 11.8, 11.12.6 and 12.1. Correctly numbered section 7.6.1.1.
6/19/2019	Added corporate header and footer. Revised based on LA DW Auditor's comments. Revised Sections 8.1.6, 10.3.2, and Attachment I.
10/16/2019	Revised section 1.2 and 10.7.1. Added section 11.12.7.

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Attachment II: Potential ICP interferences arising from analytes present in field samples at concentrations of 100mg/L

Analyte	Wavelength (nm)	Interferant ^{a,b}									
		Al	Ca	Cr	Cu	Fe	Mg	Mn	Ni	Ti	V
Aluminum	308.215	--	--	--	--	--	--	0.21	--	--	1.4
Antimony	206.833	0.47	--	2.9	--	0.08	--	--	--	0.25	0.45
Arsenic	193.696	1.3	--	0.44	--	--	--	--	--	--	1.1
Barium	455.403	--	--	--	--	--	--	--	--	--	--
Beryllium	313.042	--	--	--	--	--	--	--	--	0.04	0.05
Cadmium	226.502	--	--	--	--	0.03	--	--	0.02	--	--
Calcium	317.933	--	--	0.08	--	0.01	0.01	0.04	--	0.03	0.03
Chromium	267.716	--	--	--	--	0.003	--	0.04	--	--	0.04
Cobalt	228.616	--	--	0.03	--	0.005	--	--	0.03	0.15	--
Copper	324.754	--	--	--	--	0.003	--	--	--	0.05	0.02
Iron	259.940	--	--	--	--	--	--	0.12	--	--	--
Lead	220.353	0.17	--	--	--	--	--	--	--	--	--
Magnesium	279.079	--	0.02	0.11	--	0.13	--	0.25	--	0.07	0.12
Manganese	257.610	0.005	--	0.01	--	0.002	0.002	--	--	--	--
Molybdenum	202.030	0.05	--	--	--	0.03	--	--	--	--	--
Nickel	231.604	--	--	--	--	--	--	--	--	--	--
Selenium	196.026	0.23	--	--	--	0.09	--	--	--	--	--
Sodium	588.995	--	--	--	--	--	--	--	--	0.08	--
Thallium	190.864	0.30	--	--	--	--	--	--	--	--	--
Vanadium	292.402	--	--	0.05	--	0.005	--	--	--	0.02	--
Zinc	213.856	--	--	--	0.14	--	--	--	0.29	--	--

^a Dashes indicate that no interference was observed even when interferents were introduced at the following levels:

Al - 1000 mg/L	Mg - 1000 mg/L
Ca - 1000 mg/L	Mn - 200 mg/L
Cr - 200 mg/L	Ti - 200 mg/L
Cu - 200 mg/L	V - 200 mg/L
Fe - 1000 mg/L	

^b The figures recorded as analyte concentrations are not the actual observed concentrations; to obtain those figures, add the listed concentration to the interferant figure.

^c Interferences will be affected by background choice and other interferences may be present.

NOTE: Using the above table, if analyzing for Lead in a sample containing 1000mg/L Aluminum, the lead results could demonstrate a high bias of 0.17mg/L. (If the sample contained 10000mg/L of Al, the bias in lead could be 1.7mg/L), if background corrections are not accurately applied by the instrument.



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Attachment III: DoD Requirements

1.0 Equipment/Instrument Maintenance

Instrument maintenance must be performed routinely to optimize instrument performance and improve chromatography. Commonly performed maintenance includes changing pump tubing, replacing the torch, cleaning the nebulizer, etc. A new calibration curve must be analyzed following any major maintenance performed on the analytical system.

2.0 Computer Hardware and Software

QTegra, Version 2.4

3.0 Troubleshooting

Table 1. GC Troubleshooting Guide		
Problem	Cause	Treatment
Poor Precision	Nebulizer Pressure	Pressure should be about 0.15 mPa for aqueous solutions. If pressure is substantially higher, clean the nebulizer orifice or replace it entirely.
	Pooling in Spray Chamber	Usually caused by an oily film in the spray chamber. Aspirate 0.1% HF solution for about 20 seconds or 0.01% Triton X-100 solution.
	Center Tube	Replace the tube.
	Capillary Tubing	Air bubble migration through tubing should be smooth and consistent. Replace kinked/ pinched tubing.
	Peristaltic Pump	Adjust platen pressure. Check for leaks. Replace damaged pump.
Poor Accuracy	Pump Rate	Ensure the flush pump rate is the same as the analysis pump rate.
	Flush Time	Ensure proper time set for adequate rinse (typically 30 seconds).
Poor Detection Limits	Dirty Window or Mirror	Clean or replace dirty components.

4.0 Other Requirements

- 4.1 All hardcopy laboratory notebooks must be reviewed by the Supervisor, or their designee, on a monthly basis.
- 4.2 If not self-explanatory (e.g., a typo or transposed number), corrections to technical and quality records shall also include a justification for the change.
- 4.3 A person performing a manual integration must sign and date each manually integrated chromatogram and record the rationale for performing manual integration. Electronic signatures are acceptable.
- 4.4 The results of calibration and verification of support equipment must be within the specifications required of the application for which this equipment is used or the equipment must be removed from service until repaired. Calibration and verification records, including those of established correction

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factors, must be maintained. In the absence of method-specific requirements, the minimum requirements are as follows:

Table 2. Support Equipment Checks		
Performance Check	Frequency	Acceptance Criteria
Balance calibration check [Using two standard weights that bracket the expected mass]	Daily prior to use	Top-loading balance: $\pm 2\%$ or $\pm 0.02\text{g}$, whichever is greater Analytical balance: $\pm 0.1\%$ or $\pm 0.5\text{mg}$, whichever is greater
Verification of standard mass [Using weights traceable to the International System of Units (SI) through a NMI]	Every 5 years	Certificate of Calibration from ISO/IEC 17025 accredited calibration laboratory
Monitoring of refrigerator/freezer temperatures	Daily (i.e. 7 days per week) [use MIN/MAX thermometers or data loggers equipped with notification of out of control event capabilities if personnel not available to record daily]	Refrigerators: 0°C to 6°C Freezers: $\leq -10^{\circ}\text{C}$
Thermometer verification check [Using a thermometer traceable to the SI through an NMI] [Performed at two temperatures that bracket the target temperature(s). Assume linearity between the two bracketing temperatures.] [If only a single temperature is used, at the temperature of use]	Liquid in glass: Before first use and annually Electronic: Before first use and quarterly	Apply correction factors or replace thermometer
Volumetric labware	Class B: By lot before first use Class A and B: Upon evidence of deterioration	Bias: Mean within $\pm 2\%$ of nominal volume Precision: RSD $\leq 1\%$ of nominal volume (based on 10 replicate measurements)
Non-volumetric labware [Applicable only when used for measuring initial sample volume and final extract/ digestates volume]	By lot before first use or upon evidence of deterioration	Bias: Mean within $\pm 3\%$ of nominal volume Precision: RSD $\leq 3\%$ of nominal volume (based on 10 replicate measurements)
Mechanical volumetric pipette	Quarterly	Bias: Mean within $\pm 2\%$ of nominal volume Precision: RSD $\leq 1\%$ of nominal volume (based on minimum of 3 replicate measurements) [Note: for variable volume pipettes, the nominal volume is the volume of use]
Glass microliter syringe	Upon receipt and upon evidence of deterioration	General Certificate of Bias & Precision upon receipt Replace if deterioration is evident

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Table 2. Support Equipment Checks

Performance Check	Frequency	Acceptance Criteria
Drying oven temperature check	Daily prior to and after use	Within $\pm 5\%$ of set temperature
Water purification system	Daily prior to use	See method blank criteria given in Section 4.20 of this addendum

- 4.5 The expiration date of the prepared standard shall not exceed the expiration date of the primary standard. All containers must bear a preparation date.
- 4.6 To avoid preparing non-representative samples, the laboratory shall not “target” within a relatively small mass range (e.g., $1.00 \pm 0.01\text{g}$) because such targeting will produce non-representative subsamples if the sample has high heterogeneity. The laboratory shall not manipulate the sample material so the sample aliquot weighs exactly $1.00\text{g} \pm 0.01\text{g}$, as an example.
- 4.7 In the absence of project-specific requirements, the minimum standard data qualifiers to be used are:
- U Analyte was not detected and is reported as less than the LOD or as defined by the customer. The LOD has been adjusted for any dilution or concentration of the sample.
 - J The reported result is an estimated value (e.g., matrix interference was observed or the analyte was detected at a concentration outside the quantitation range).
 - B Blank contamination. The recorded result is associated with a contaminated blank.
 - N Non-target analyte. The analyte is a tentatively identified compound using mass spectrometry or any non-customer requested compounds that are tentatively identified.
 - Q One or more quality control criteria failed (e.g., LCS recovery, surrogate spike recovery, or CCV recovery).
- Additional data qualifiers may be used, or different letters or symbols to denote the qualifiers listed above, as long as they are appropriately defined and their use is consistent with project-specific requirements (e.g., QSM 5.0, the contract, and project-planning documents).
- 4.8 If the time of the sample collection is not provided, assume the most conservative time of day. For the purpose of batch processing, the start and stop dates and times of the batch preparation shall be recorded.
- 4.9 Each preparation method listed on the scope of accreditation must have quarterly LOD/LOQ verifications. However, not all possible combinations of preparation and cleanup techniques are required to have LOD/LOQ verifications. If LOD/LOQ verifications are not performed on all combinations, the laboratory must base the LOD/LOQ verifications on the worst case basis (preparation method with all applicable cleanup steps).
- 4.10 After each MDL determination, the laboratory must establish the LOD by spiking a quality system matrix at a concentration of at least 2 times but no greater than four times the MDL. This spike concentration establishes the LOD and the concentration at which the LOD shall be verified. It is specific to each suite of analyte, matrix, and method (including sample preparation). The following requirements apply to the initial LOD establishment and to the LOD verifications:

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- The apparent signal to noise (S/N) ratio at the LOD must be at least three and the results must meet all method requirements for analyte identification (e.g., ion abundance, second column confirmation, or pattern recognition). For data systems that do not provide a measure of noise, the signal produced by the verification sample must produce a result that is at least three standard deviations greater than the mean method blank concentration. This is initially estimated based on a minimum of four method blank analyses and later established with a minimum of 20 method blank results.
 - If the LOD verification fails, then the laboratory must repeat the MDL determination and LOD verification or perform and pass two consecutive LOD verifications at a higher spike concentration and set the LOD at the higher concentration.
 - The laboratory shall maintain documentation for all MDL determinations and LOD verifications.
 - The DL and LOD must be reported for all analyte-matrix-methods suites, unless it is not applicable to the test or specifically excluded by project requirements.
- 4.11 The LOD shall be verified quarterly. In situations where methods are setup and used on an infrequent basis, the laboratory may choose to perform LOD verifications on a one per batch basis. All verification data will be in compliance, reported, and available for review.
- 4.12 For DoD, at a minimum, the LOQ shall be verified quarterly. In situations where methods are setup and used on an infrequent basis, the laboratory may choose to perform LOQ verifications on a one per batch basis.
- 4.13 All initial instrument calibrations must be verified with a standard obtained from a second manufacturer prior to analyzing any samples. The use of a standard from a second lot obtained from the same manufacturer (independently prepared from different source materials) is acceptable for use as a second source standard. The concentration of the second source standard shall be at or near the midpoint of the calibration range. The acceptance criteria for the initial calibration verification must be at least as stringent as those for the continuing calibration verification.
- 4.14 Exclusion of calibration points without documented scientifically valid technical justification is not permitted.
- 4.15 The concentration of the CCV standard shall be greater than the low calibration standard and less than or equal to the midpoint of the calibration range.
- 4.16 All CCVs analyzed must be evaluated and reported. If a CCV fails, reanalysis or corrective actions must be taken.
- If a CCV fails, the laboratory can immediately analyze two additional consecutive CCVs (immediately is defined as starting a consecutive pair within one hour; no samples can be run between the failed CCV and the two additional CCVs). This approach allows for spurious failures of analytes to be reported without reanalysis of samples. Any corrective actions that change the dynamics of the system (e.g., clip column, clean injection port, run blanks) requires that all samples since the last acceptable CCV be reanalyzed.
 - Both of these CCVs must meet acceptance criteria in order for the samples to be reported without reanalysis.

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- If either of these two CCVs fail or if the laboratory cannot immediately analyze two CCVs, the associated samples cannot be reported and must be reanalyzed.
 - Corrective action(s) and recalibration must occur if the above scenario fails. All affected samples since the last acceptable CCV must be reanalyzed.
 - Flagging of data for a failed CCV is only appropriate when the affected samples cannot be reanalyzed. The laboratory must notify the client prior to reporting data associated with a failed CCV.
- 4.17 The results of all MS/MSDs must be evaluated using the same acceptance criteria used for the DoD LCS limits (see Addendum Tables 3 and 4) or project limits, if specified. If the specific analyte(s) are not available in the Addendum Tables 3 and 4, the laboratory shall use their LCS in-house limits (see the LIMS) as a means of evaluating MS/MSDs. The MS and MSD must be spiked with all reported analytes.
- 4.18 Surrogate spike results shall be compared with DoD LCS limits (see Addendum Tables 3 and 4) or acceptance criteria specified by the client. If these criteria are not available, the laboratory shall compare the results with its in-house statistically established LCS criteria (see the LIMS).
- 4.19 The method blank shall be considered to be contaminated if:
- The concentration of any target analyte (chemical of concern) in the blank exceeds 1/2 the LOQ and is greater than 1/10th the amount measured in any associated sample, or 1/0th the regulatory limit, whichever is greater;
 - The concentration of any common laboratory contaminant in the blank exceeds the LOQ;
 - If a method blank is contaminated as described above, then the laboratory shall reprocess affected samples in a subsequent preparation batch, except when sample results are below the LOD. If insufficient sample volume remains for reprocessing, the results shall be reported with appropriate data qualifiers.
- 4.20 Sporadic Marginal Exceedances are not allowed for target analytes (chemicals of concern as identified by a project) without project-specific approval. Target analytes are considered those few analytes that are critical for the success of a project (such as risk drivers) where sporadic marginal exceedances cannot be allowed. Laboratories should consult with clients whenever long lists of analytes are requested for analysis to determine if marginal exceedances will not be allowed.
- 4.21 DoD considers the same analyte exceeding the LCS control limit two (2) out of three (3) consecutive LCS to be indicative of non-random behavior, which requires corrective action and reanalysis of the LCS.



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Table 3. LCS Control Limits – Method 6010 Solid Matrix

CAS ID	Analyte	N Records	Mean	Standard Deviation	Lower Control Limit	Upper Control Limit
7429-90-5	Aluminum	6258	96.7	7.5	74	119
7440-36-0	Antimony	5997	96.4	5.7	79	114
7440-38-2	Arsenic	9530	96.2	4.9	82	111
7440-39-3	Barium	9236	98.3	5	83	113
7440-41-7	Beryllium	6799	97.8	5.1	83	113
7440-42-8	Boron	2312	93	7.1	72	114
7440-43-9	Cadmium	9466	97.5	5.3	82	113
7440-70-2	Calcium	6347	98.1	5.8	81	116
7440-47-3	Chromium	9598	98.9	4.6	85	113
7440-48-4	Cobalt	6725	98.7	4.5	85	112
7440-50-8	Copper	7839	99.1	6	81	117
7439-89-6	Iron	5746	99.7	6.1	81	118
7439-92-1	Lead	10160	96.8	5.1	81	112
7439-93-2	Lithium	551	98.8	4.5	85	112
7439-95-4	Magnesium	6283	96.1	6.1	78	115
7439-96-5	Manganese	6732	99.1	4.9	84	114
7439-98-7	Molybdenum	4424	98.7	5.7	82	116
7440-02-0	Nickel	7412	98.1	4.9	83	113
7723-14-0	Phosphorus	189	103.1	3.8	92	114
7440-09-7	Potassium	6574	98.3	5.8	81	116
7782-49-2	Selenium	8862	94.5	5.6	78	111
7440-22-4	Silver	9105	97.3	5	82	112
7440-23-5	Sodium	5825	100.1	5.8	83	118
7440-24-6	Strontium	2573	98.5	5	83	114
7440-28-0	Thallium	6416	96.8	4.6	83	111
7440-31-5	Tin	2780	100.1	6.6	80	120
7440-32-6	Titanium	2107	98.2	5.2	83	114
7440-61-1	Uranium	109	97.4	5.2	82	113
7440-62-2	Vanadium	6934	98.3	5.4	82	114
7440-66-6	Zinc	7882	97.4	5	82	113

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Table 4. LCS Control Limits – Method 6010 Water Matrix

CAS ID	Analyte	N Records	Mean	Standard Deviation	Lower Control Limit	Upper Control Limit
7429-90-5	Aluminum	11532	100	4.8	86	115
7440-36-0	Antimony	10737	100.2	4.2	88	113
7440-38-2	Arsenic	14123	99.9	4.3	87	113
7440-39-3	Barium	14476	100.3	4.1	88	113
7440-41-7	Beryllium	11552	100.4	4	89	112
7440-69-9	Bismuth	147	95.8	3.2	86	105
7440-42-8	Boron	3871	98.8	4.8	85	113
7440-43-9	Cadmium	13922	100.8	4.1	88	113
7440-70-2	Calcium	11382	100	4.2	87	113
7440-47-3	Chromium	15027	101.1	3.9	90	113
7440-48-4	Cobalt	11824	101.2	4.2	89	114
7440-50-8	Copper	12910	100.2	4.6	86	114
7439-89-6	Iron	13797	100.7	4.7	87	115
7439-92-1	Lead	14391	99.3	4.4	86	113
7439-93-2	Lithium	938	100.7	5.3	85	117
7439-95-4	Magnesium	11423	98.8	4.8	85	113
7439-96-5	Manganese	12767	101.9	4.1	90	114
7439-98-7	Molybdenum	8251	101.1	4	89	113
7440-02-0	Nickel	12699	100.5	4.1	88	113
7440-05-3	Palladium	492	99.8	4	88	112
7723-14-0	Phosphorus	203	100.5	4.2	88	113
7440-09-7	Potassium	11006	99.9	4.7	86	114
7782-49-2	Selenium	13264	98.5	5.2	83	114
7440-21-3	Silicon	1525	100.6	6.1	82	119
7440-22-4	Silver	13770	99.1	5.1	84	115
7440-23-5	Sodium	10893	100.9	4.7	87	115
7440-24-6	Strontium	3782	101.3	3.8	90	113
7704-34-9	Sulfur	145	100.7	3.9	89	112
7440-28-0	Thallium	10063	99.5	4.7	85	114
7440-31-5	Tin	4502	101.3	4.4	88	115
7440-32-6	Titanium	5625	101.1	3.4	91	111
7440-61-1	Uranium	223	101.3	5.8	84	119
7440-62-2	Vanadium	12032	100.2	3.6	90	111
7440-66-6	Zinc	13549	100.6	4.6	87	115

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Table 5. Quality Control Requirements – Inorganic Analysis by Inductively Coupled Plasma Atomic Emission Spectrometry (ICP/AES)

QC Check	Minimum Frequency	Acceptance Criteria	Corrective Action	Flagging Criteria	Comments
Linear Dynamic Range (LDR) or high-level check standard	At initial set up and checked every 6 months with a high standard at the upper limit of the range.	Within $\pm 10\%$ of true value.	Dilute samples within the calibration range, or re-establish/ verify the LDR.	Flagging is not appropriate.	Data cannot be reported above the high calibration range without an established/passing high-level check standard.
Initial Calibration (ICAL) for all analytes	Daily ICAL prior to sample analysis.	If more than one calibration standard is used, $r^2 \geq 0.99$.	Correct problem, then repeat ICAL.	Flagging is not appropriate.	Minimum one high standard and a calibration blank. No samples shall be analyzed until ICAL has passed.
Initial Calibration Verification (ICV)	Once after each ICAL, analysis of a second source standard prior to sample analysis.	All reported analytes within $\pm 10\%$ of true value.	Correct problem. Rerun ICV. If that fails, repeat ICAL.	Flagging is not appropriate.	No samples shall be analyzed until calibration has been verified with a second source.

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Table 5. Quality Control Requirements – Inorganic Analysis by Inductively Coupled Plasma Atomic Emission Spectrometry (ICP/AES)

QC Check	Minimum Frequency	Acceptance Criteria	Corrective Action	Flagging Criteria	Comments
Continuing Calibration Verification (CCV)	After every 10 field samples, and at the end of the analysis sequence.	All reported analytes within $\pm 10\%$ of the true value.	Immediately analyze two additional consecutive CCVs. If both pass, samples may be reported without reanalysis. If either fails or if two consecutive CCVs cannot be run, perform corrective action(s) and repeat CCV and all associated samples since the last successful CCV. Alternately, recalibrate if necessary; then reanalyze all associated samples since the last acceptable CCV	If reanalysis cannot be performed, data must be qualified and explained in the case narrative. Apply Q-flag to all results for the specific analyte(s) in all samples since the last acceptable calibration verification.	Results may not be reported without a valid CCV. Flagging is only appropriate in cases where the samples cannot be reanalyzed.

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Table 5. Quality Control Requirements – Inorganic Analysis by Inductively Coupled Plasma Atomic Emission Spectrometry (ICP/AES)

QC Check	Minimum Frequency	Acceptance Criteria	Corrective Action	Flagging Criteria	Comments
Low-Level Calibration Check Standard (LLCCV)	Daily	All reported analytes within $\pm 20\%$ of true value.	Correct problem and repeat ICAL.	Flagging is not appropriate.	No samples shall be analyzed without a valid low-level calibration check standard (LLCCV). Low-level calibration check standard should be less than or equal to the LOQ. If the concentration of the lowest calibration standard is less than or equal to the LOQ, the lowest standard may be re-quantified against the calibration curve as a LLCCV. Otherwise, a separate standard must be analyzed as LLCCV prior to the analysis of any samples.

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TITLE: ENV-SOP-MTJL-0215 Determination Metals and Trace Elements in Various Matrices by ICP-AES (EPA Methods 6010B, 6010C, 6010D [ICP-OES], and 200.7) Including Hardness (EPA Methods 200.7 and 6010B/C/D and SM 2340B)

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Table 5. Quality Control Requirements – Inorganic Analysis by Inductively Coupled Plasma Atomic Emission Spectrometry (ICP/AES)

QC Check	Minimum Frequency	Acceptance Criteria	Corrective Action	Flagging Criteria	Comments
Method Blank (MB)	One per preparatory batch.	The absolute values of all analytes must be $< \frac{1}{2}$ LOQ or $< 1/10^{\text{th}}$ the amount measured in any sample or $1/10$ the regulatory limit, whichever is greater.	Correct problem. If required, reprep and reanalyze MB and all QC and field samples processed with the contaminated blank.	If reanalysis cannot be performed, data must be qualified and explained in the case narrative. Apply B-flag to all results for the specific analyte(s) in all samples in the associated preparatory batch.	Results may not be reported without a valid method blank. Non-detects associated with positive blank infractions may be reported. Sample results $>10X$ the LOQ associated with negative blanks may be reported. Flagging is only appropriate in cases where the samples cannot be reanalyzed.
Initial and Continuing Calibration Blank (ICB/CCB)	Immediately after the ICV and immediately after every CCV.	The absolute values of all analytes must be $< \frac{1}{2}$ LOQ or $< 1/10^{\text{th}}$ the amount measured in any sample.	ICB: Correct problem and repeat ICV/ICB analysis. If that fails, rerun ICAL. All samples following the last acceptable Calibration Blank must be reanalyzed. CCBs may not be reanalyzed without reanalysis of the associated samples and CCV(s).	Flagging is not appropriate.	Results may not be reported without a valid calibration blank. Non-detects associated with positive blank infractions may be reported. Sample results $>10X$ the LOQ associated with negative blanks may be reported. For CCB, failures due to carryover may not require an ICAL.

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Table 5. Quality Control Requirements – Inorganic Analysis by Inductively Coupled Plasma Atomic Emission Spectrometry (ICP/AES)

QC Check	Minimum Frequency	Acceptance Criteria	Corrective Action	Flagging Criteria	Comments
Interference Check Solutions (ICS) (also called Spectral Interference Checks)	After ICAL and prior to sample analysis.	ICS-A: Absolute value of concentration for all non-spiked project analytes <1/2 LOQ (unless they are a verified trace impurity from one of the spiked analytes); ICS-AB: Within $\pm$ 20% of true value.	Terminate analysis; locate and correct problem; reanalyze ICS, reanalyze all samples.	If corrective action fails, apply Q-flag to all results for specific analyte(s) in all samples associated with the failed ICS.	All analytes must be within the LDR. ICS-AB is not needed if instrument can read negative responses.
Laboratory Control Sample (LCS)	One per preparatory batch.	A laboratory must use Table 3 and 4 limits for batch control if project limits are not specified. If the analyte(s) are not listed, use in-house LCS limits if project limits are not specified.	Correct problem, then reprep and reanalyze the LCS and all samples in the associated preparatory batch for failed analytes, if sufficient sample material is available.	If reanalysis cannot be performed, data must be qualified and explained in the case narrative. Apply Q-flag to specific analyte(s) in all samples in the associated preparatory batch.	Must contain all reported analytes. Results may not be reported without a valid LCS. Flagging is only appropriate in cases where the samples cannot be reanalyzed.
Matrix Spike (MS)	One per preparatory batch.	A laboratory must use Table 3 and 4 limits for batch control if project limits are not specified. If the analyte(s) are not listed, use in-house LCS limits if project limits are not specified.	Examine the project-specific requirements. Contact the client as to additional measures to be taken.	For the specific analyte(s) in the parent sample, apply J flag if acceptance criteria are not met and explain in the case narrative.	For matrix evaluation only. If MS results are outside the limits, the data shall be evaluated to the source(s) of difference (i.e., matrix effect or analytical error).

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Table 5. Quality Control Requirements – Inorganic Analysis by Inductively Coupled Plasma Atomic Emission Spectrometry (ICP/AES)

QC Check	Minimum Frequency	Acceptance Criteria	Corrective Action	Flagging Criteria	Comments
Matrix Spike Duplicate (MSD) or Matrix Duplicate (MD)	One per preparatory batch.	A laboratory must use Table 3 and 4 limits for batch control if project limits are not specified. If the analyte(s) are not listed, use in-house LCS limits if project limits are not specified. MSD or MD: RPD of all analytes $\leq 20\%$ (between MS and MSD or sample and MD).	Examine the project-specific requirements. Contact the client as to additional measures to be taken.	For the specific analyte(s) in the parent sample, apply J flag if acceptance criteria are not met and explain in the case narrative.	The data shall be evaluated to determine the source of difference. For Sample/MD: RPD criteria only apply to analytes whose concentration in the sample is greater than or equal to the LOQ.
Dilution Test	One per preparatory batch if MS or MSD fails.	Five-fold dilution must agree within $\pm 10\%$ of the original measurement.	No specific CA, unless required by the project.	For the specific analyte(s) in the parent sample, apply J flag if acceptance criteria are not met and explain in the case narrative.	Only applicable for samples with concentrations $> 50 \times$ LOQ (prior to dilution). Use along with MS/MSD and PDS data to confirm matrix effects.
Post-Digestion Spike (PDS) Addition (ICP only)	Perform if MS/MSD fails. One per preparatory batch (using the same sample as used for the MS/MSD if possible).	Recovery within 80-120%.	No specific CA, unless required by the project.	For the specific analyte(s) in the parent sample, apply J flag if acceptance criteria are not met and explain in the case narrative.	Criteria applies for samples with concentrations $< 50 \times$ LOQ prior to dilution.

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Table 5. Quality Control Requirements – Inorganic Analysis by Inductively Coupled Plasma Atomic Emission Spectrometry (ICP/AES)

QC Check	Minimum Frequency	Acceptance Criteria	Corrective Action	Flagging Criteria	Comments
Method of Standard Additions (MSA)	When dilution test or post digestion spike fails and if required by project.	NA	NA	NA	Document use of MSA in the case narrative.

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