# RIGHT-OF-WAY AND SITE PERIMETER SOIL VAPOR AND GROUNDWATER EVALUATION WORK PLAN

**245-265 & 271 HOLLENBECK STREET AND 50 BALFOUR DRIVE**
**ROCHESTER, NEW YORK**

**NYSDEC SITE #828188**


**Prepared for:** OBI, LLC
Rochester, New York

**Prepared by:** Day Environmental, Inc.
1563 Lyell Ave
Rochester, New York 14606



**Project No.:** 4845S-13

**Date:** January 2014

**New York State Department of Environmental Conservation**
**Division of Environmental Remediation, Region 8**
6274 East Avon-Lima Road, Avon, New York 14414-9519
**Phone: (**585) 226-5353 • **Fax: (**585) 226-8139
**Website:** www.dec.ny.gov

Joe Martens
Commissioner

January 9, 2014

Mr. Mike McAlpin
OBI, LLC
255 Hollenbeck Street
Rochester, New York 14621

Dear Mr. McAlpin;

**Re: McAlpin Industries Site #828188**
**Right-of-Way and Site Perimeter Soil Vapor and Groundwater Evaluation Work Plan; January 2014**
**245-265 & 271 Hollenbeck Street and 50 Balfour Drive**
**City of Rochester, Monroe County**

The New York State Departments of Environmental Conservation (NYSDEC) and Health (NYSDOH), collectively referred to as the Departments, have completed their review of the documents entitled "*Right-of-Way and Site Perimeter Soil Vapor and Groundwater Evaluation Work Plan"* and "*Health and Safety Plan"* (collectively referred to as the Work Plan) dated January 2014 and prepared by Day Environmental, Inc for the McAlpin Industries site in the City of Rochester, Monroe County. Based on the information and representations provided in the Work Plan, the Departments have determined that the Work Plan substantially addresses the requirements of the Order on Consent and the Work Plan is hereby approved. With the exception of the Community Air Monitoring Plan, this approval does not extend to the Health and Safety Plan as the Departments are not responsible for the health and safety of remediation workers.

Prior to the start of field work, please attach this letter to the Work Plan and distribute as follows:

- Frank Sowers (NYSDEC, Avon) - 2 hard copies;
- Jacquelyn Nealon (NYSDOH, Albany) – 1 hard copy;
- John Frazer (MCHD) - 1 electronic copy; and
- Lincoln Branch Library - 1 hard copy.

The hard copies should be submitted double-sided.

Based on the schedule in the approved Work Plan, field activities are scheduled to begin by February 10, 2014. Please notify me at least 7 days in advance of the start of field activities.

This letter represents approval of the first work plan under the Order on Consent and initiates the monthly Progress Report provision of the Order. Progress Reports are due by the 10th day of each month with the initial Progress Report due on February 10, 2014. Progress Reports should be submitted as electronic (.pdf) files only.

Additionally, please use “McAlpin Industries” as the site name in future correspondence including work plans, reports, letters, and emails.

Thank you for your cooperation in this matter and please contact me at (585) 226-5357 if you have any questions.

Sincerely,

Frank Sowers, P.E.
Environmental Engineer II

ec:
B. Putzig
J. Mahoney
J. Nealon
R. Palumbo
R. Kampff
N. Simon
J. Frazer

**RIGHT-OF-WAY AND SITE PERIMETER SOIL VAPOR AND GROUNDWATER EVALUATION WORK PLAN**

**245-265 & 271 HOLLENBECK STREET AND 50 BALFOUR DRIVE**
**ROCHESTER, NEW YORK**

**NYSDEC SITE #828188**

I, Nathan E. Simon certify that I am currently a NYS registered professional engineer and that this Right-of-Way and Site Perimeter Soil Vapor and Groundwater Evaluation Work Plan was prepared in accordance with applicable statutes and regulations and in substantial conformance with DER Technical Guidance for Site Investigation and Remediation (DER-10).


Nathan E. Simon, P.E.
Project Manager (NYS P.E. License #087172)
Day Environmental, Inc.

Raymond L. Kampff
Associate Principal
Day Environmental, Inc.

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## 1.0 INTRODUCTION

Day Environmental, Inc. (DAY) prepared this *Right-of-Way and Site Perimeter Soil Vapor and Groundwater Evaluation Work Plan* (the Work Plan) on behalf of OBI, LLC. The Work Plan describes studies proposed as part of the remedial investigation (RI) of the site located at 245-265 & 271 Hollenbeck Street and 50 Balfour Drive, Rochester, New York (Site) and the surrounding neighborhood to evaluate volatile organic compound (VOC) impact within the soil vapor and groundwater. A Project Locus Map is included as Figure 1.

The Site is zoned for industrial use, and it is located in proximity to residential properties and other properties zoned for industrial and commercial use. The Site Plan presented as Figure 2 identifies the locations of the proposed groundwater/soil vapor locations to be completed as part of the studies described in the Work Plan. In addition, the locations of other industrial/commercial facilites in the area of the Site are depicted on Figure 2.

## 1.1 BACKGROUND

Previous studies were completed that included the installation of groundwater monitoring wells in various locations on the Site and the subsequent testing of samples collected from these monitoring wells. The analytical laboratory results indicated that the overburden groundwater in northern central portion of the Site contains halogenated VOCs (typically tetrachloroethene, trichloroethene, cis-1,2-dichloroethene, and/or vinyl chloride). Samples collected from some of the monitoring wells, including monitoring wells positioned in hydraulically downgradient positions in proximity of the boundary of the Site, contained concentrations of halogenated VOCs at concentrations exceeding the New York State Department of Environmental Conservation (NYSDEC) applicable groundwater standards and guidance values. A Site Plan showing the location of the monitoring wells currently installed at the Site and groundwater contours determined using static water level measurements made on December 7, 2012 is attached as Figure 3. A historic summary of the halogentated VOCs measured in samples collected from the monitoring wells currently installed at the Site is presented on Table 1 *Summary of Analytical Laboratory Results: Groundwater Samples.*

## 1.2 APPLICABLE PROJECT STANDARDS, CRITERIA AND GUIDANCE

The requirements, applicable standards, criteria and guidance documents that will be used for this project are outlined below:

- Unrestricted Use, Restricted Residential Use, Restricted Commercial Use, Restricted Industrial Use, Protection of Ecological Resources and Protection of Groundwater Soil Cleanup Objectives (SCOs) and other guidance as set forth in 6 New York Codes, Rules and Regulations (NYCRR) Part 375-1 and Part 375-2 Inactive Hazardous Waste Disposal Site Remedial Program dated December 14, 2006.

- Guidelines referenced in NYSDEC document titled “DER-10 Technical Guidance for Site Investigation and Remediation”, dated May 2010 (DER-10).

- Appropriate water quality standards and guidance values as set forth in NYSDEC Division of Water Technical and Operational Series (TOGS 1.1.1) document titled “Ambient Water Quality Standards and Guidance Values and Groundwater Effluent

Limitations", dated June 1998 as amended by a January 1999 Errata Sheet, an April 2000 Addendum and a June 2004 Addendum.

- Guidelines referenced in the New York State Department of Health (NYSDOH) document titled "Final Guidance for Evaluating Soil Vapor Intrusion in the State of New York" dated October 2006.

- City of Rochester Right-of-Way permit requirements.

## 1.3 PURPOSE

The purpose of the work presented herein is to complete soil vapor and groundwater sampling in select locations positioned near the perimeter of the Site and within the public right-of-way (ROW) in proximity to potentially susceptible receptors (e.g., the nearby residential properties, in proximity to buried utilities, etc.), and to test the samples collected to evaluate VOC content. The data generated will be used to evaluate potential VOC impact within soil vapor and overburden groundwater, and to make decisions on appropriate actions to address potential exposures, if such work is determined to be necessary.

## 2.0 SCOPE OF WORK

Subcontractors retained by DAY, and DAY personnel will perform the work presented herein. The subcontractor will be responsible for coordinating a utility stakeout for identification and clearance of nearby utilities prior to commencement of work. Prior to requesting a utility clearance, DAY will mark the tentative sample locations presented on Figure 2 with white marking paint and/or pin flags. [Note: Based on surface conditions and proximity of underground utilities, the sample locations may vary from those presented on Figure 2. The actual sample locations will be located using a GPS unit with sub-meter accuracy or by a licensed surveyor. The sample locations will be located using the coordinate system (i.e., World Geodetic System of 1984 datum (WGS84) or NAD83) and reference datum (i.e., mean sea level) required by EQuIS. It is not anticipated that the field locations will be significantly different than those presented on Figure 2.]

Prior to advancing test borings within the city streets, sidewalks or landscaped areas a City of Rochester ROW permit will be acquired. The requirements of ROW permit will be adhered to during work conducted within the City of Rochester ROWs.

### 2.1 GROUNDWATER SAMPLING

DAY will retain the services of a drilling subcontractor to advance nine test borings and subsequently collect grab groundwater samples in the approximate locations depicted on Figure 2. The test borings will be advanced by direct-push methods to the first observed water-bearing zone [i.e., anticipated to occur at depths of about 8 feet (ft.) below ground surface (bgs) to 12 ft. bgs, depending on location] or to equipment refusal (i.e., anticipated equipment refusal depths are about 12 ft. to 15 ft. bgs]. The soil samples collected will be observed for soil type, potential contamination (e.g., staining, unusual odors, etc.), and screened with a photoionization detector (PID).

Upon advancement of test borings into the top of the saturated zone, a Geoprobe® Screen Point 16 Groundwater Sampler or similar with an approximate 4-foot long well screen will be connected to the sample rods and advanced two additional feet into undisturbed material (or to equipment refusal), but no more than four feet, to collect representative groundwater samples for subsequent testing. Refer to Appendix A for a copy of the Geoprobe® Screen Point 16 Groundwater Sampler Standard Operating Procedure that will be followed during the groundwater sample collection.

Subsequent to the advancement of the Geoprobe® Screen Point 16 Groundwater Sampler to the desired depth, a peristaltic pump will be connected to flexible inert tubing installed within the screen cavity. The peristaltic pump effluent will be transferred to analytical laboratory supplied containers for subsequent testing. In the event groundwater is not encountered during test boring advancement, or a suitable volume of groundwater cannot be collected from a sample location, a soil sample from the interval in which equipment refusal occurred would be collected and submitted for analytical laboratory testing in lieu of a groundwater sample. NYSDEC approval will be obtained in each instance a soil sample is collected in lieu of a groundwater sample. Soil samples being submitted for analytical laboratory testing will be collected in accordance with United States Environmental Protection Agency (USEPA) Method 5035A (refer to Appendix B).

At the conclusion of groundwater sampling, the reusable equipment including portions of the

Geoprobe® Screen Point 16 Groundwater Sampler will be decontaminated using Alconox soap, or similar, and clean tap water. Assuming field evidence of apparent contamination is not detected, the direct push soil samples not being submitted for testing will be placed back in the test boring from which they originated and the remaining annulus will be filled with grout/concrete to the ground surface. If field evidence of potential impact is identified (e.g., PID readings in excess of background, staining, chemical-type odors) samples will be containerized for subsequent testing and disposal (refer to Section 2.3). Test borings advanced within asphalt or concrete will be repaired with a concrete patch with a similar thickness as the surrounding road/sidewalk.

In addition to the test samples, an equipment blank (i.e., equipment rinsate) sample, a matrix spike (MS) sample, a matrix spike duplicate (MSD) sample, and a duplicate sample will be collected from one or more of the groundwater sampling locations and submitted for analytical laboratory testing (refer to Section 2.4).

The equipment blank, duplicate, MS/MSD, trip blank and groundwater samples collected will be submitted to a New York State Department of Health (NYSDOH) Environmental Laboratory Approval Program (ELAP)-certified laboratory for testing of target compound list (TCL) VOCs plus tentatively identified compounds (TICs) using NYSDEC Analytical Services Protocol (ASP). The analytical laboratory results will be provided in an ASP Category B data package. The analytical laboratory data will be submitted to an independent party for review and preparation of a Data Usability Summary Report (DUSR). The DUSR will comply with NYSDEC requirements.

## 2.2 SUBSURFACE SOIL VAPOR POINT INSTALLATION AND SAMPLING

The soil vapor survey will consist of the following work completed in accordance with applicable provisions outlined by the NYSDOH in the document titled *Guidance for Evaluating Soil Vapor Intrusion in the State of New York* dated October 2006, including Section 2.7 (Sampling Protocols) and Section 2.8 (Quality Assurance/Quality Control), refer to Appendix C (Soil Vapor Sampling Guidance).

DAY will retain the services of a drilling subcontractor to advance soil vapor points in the approximate locations presented on Figure 2 (i.e., in locations in proximity to the groundwater sampling points described in Section 2.1). The temporary soil vapor points will be installed by advancing a direct-push test boring to a depth approximately 1 foot above the top of the groundwater as determined based upon conditions observed during the collection of the groundwater samples. After reaching the targeted depth, a soil vapor probe (e.g., 6-inch long double woven stainless steel screen attached to 3/8-inch Teflon lined tubing) will be installed in the borehole at the targeted depth. Refer to Appendix D for a specification sheet of the soil vapor probe. The borehole will then be backfilled with clean filter sand to a depth of at least 6 inches above the top of the soil vapor probe. Thereafter the remaining borehole will be backfilled with bentonite. [Note: A second soil vapor point will be installed in proximity to sample point ROW-2 to collect a QA/QC duplicate sample.]

Subsequent to soil vapor probe installation; the reusable equipment will be cleaned with Alconox soap, or similar, and clean tap water.

Prior to sampling, the soil vapor sample points will be tested for potential surface air infiltration

using a helium tracer gas test in accordance with the provisions outlined in the NYSDOH guidance document. Assuming the helium concentration measured in the soil vapor probe is below 10% of the enriched atmosphere as required by NYSDOH guidance, the soil vapor point will be purged of 1 to 3 volumes of air at a flow rate that does not exceed 0.2 liters per minute. Subsequent to purging of the soil vapor point, sampling will commence. In the event a soil vapor point fails the helium tracer test, the surface seal will be repaired and the test repeated until the helium is measured below the NYSDOH guidance.

Samples will be collected using 6-liter Summa canisters equipped with 2-hour regulators. The vacuum reading will be recorded at the start of the test and monitored throughout the test. In the event that the cold weather promotes condensation in the sample tubing during sample collection, tube warmers may be used to address this condition. In addition to the soil vapor samples, one background outdoor air sample will be collected approximately three feet off the ground form an upwind location, as determined at the time of sample collection, in a batch certified Summa canister during the same general two hour period. Following collection, the Summa canisters will be transported under chain-of-custody control to the analytical laboratory for testing.

The soil vapor samples (including the duplicate sample collected from ROW-2) and the background outdoor air sample will be submitted to a NYSDOH ELAP-certified analytical laboratory for analysis of VOCs via USEPA Method TO-15 using applicable ASP protocol. The analytical laboratory results will be provided in an ASP Category B data package. The analytical laboratory will be requested to meet the minimum reporting limit of 0.25 ug/$m^3$ for TCE and vinyl chloride, and 3 ug/$m^3$ for the remaining TO-15 list VOCs. The analytical laboratory data will be submitted to an independent party for review and preparation of a DUSR. The DUSR will comply with NYSDEC requirements, and be submitted to EQuIS within 90 days of receipt of the analytical laboratory data. [Note: Preliminary analytical laboratory results will be submitted to the NYSDEC and NYSDOH upon receipt from the laboratory.]

Soil samples collected during the advancement of the probe holes will be containerized and returned to the Site. If field evidence of potential impact is identified (e.g., PID readings in excess of background, staining, chemical-type odors) samples will be containerized for subsequent testing and disposal (refer to Section 2.3). Test borings advanced within asphalt or concrete will be repaired with a concrete patch with a similar thickness as the surrounding road/sidewalk.

## 2.3 INVESTIGATION DERIVED WASTES MANAGEMENT AND DISPOSAL

It is anticipated that solid and liquid study-derived wastes will be generated during the studies presented herein. Investigation derived wastes will be managed in accordance with applicable provisions set forth of DER-10 Section 3.3(e).

Potentially contaminated liquid wastes will likely consist of decontamination water, and soil cuttings that cannot be returned to the borehole. Storage of liquid IDW will be generally collected in 5-gallon buckets and placed on-site in a New York State Department of Transportation (NYSDOT) 55-gallon drum. The anticipated location of this storage drum is on the northern portion of the Site. Liquids that are grossly contaminated or suspected to contain non-aqueous phase liquid (NAPL) will be placed in separate drums and labeled accordingly. Management of liquid IDW following completion of the groundwater sampling may be modified

following review of the laboratory results. It is anticipated that liquid IDW will be discharged to the Monroe County Pure Waters sanitary sewer system under a sewer use permit. Sampling of IDW necessary to obtain a sewer use permit will be incorporated into the Report of Findings. A copy of the Monroe County Pure Waters sewer use permit will be provided to the NYSDEC prior to any discharge to the sanitary sewer system. Drummed liquid IDW that is grossly contaminated or suspected to contain NAPL will also be characterized using the investigation test results and other sampling data as necessary to dispose or treat the material in accordance with the applicable rules and regulations.

Potentially contaminated solid wastes will likely include disposable sampling equipment and personal protective equipment (PPE), soil samples that were collected but not selected for analytical laboratory testing and excess soil cuttings from direct-push operations. It is anticipated that the solid IDW will be placed in NYSDOT 55-gallon drums stored on the northern portion of the Site. Solids that are grossly contaminated or suspected to contain NAPL will be placed in separate drums and labeled accordingly. The IDW solids will be characterized and disposed of off-site in accordance with the applicable rules and regulations. If, based on a review of the analytical laboratory results that re-use of the IDW is possible, the NYSDEC will be notified of the proposed re-use of IDW for approval prior to implementation.

## 2.4 ANALYTICAL LABORATORY QUALITY ASSURANCE/QUALITY CONTROL

Spectrum Analytical Inc. of Warwick, Rhode Island (Spectrum) will be retained to complete the analytical laboratory testing. Spectrum is a NYSDOH ELAP certified laboratory (ELAP ID LAI00329). Refer to Appendix E for a copy of Spectrum's Quality Assurance Plan. The proposed analytical laboratory testing program for the samples collected as part of the studies conducted during the Work Plan is presented on Table 2 *Proposed Analytical Laboratory Testing Program.*

Dr. Maxine Wright-Walters of Environmental Data Validation Inc. (EDV) of Pittsburgh, Pennsylvania will complete a DUSR on the Category B deliverables analytical laboratory data that is generated as part of the scope of work this work plan. The DUSR will be conducted in accordance with the provisions set forth in Appendix 2B of DER-10 Technical Guidance for Site Investigation and Remediation dated May 2010. The findings of the DUSR will be incorporated in the Report of Findings. Refer to Appendix F for a copy of EDV's Quality Assurance/Quality Control Plan and a copy of Dr. Maxine Wright-Walters curriculum vitae.

## 2.5 Health and Safety

A site-specific Health and Safety Plan (HASP) that includes a Community Air Monitoring Program (CAMP) has been prepared, and the provisions of these documents must be followed during the completion of the field studies described in the Work Plan. The HASP will be reviewed by DAY employees assigned to this project before starting work. Other entities can adopt the protocols set forth in the HASP or can develop their own HASP, which must, at a minimum, satisfy the requirements of the HASP prepared by DAY and approved by the NYSDEC and NYSDOH.

## 2.6 CAMP Monitoring Specific to the Right-of-Way Groundwater and Soil Vapor Testing

In addition to the requirements described in the CAMP, the requirements presented in this

section will also be adhered to during implementation of this Work Plan.

Establishment of CAMP Monitoring Stations

Due to the distance between sample locations, new monitoring stations will be set up around the work zone for each test location. The specific locations will be determined based upon wind conditions at the time of fieldwork. CAMP stations will be placed between the area of intrusive activities and the immediate receptors (e.g., the nearest residential property or other occupied structure), and in an upwind location. As such, a minimum of one CAMP monitoring station will be placed downwind of the work zone, and the upwind CAMP station will be periodically monitored to obtain background levels.

## 2.7 REPORT OF FINDINGS

Upon receipt of the DUSR, a report describing the work completed, and presenting conclusions and recommendations will be submitted. This report will include copies of test boring logs; analytical laboratory results test results and executed chain-of-custody documentation; tables comparing test results to applicable guidance values and standards; a copy of the DUSR; an updated Site Plan; and other applicable documentation.

## 3.0 SCHEDULE

The schedule for the studies outlined the Work Plan is as follows:

**Groundwater Sampling and Soil Vapor Point Testing and Sampling:** The work will commence within 30 calendar days of the approval of the Work Plan by the NYSDEC and NYSDOH. The groundwater and soil vapor samples will be submitted to the analytical laboratory within 14 calendar days of the initiation of the fieldwork.

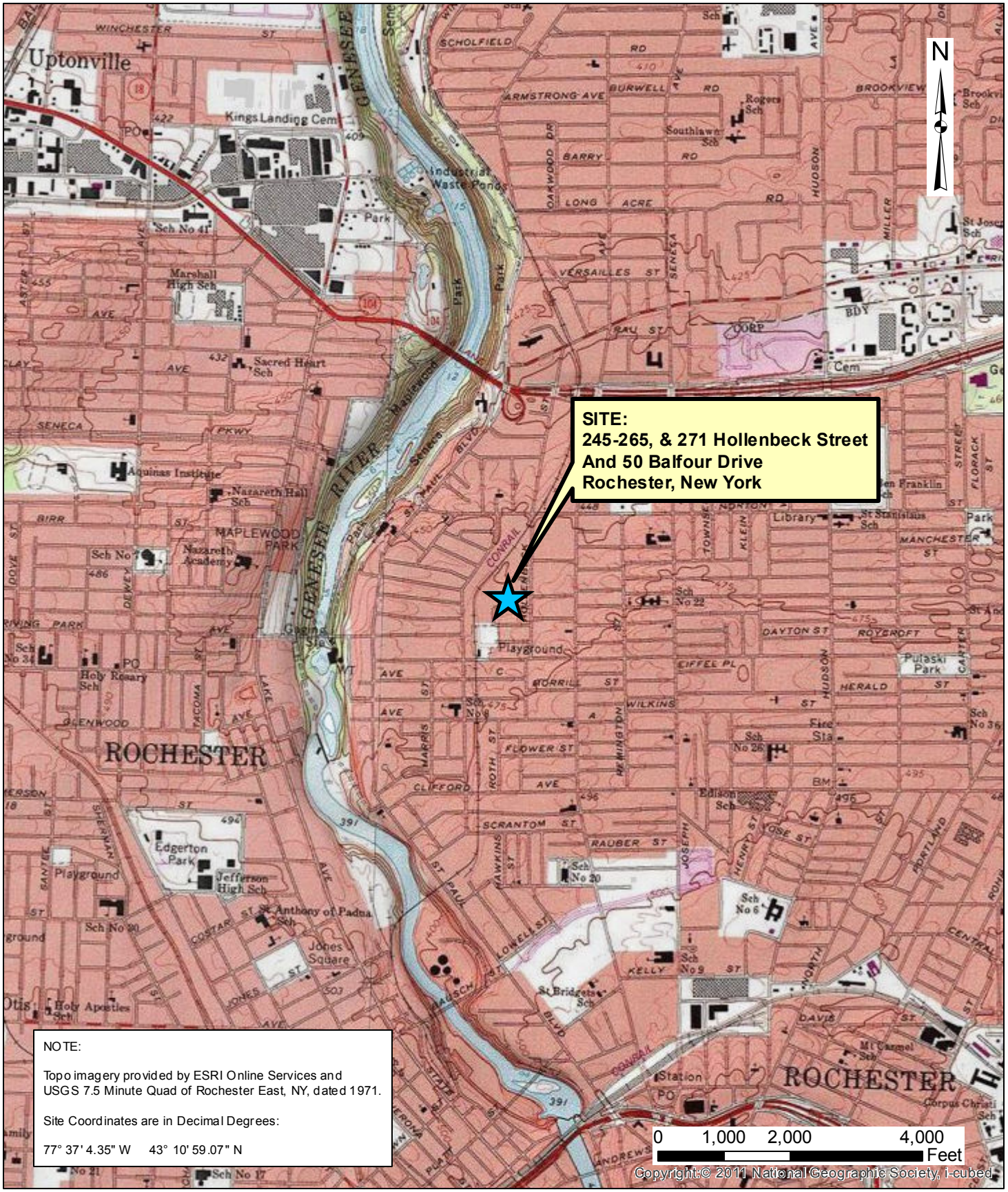
**Laboratory Testing:** The preliminary results of laboratory testing will be provided to the NYSDEC and NYSDOH within 15 workdays of sample submittal to the analytical laboratory.

**DUSR:** A DUSR will be submitted within 90 calendar days of receipt of the laboratory testing report, and the validated data will also be submitted to EQuIS within 90 calendar days of receipt.

**Report of Findings:** A draft report will be submitted to the NYSDEC and NYSDOH within 30 calendar days of the receipt of the DUSR.

DAY will coordinate and communicate with the NYSDEC and NYSDOH project managers and their staff regarding implementation of the various aspects of this project. This includes, but is not limited to, participation in progress meetings, presentation of field findings and analytical laboratory results.

**FIGURES**




| | | | |
|---|---|---|---|
| Date: 08-16-2013 | **DAY ENVIRONMENTAL, INC.**<br>Environmental Consultants<br>Rochester, New York 14606<br>New York, New York 10170 | Project Title: 245-265, & 271 HOLLENBECK STREET AND 50 BALFOUR DRIVE ROCHESTER, NEW YORK | Project No.: 4845E-13 |
| Drawn By: CPS | | Drawing Title: Project Locus Map | FIGURE 1 |
| Scale: AS NOTED | | | |

Document Path: E:\GIS\Aerial Locus Maps\4845S-13Mcalpn - Locus.mxd

315 Hollenbeck Street
Recycling Facility
destroyed by fire June 2007

333 Hollenbeck Street
Rochester Midland

480 Conkey Ave
Gas Station
Repair Shop
NYSDEC
Spill: 9700254

Conkey Ave

ROW-9
ROW-3
ROW-2
ROW-4
ROW-1
ROW-8
ROW-6
ROW-5
ROW-7

Printer

Vehicle Repair Shop

Mazda Ter

Hollenbeck St

Rialto St

Balfour Dr

Ok Ter

N

0 50 100 200 Feet

## Legend

- Tentative soil vapor and grab groundwater sample locations
- Site Boundary

NOTES:

Parcel boundaries provided by the Monroe County Department of Environmental Services, dated 2013.

Aerial imagery provided by the New York State GIS Clearinghouse,dated 2012.

Last Date Saved: 12 Dec 2013 Document Path: E:\GIS\GIS Mapping\Mxd\pin4845S-13Mxd\pin4845S-5 - Tentative Soil Vapor and GW Samples.mxd

| DESIGNED BY | DATE |
|---|---|
| RLK | 12-2013 |
| DRAWN BY | DATE DRAWN |
| CPS | 12-2013 |
| SCALE | DATE ISSUED |
| AS NOTED | 12-11-2013 |

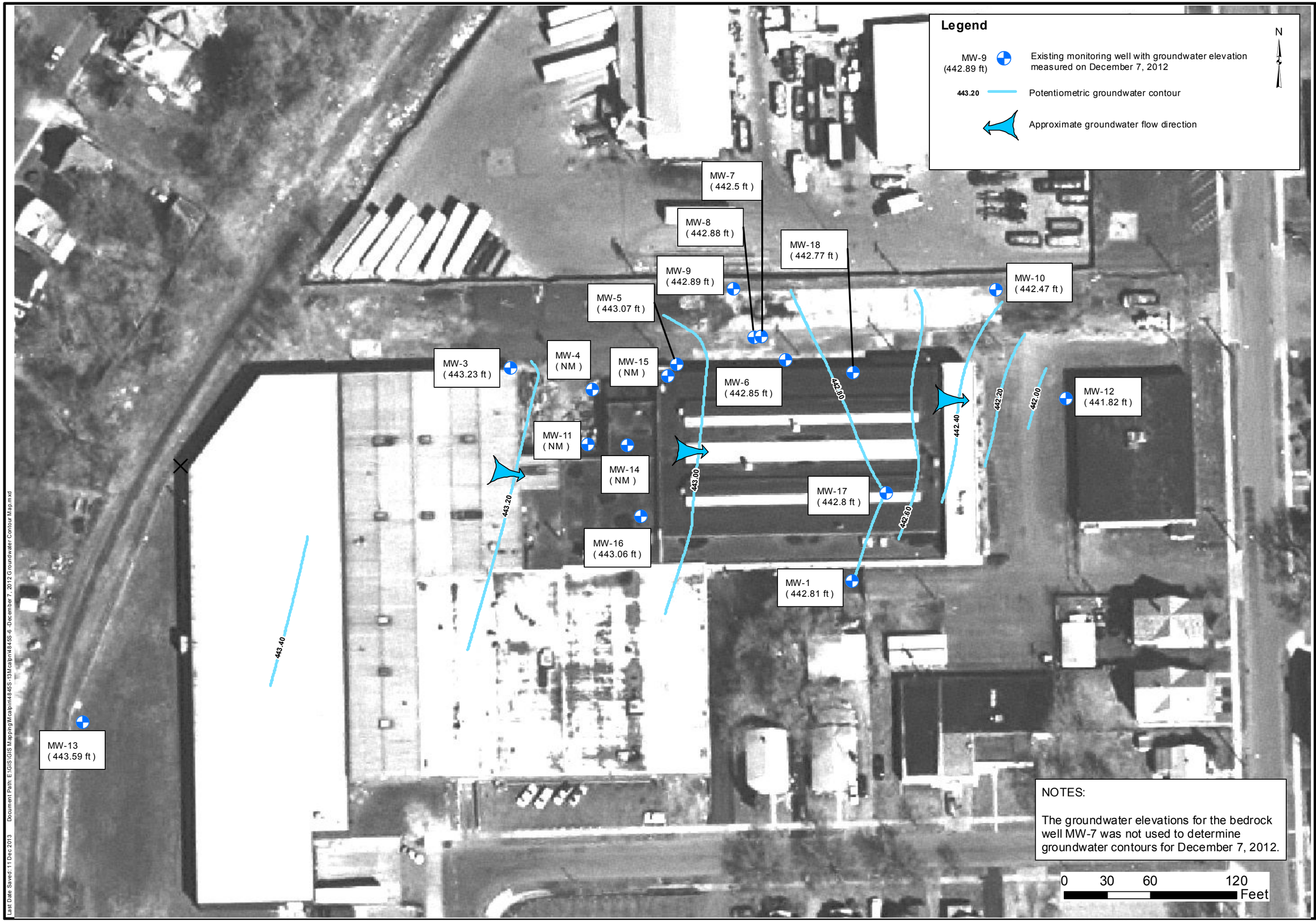

DAY ENVIRONMENTAL, INC.
Environmental Consultants
Rochester, New York 14606
New York, New York 10170


Project Title
47, 50, 53 AND 88 BALFOUR DRIVE
245-265 AND 271 HOLLENBECK STREET
ROCHESTER, NEW YORK

Drawing Title
Proposed Site Plan of Soil Vapor and Grab Groundwater Sample Locations

Project No.
4845S-13

FIGURE 2




DESIGNED BY RLK
DATE 12-2013
DRAWN BY CPS/CAH
DATE DRAWN 12-2013
SCALE AS NOTED
DATE ISSUED 12-11-2013


day
DAY ENVIRONMENTAL, INC.
Environmental Consultants
Rochester, New York 14606
New York, New York 10170


Project Title
50 BALFOUR DRIVE
245-265 AND 271 HOLLENBECK STREET
ROCHESTER, NEW YORK

Drawing Title
Potentiometric Groundwater Contour Map for December 7, 2012

Project No.
4845S-13

FIGURE 3

Last Date Saved: 11 Dec 2013 Document Path: E:\GIS\GIS Mapping\Mcalpin\4845S-13\Mcalpin4845S-8 - December 7, 2012 Groundwater Contour Map.mxd

# TABLES

**TABLE 1**

**SUMMARY OF ANALYTICAL LABORATORY RESULTS - GROUNDWATER SAMPLES**

**RIGHT-OF-WAY AND SITE PERIMETER SOIL VAPOR AND GROUNDWATER EVALUATION WORK PLAN**

**245-265 271 HOLLENBECK STREET AND 50 BALFOUR DRIVE**
**ROCHESTER, NEW YORK**

| COMPOUND | NYSDEC Standard or Guidance Value[1] | SAMPLE LOCATIONS AND SAMPLE DATES | | | | | | | | | | | | | | | | | | | |
|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|
| | | MW-1 | | | | | | | | | | MW-2 | MW-3 | | | | | | | MW-4 | |
| | | 10/2/97 | 10/15/04 | 6/7/06 | 6/28/07 | 5/1/08 | 8/21/08 | 4/14/10 | 10/25/10 | 9/22/11 | 11/29/12 | 10/2/97 | 10/2/97 | 10/15/04 | 6/7/06 | 5/1/08 | 8/21/08 | 10/25/10 | 9/22/11 | 11/29/12 | 10/2/97 | 8/21/08 |
| PCE | 5 | 11.9 | 5.4 | 3.85 | ND (20) | ND (2.0) | 2.34 | 7.61 | 8.87 | 7.02 | 4.1 | ND (2.0) | 9.7E | 7.1 | ND (2.0) | ND (2.0) | 2.70 | ND (20.0) | ND (10) | 4.15 | ND (2.0) | ND (2.0) |
| TCE | 5 | 546.9 | 78 | 112 | 216 | 23.2 | 46.3 | 123 | 119 | 97.2 | 61.1 | 206.3 | 607.4 | 170 | 95.4 | 28.9 | 98.3 | 156 | 190 | 214 | 11.2 | ND (2.0) |
| trans 1,2-DCE | 5 | ND (10) | ND (1.0) | ND (2.0) | ND (20) | ND (2.0) | ND (2.0) | ND (2.0) | ND (2.0) | ND (2.0) | ND (2.0) | ND (2.0) | ND (10) | ND (2.0) | ND (2.0) | ND (2.0) | ND (2.0) | ND (20.0) | ND (10) | ND (2.0) | 6.4 | ND (2.0) |
| Cis 1,2-DCE | 5 | 14.8E | 15 | 19.1 | 45.2 | 5.46 | 13.5 | 21.6 | 18.6 | 22.3 | 39.4 | 38.2E | 39.9E | 33 | 17.7 | 8.29 | 25.2 | 40.8 | 47.1 | 57.7 | 295E | ND (2.0) |
| VC | 2 | ND (10) | ND (2.0) | ND (2.0) | ND (20) | ND (2.0) | ND (2.0) | ND (2.0) | ND (2.0) | ND (2.0) | ND (2.0) | ND (2.0) | ND (10) | ND (4.0) | ND (2.0) | ND (2.0) | 2.35 | ND (20.0) | ND (10) | 2.79 | 229.6 | ND (2.0) |
| TOTAL VOCs | -- | 573.6 | 98.4 | 134.95 | 261.2 | 28.66 | 62.14 | 152.21 | 146.47 | 126.52 | 104.6 | 244.5 | 657 | 210.1 | 113.1 | 37.19 | 128.55 | 196.8 | 237.1 | 278.64 | 542.2 | 0 |

| COMPOUND | NYSDEC Standard or Guidance Value[1] | SAMPLE LOCATIONS AND SAMPLE DATES | | | | | | | | | | | | | | | | | | | | | | | | |
|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|
| | | MW-5 | | | | | | | | | | | | | | | | | MW-6 | | | | | | | | |
| | | 10/2/97 | 10/15/04 | 6/7/06 | 7/31/07 | 5/1/08 | 8/21/08 | 1/23/09 | 3/31/09 | 5/8/09 | 7/20/09 | 9/14/09 | 3/24/10 | 4/14/10 | 10/26/10 | 9/22/11 | 11/29/12 | 1/4/13 | 10/2/97 | 10/1/08 | 3/31/09 | 9/14/09 | 3/24/10 | 10/26/10 | 9/22/11 | 11/29/12 | 1/4/13 |
| PCE | 5 | ND (10) | ND (2.5) | ND (200) | ND (200) | ND (200) | ND (50) | ND (20) | ND (40) | ND (20) | ND (20) | ND (20) | ND (100) | ND (50) | ND (20) | ND (10) | ND (400) | ND (50) | ND (50) | ND (200) | ND (400) | ND (100) | ND (100) | ND (400) | ND (200) | ND (2.0) | ND (2.0) |
| TCE | 5 | 909.5 | 5.5 | ND (200) | ND (200) | ND (200) | ND (50) | ND (20) | 136 | 51.7 | ND (20) | ND (20) | 632 | 205 | ND (20) | ND (10) | ND (400) | ND (50) | 4,917.1 | ND (200) | ND (400) | ND (100) | ND (100) | ND (400) | ND (200) | ND (2.0) | ND (2.0) |
| trans 1,2-DCE | 5 | 15.0 | 4.0 | ND (200) | ND (200) | ND (200) | ND (50) | ND (20) | ND (40) | ND (20) | ND (20) | ND (20) | ND (100) | ND (50) | ND (20) | ND (10) | ND (400) | ND (50) | 43.5E | ND (200) | ND (400) | ND (100) | ND (100) | ND (400) | ND (200) | ND (2.0) | ND (2.0) |
| Cis 1,2-DCE | 5 | 2,840E | 260 | 11,300 | 2,130 | 4,020 | 3,600 | 845 | 2,820 | 1,880 | 2,150 | 240 | 6,330 | 5,800 | 736 | 909 | 10,000 | 4,600 | 4,390E | 6,230 | 11,500 | 7,200 | 7,960 | 6,040 | 5,410 | 42.7 | 5.3 |
| VC | 2 | 654.8 | 43 | 1,080 | 457 | 289 | 764 | 179 | 168 | 192 | 365 | 129 | 412 | 447 | 126 | 153 | 668 | 390 | 837 | 4,300 | 4,730 | 5,400 | 4,110 | 3,570 | 4,440 | 24.8 | ND (2.0) |
| TOTAL VOCs | -- | 4,419.30 | 312.5 | 12,380 | 2,587 | 4,309 | 4,364 | 1,024 | 3,144 | 2,124 | 2,515 | 369 | 7,374 | 6,452 | 862 | 1,062 | 10,668 | 4,990 | 10,187.6 | 10,530 | 16,230 | 12,600 | 12,070 | 9,610 | 0 | 67.5 | 5.3 |

| COMPOUND | NYSDEC Standard or Guidance Value[1] | SAMPLE LOCATIONS AND SAMPLE DATES | | | | | | | | | | | | | | | | | | | | |
|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|
| | | MW-7 (ROCK) | | | | | | | | | | | MW-8 | | | | | | | | | |
| | | 6/27/07 | 7/31/07 (14-15') | 7/31/07 (25.5-26.5') | 5/1/08 | 8/21/08 | 3/31/09 | 9/14/09 | 3/24/10 | 10/26/10 | 9/22/11 | 11/29/12 | 6/27/07 | 5/1/08 | 8/21/08 | 3/31/09 | 7/20/09 | 9/14/09 | 3/24/10 | 10/26/10 | 9/22/11 | 11/29/12 |
| PCE | 5 | ND (20) | ND (10) | ND (2.0) | ND (5.0) | ND (5.0) | ND (5.0) | 3.84 | 6.19 | ND (5.0) | ND (5.0) | ND (4.0) | ND (20) | ND (20) | ND (20) | ND (20) | ND (20) | ND (200) | ND (10) | ND (20) | ND (20) | ND (20) |
| TCE | 5 | 175 | 282 | 127 | 367 | ND (5.0) | 183 | 135 | 160 | 108 | 42.3 | 204 | ND (20) | ND (20) | ND (20) | ND (20) | ND (20) | ND (200) | ND (10) | ND (20) | ND (20) | ND (20) |
| trans 1,2-DCE | 5 | ND (20) | ND (10) | ND (2.0) | ND (5.0) | ND (5.0) | ND (5.0) | ND (2.0) | ND (2.0) | ND (5.0) | ND (5.0) | ND (4.0) | ND (20) | ND (20) | ND (20) | ND (20) | ND (20) | ND (200) | ND (10) | ND (20) | ND (20) | ND (20) |
| Cis 1,2-DCE | 5 | 103 | 144 | 78.2 | 135 | 139 | 69 | 117 | 86 | 130 | 114 | 330 | 1,220 | 220 | 862 | 1,460 | 2,330 | 1,600 | 727 | 841 | 1,080 | 808 |
| VC | 2 | ND (20) | 37.6 | 21.4 | 25.4 | 19.7 | 10.2 | 27 | 15 | 31 | 47.9 | 107 | 321 | 102 | 352 | 479 | 1,250 | 1,030 | 506 | 845 | 1,560 | 1,050 |
| TOTAL VOCs | -- | 278 | 463.2 | 226.8 | 527.4 | 158.7 | 261.8 | 282.8 | 267.5 | 269.0 | 204.2 | 641.0 | 1,541 | 322 | 1,214 | 1,939 | 3,580 | 2,630 | 1,233 | 1,686 | 2,640 | 1,858 |

Notes:
(1) NYSDEC Division of Water Technical Operations and Guidance Series (1.1.1): Ambient Water Quality Standards and Guidance Values and Groundwater Effluent Limitations; Class GA (source of drinking water from groundwater
Concentrations are shown in ug/l or parts per billion (ppb)
ND(200) - constituent not detected at the concentration shown in parenthesis
E = Denotes an estimated concentration
PCE = tetrachloroethene
TCE = trichloroethene
Trans 1,2-DCE = trans 1,2-dichloroethene
Cis 1,2 DCE = Cis 1,2-dichloroethene
VC = vinyl chloride

**TABLE 1**

**SUMMARY OF ANALYTICAL LABORATORY RESULTS - GROUNDWATER SAMPLES**

**RIGHT-OF-WAY AND SITE PERIMETER SOIL VAPOR AND GROUNDWATER EVALUATION WORK PLAN**

**245-265 271 HOLLENBECK STREET AND 50 BALFOUR DRIVE**
**ROCHESTER, NEW YORK**

| COMPOUND | NYSDEC Standard or Guidance Value[1] | SAMPLE LOCATIONS AND SAMPLE DATES | | | | | | | | | | | | | | | | | | | | | |
|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|
| | | MW-9 | | | | | | | | | MW-10 | | | | | | | | | MW-11 | | | |
| | | 6/27/07 | 5/1/08 | 8/21/08 | 3/31/09 | 9/14/09 | 3/24/10 | 10/25/10 | 9/22/11 | 11/29/12 | 6/27/07 | 5/1/08 | 8/21/08 | 3/31/09 | 9/14/09 | 3/24/10 | 10/25/10 | 9/22/11 | 11/29/12 | 6/27/07 | 5/1/08 | 8/21/08 | |
| PCE | 5 | ND (20) | ND (2.0) | ND (2.0) | ND (20) | ND (2.0) | ND (2.0) | ND (2.0) | ND (2.0) | ND (2.0) | ND (20) | ND (2.0) | ND (2.0) | ND (20) | ND (2.0) | ND (2.0) | ND (2.0) | ND (2.0) | ND (2.0) | ND (20) | ND (2.0) | ND (2.0) | |
| TCE | 5 | 57.6 | 47.5 | 79.9 | 45.0 | 87.8 | 56.4 | ND (2.0) | ND (2.0) | ND (2.0) | 86.3 | 37.9 | 62.0 | 33.0 | 56.9 | 32.7 | ND (2.0) | ND (2.0) | ND (2.0) | 50.3 | ND (2.0) | ND (2.0) | |
| trans 1,2-DCE | 5 | ND (20) | ND (2.0) | ND (2.0) | ND (20) | ND (2.0) | ND (2.0) | ND (2.0) | ND (2.0) | ND (2.0) | ND (20) | ND (2.0) | ND (2.0) | ND (20) | ND (2.0) | ND (2.0) | ND (2.0) | ND (2.0) | ND (2.0) | ND (20) | ND (2.0) | ND (2.0) | |
| Cis 1,2-DCE | 5 | ND (20) | 15.2 | 32.3 | 26.8 | 30.9 | 24.9 | ND (2.0) | ND (2.0) | ND (2.0) | 202 | 158 | 196 | 106 | 122 | 76 | ND (2.0) | ND (2.0) | ND (2.0) | 101 | ND (2.0) | ND (2.0) | |
| VC | 2 | ND (20) | ND (2.0) | 3.48 | ND (20) | 4.18 | 2.4 | ND (2.0) | ND (2.0) | ND (2.0) | 227 | 96.5 | 103 | 59.8 | 116 | 51.4 | ND (2.0) | ND (2.0) | 2.78 | ND (20) | ND (2.0) | ND (2.0) | |
| TOTAL VOCs | -- | 57.6 | 62.7 | 115.68 | 71.80 | 122.88 | 83.70 | 0 | 0 | 0 | 515.3 | 292.4 | 361 | 198.8 | 294.9 | 160.2 | 0 | 0 | 2.78 | 151.3 | 0 | 0 | |

| COMPOUND | NYSDEC Standard or Guidance Value[1] | SAMPLE LOCATIONS AND SAMPLE DATES | | | | | | | | | | | | | | | | | | | | | | | |
|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|
| | | MW-12 | | | | | | | MW-13 | | | | | | | MW-14 | MW-16 | | | MW-17 | | | MW-18 | | | |
| | | 6/27/07 | 5/1/08 | 8/21/08 | 4/14/10 | 10/25/10 | 9/22/11 | 11/29/12 | 6/27/07 | 5/1/08 | 8/21/08 | 10/25/10 | 9/22/11 | 11/29/12 | 12/3/08 | 3/28/11 | 9/22/11 | 11/29/12 | 3/28/11 | 9/22/11 | 11/29/12 | 3/28/11 | 9/22/11 | 11/29/12 | 1/4/13 |
| PCE | 5 | ND (20) | ND (2.0) | 2.81 | ND (20) | ND (20) | ND (10) | ND (10) | ND (2.0) | ND (2.0) | ND (2.0) | ND (2.0) | ND (2.0) | ND (2.0) | 3.26 | ND (20) | ND (10) | ND (10) | 2.68 | ND (2.0) | ND (2.0) | ND (20) | ND (20) | ND (20) | ND (2.0) |
| TCE | 5 | 289 | 196 | 229 | 218 | 314 | 170 | 83.6 | 5.48 | 3.99 | 4.54 | 21.9 | 12.8 | ND (2.0) | 18.9 | 495 | 282 | 18.8 | 103 | 63.6 | ND (2.0) | ND (20) | ND (20) | ND (20) | ND (2.0) |
| trans 1,2-DCE | 5 | ND (20) | ND (2.0) | ND (2.0) | ND (20) | ND (20) | ND (10) | ND [10] | ND (2.0) | ND (2.0) | ND (2.0) | ND (2.0) | ND (2.0) | ND (2.0) | ND (2.0) | ND (20) | 26.9 | 32.4 | 4.92 | 4.79 | 5.14 | ND (20) | ND (20) | ND (20) | ND (2.0) |
| Cis 1,2-DCE | 5 | 83.6 | 53.7 | 85.6 | 133.0 | 128.0 | 83.9 | 60.8 | 6.40 | 3.97 | 11.1 | 16.9 | 13.0 | ND (2.0) | 10.9 | 156 | 253 | 280 | 49.3 | 62.4 | 78.4 | 1,080 | 384 | 11.8 | 33 |
| VC | 2 | ND (20) | 18.4 | 27.5 | 36.4 | 53.6 | 47.8 | 35.4 | ND (2.0) | ND (2.0) | ND (2.0) | ND (2.0) | ND (2.0) | ND (2.0) | 4.54 | 28.1 | 29.2 | 60.7 | 8.85 | 27.3 | 26.9 | 1,520 | 1,200 | 29.1 | 75 |
| TOTAL VOCs | -- | 372.6 | 268.1 | 344.91 | 387.4 | 495.6 | 301.70 | 179.8 | 11.88 | 7.96 | 15.54 | 38.80 | 25.80 | 0 | 37.6 | 679.1 | 591.1 | 391.9 | 168.75 | 158.09 | 110.04 | 2,600 | 1,584 | 40.9 | 108 |

Notes:
(1) NYSDEC Division of Water Technical Operations and Guidance Series (1.1.1): Ambient Water Quality Standards and Guidance Values and Groundwater Effluent Limitations; Class GA (source of drinking water from groundwater
Concentrations are shown in ug/l or parts per billion (ppb)
ND(200) - constituent not detected at the concentration shown in parenthesis
E = Denotes an estimated concentration
PCE = tetrachloroethene
TCE = trichloroethene
Trans 1,2-DCE = trans 1,2-dichloroethene
Cis 1,2 DCE = Cis 1,2-dichloroethene
VC = vinyl chloride

**TABLE 2**

**SUMMARY OF ANALYTICAL LABORATORY TESTING**
**RIGHT-OF-WAY AND SITE PERIMETER SOIL VAPOR AND GROUNDWATER EVALUATION WORK PLAN**

**245-265 271 HOLLENBECK STREET AND 50 BALFOUR DRIVE**
**ROCHESTER, NEW YORK**

| TASK | ANALYTICAL LABORATORY | PARAMETERS | METHOD | SAMPLE MATRIX | MAXIMUM ANTICIPATED # OF FIELD SAMPLES | DUPLICATE SAMPLES | EQUIPMENT BLANKS | TRIP BLANKS | MS/MSD OR MS/MD |
|---|---|---|---|---|---|---|---|---|---|
| Soil Samples [1] | Spectrum | TCL VOCs+TICs | 5030A | Soil | 9 | 1 | 1 | 1 | 1 |
| Groundwater | Spectrum | TCL VOCs+TICs | 8260 | Groundwater | 9 | 1 | 1 | 1 | 1 |
| Soil Vapor | Spectrum | TCL VOCs | TO-15 | Vapor | 9 | 1 | 0 | 0 | 0 |
| Waste Characterization - Investigation Derived Waste (IDW) | Spectrum | Soil IDW waste characterization program will be determined prior to disposal, based on quantity and the testing requirements of the disposal facility. | | | | | | | |
| | | Groundwater IDW waste characterization program will be determined prior to disposal, based on quantity and in accordance with the current City of Olean industrial wastewater permit for the Site. | | | | | | | |

**Notes**

1) Soil samples will only be collected in the event groundwater samples cannot be collected (i.e., insuffiect water, equipment refusal prior to saturated zone, etc.).

# APPENDIX A

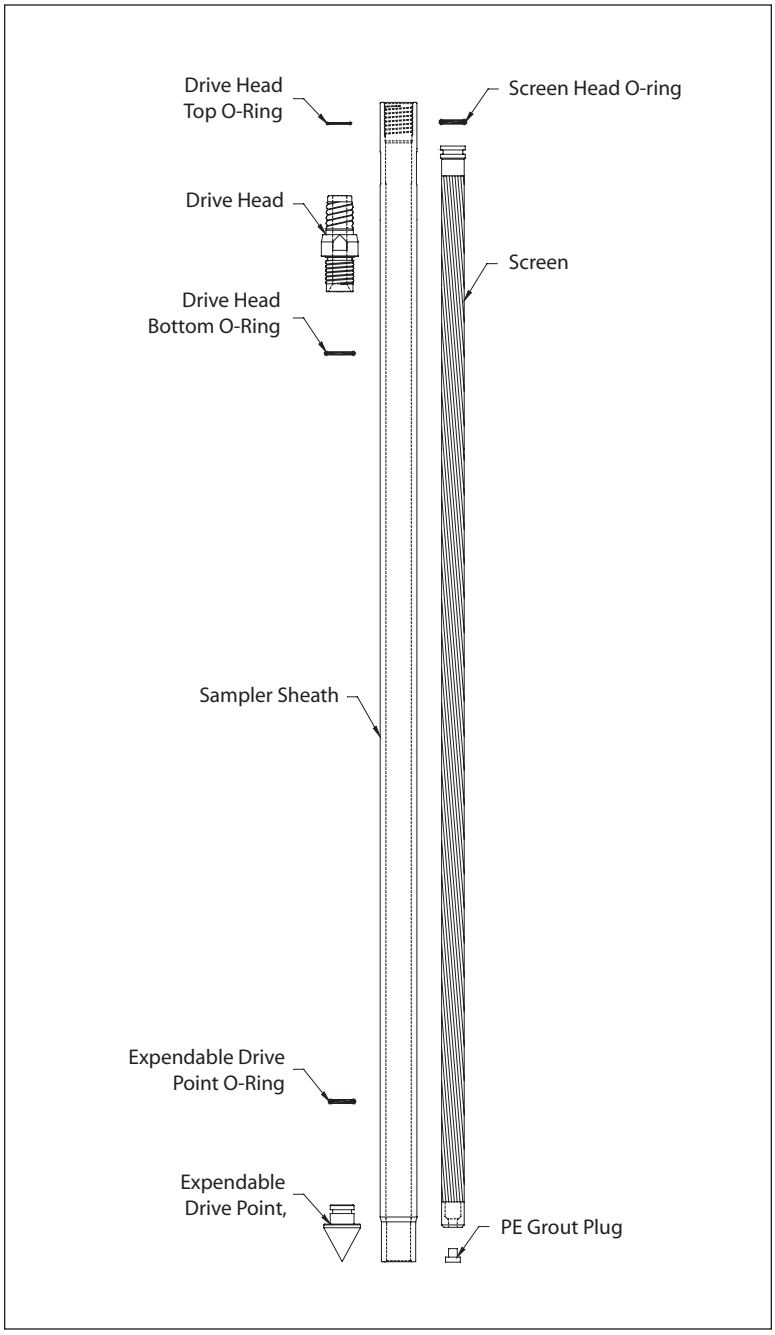
Geoprobe® Screen Point 16 Groundwater Sampler Standard Operating Procedure

# GEOPROBE® SCREEN POINT 16 GROUNDWATER SAMPLER

## STANDARD OPERATING PROCEDURE

**Technical Bulletin No. MK3142**

**PREPARED: November, 2006**




**GEOPROBE® SCREEN POINT 16 GROUNDWATER SAMPLER PARTS**


**Geoprobe® and Geoprobe Systems®, Macro-Core® and Direct Image® are Registered Trademarks of Kejr, Inc., Salina, Kansas**

**Screen Point 16 Groundwater Sampler is manufactured under U.S. Patent 5,612,498**

## 1.0 OBJECTIVE

The objective of this procedure is to drive a sealed stainless steel or PVC screen to depth, deploy the screen, obtain a representative water sample from the screen interval, and grout the probe hole during abandonment. The Screen Point 16 Groundwater Sampler enables the operator to conduct abandonment grouting that meets American Society for Testing and Materials (ASTM) Method D 5299 requirements for decommissioning wells and borings for environmental activities (ASTM 1993).

## 2.0 BACKGROUND

### 2.1 Definitions

**Geoprobe®:** A brand name of high quality, hydraulically powered machines that utilize both static force and percussion to advance sampling and logging tools into the subsurface. The Geoprobe® brand name refers to both machines and tools manufactured by Geoprobe Systems®, Salina, Kansas. Geoprobe® tools are used to perform soil core and soil gas sampling, groundwater sampling and monitoring, soil conductivity and contaminant logging, grouting, and materials injection.

**Screen Point 16 (SP16) Groundwater Sampler:** A direct push device consisting of a PVC or stainless steel screen that is driven to depth within a sealed, steel sheath and then deployed for the collection of representative groundwater samples. The assembled SP16 Sampler is approximately 51.5 inches (1308 mm) long with an OD of 1.625 inches (41 mm). Upon deployment, up to 41 inches (1041 mm) of screen can be exposed to the formation. The Screen Point 16 Groundwater Sampler is designed for use with 1.5-inch probe rods and machines equipped with the more powerful GH60 Hydraulic Hammer. Operators with GH40 Series hammers may chose to use this sampler in soils where driving is difficult.

**Rod Grip Pull System:** An attachment mounted on the hydraulic hammer of a direct push machine which makes it possible to retract the tool string with extension rods or flexible tubing protruding from the top of the probe rods. The Rod Grip Pull System includes a pull block with rod grip jaws that are bolted directly to the machine. A removable handle assembly straddles the tool string while hooking onto the pull block to effectively grip the probe rods as the hammer is raised. A separate handle assembly is required for each probe rod diameter.

### 2.2 Discussion

In this procedure, the assembled Screen Point 16 Groundwater Sampler (Fig. 2.1A) is threaded onto the leading end of a Geoprobe® probe rod and advanced into the subsurface with a Geoprobe® direct push machine. Additional probe rods are added incrementally and advanced until the desired sampling interval is reached. While the sampler is advanced to depth, O-ring seals at each rod joint, the drive head, and the expendable drive point provide a watertight system. This system eliminates the threat of formation fluids entering the screen before deployment and assures sample integrity.

Once at the desired sampling interval, extension rods are sent downhole until the leading rod contacts the bottom of the sampler screen. The tool string is then retracted approximately 44 inches (1118 mm) while the screen is held in place with the extension rods (Fig. 2.1B). As the tool string is retracted, the expendable point is released from the sampler sheath. The tool string and sheath may be retracted the full length of the screen or as little as a few inches if a small sampling interval is desired.

There are three types of screens that can be used in the Screen Point 16 Groundwater Sampler. Two of the these, a stainless steel screen with a standard slot size of 0.004 inches (0.10 mm) and a PVC screen with a standard slot size of 0.010 inches (0.25 mm), are recovered with the tool string after sampling. The third screen is also manufactured from PVC with a standard slot size of 0.010 inches (0.25 mm), but is designed to be left downhole when sampling is complete. This disposable screen has an exposed screen length of approximately 43 inches (1092 mm). The two screens that are recovered with the sampler both have an exposed screen length of approximately 41 inches (1041 mm).

**(continued on following page)**

An O-ring on the head of the stainless steel screens maintains a seal at the top of the screen. As a result, any liquid entering the sampler during screen deployment must first pass through the screen. PVC screens do not require an O-ring because the tolerance between the screen head and sampler sheath is near that of the screen slot size.

The screens are constructed such that flexible tubing, a mini-bailer, or a small-diameter bladder pump can be inserted into the screen cavity. This makes direct sampling possible from anywhere within the saturated zone. A removable plug in the lower end of the screens allows the user to grout as the sampler is extracted for further use.

Groundwater samples can be obtained in a number of ways. A common method utilizes polyethylene (TB25L) or Teflon® (TB25T) tubing and a Check Valve Assembly (GW4210). The check valve (with check ball) is attached to one end of the tubing and inserted down the casing until it is immersed in groundwater. Water is pumped through the tubing and to the ground surface by oscillating the tubing up and down.

An alternative means of collecting groundwater samples is to attach a peristaltic or vacuum pump to the tubing. This method is limited in that water can be pumped to the surface from a maximum depth of approximately 26 feet (8 m). Another technique for groundwater sampling is to use a stainless steel Mini-Bailer Assembly (GW41). The mini-bailer is lowered down the inside of the casing below the water level where it fills with water and is then retrieved from the casing.

The latest option for collecting groundwater from the SP16 sampler is to utilize a Geoprobe® MB470 Series Mechanical Bladder Pump (MBP)*. The MBP may be used to meet requirements of the low-flow sampling protocol (Puls and Barcelona 1996, ASTM 2003). Through participation in a U.S. EPA Environmental Technology Verification study, it was confirmed that the MB470 can provide representative samples (EPA 2003).

**The Mechanical Bladder Pump is manufactured under U.S. Patent No. 6,877,965 issued April 12, 2005.*

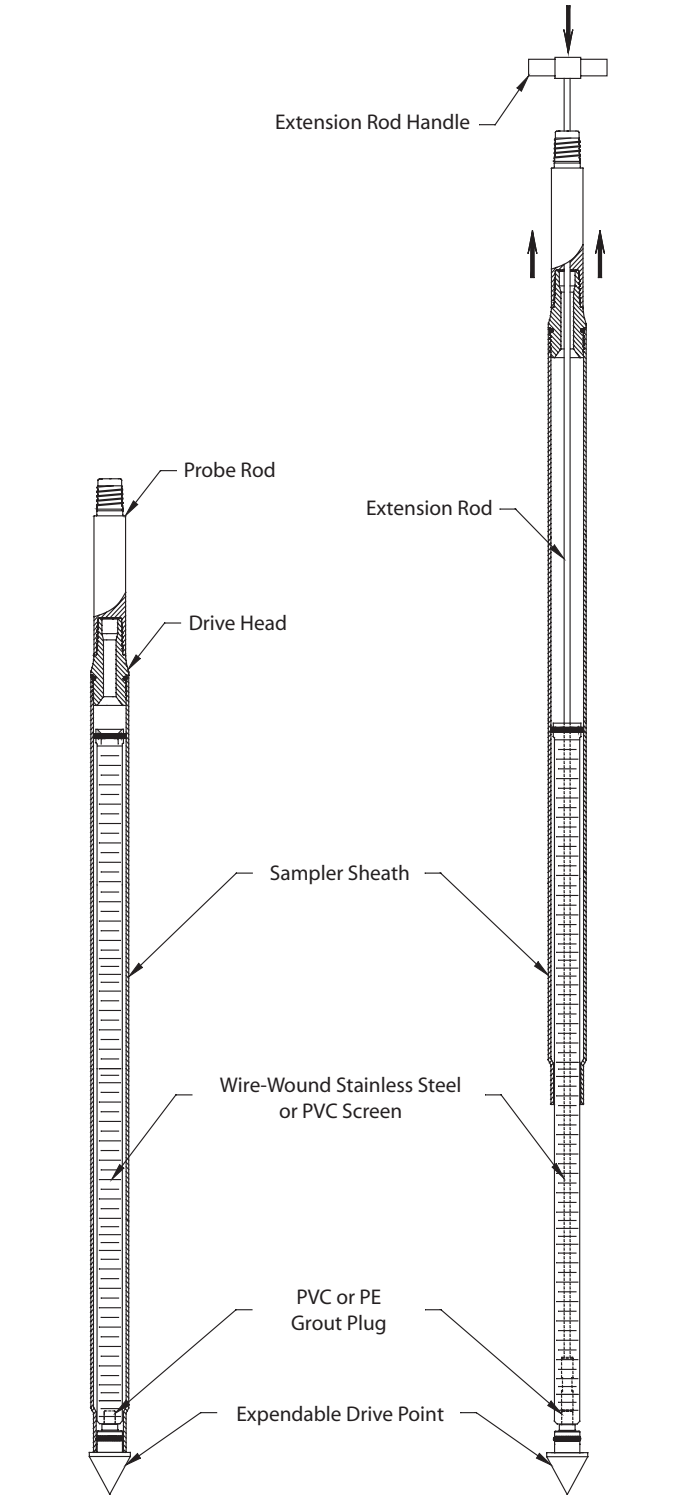



**FIGURE 2.1**
**Screen Point 16 Groundwater Sampler**

## 3.0 TOOLS AND EQUIPMENT

The following tools and equipment can be used to successfully recover representative groundwater samples with the Geoprobe® Screen Point 16 Groundwater Sampler. Refer to Figures 3.1 and 3.2 for identification of the specified parts. Tools are listed below for the most common SP16 / 1.5-inch probe rod configurations. Additional parts for optional rod sizes and accessories are listed in Appendix A.

| **SP16 Sampler Parts** | **Part Number** |
|---|---|
| SP16 Sampler Sheath | 15187 |
| SP16 Drive Head, 0.5-inch bore, 1.5-inch rods* | 18307 |
| SP16 O-ring Service Kit, 1.5-inch rods *(includes 4 each of the O-ring packets below)* | 15844 |
| *O-rings for Top of SP16 Drive Head, 1.5-inch rods only (Pkt. of 25)* | *15389* |
| *O-rings for Bottom of SP16 Drive Head (Pkt. of 25)* | *13196* |
| *O-rings for GW1520 Screen Head (Pkt. of 25)* | *GW1520R* |
| *O-rings for SP16 Expendable Drive Point (Pkt. of 25)* | *GW1555R* |
| Screen, Wire-Wound Stainless Steel, 4-Slot* | GW1520 |
| Grout Plugs, PE (Pkg. of 25) | GW1552K |
| Expendable Drive Points, steel, 1.625-inch OD (Pkg. of 25)* | GW1555K |
| Screen Point 16 Groundwater Sampler Kit, 1.5-inch Probe Rods *(includes 1 each of: 15187, 18307, 15844, GW1520, GW1535, GW1540, GW1555K, and GW1552K)* | 15770 |

| **Probe Rods and Probe Rod Accessories** | **Part Number** |
|---|---|
| Drive Cap, 1.5-inch probe rods, threadless, (for GH60 Hammer) | 12787 |
| Pull Cap, 1.5-inch probe rods | 15090 |
| Probe Rod, 1.5-inch x 60-inch* | 11121 |

| **Extension Rods and Extension Rod Accessories** | **Part Number** |
|---|---|
| Screen Push Adapter | GW1535 |
| Grout Plug Push Adapter | GW1540 |
| Extension Rod, 60-inch* | 10073 |
| Extension Rod Coupler | AT68 |
| Extension Rod Handle | AT69 |
| Extension Rod Jig | AT690 |
| Extension Rod Quick Link Coupler, pin | AT695 |
| Extension Rod Quick Link Coupler, box | AT696 |

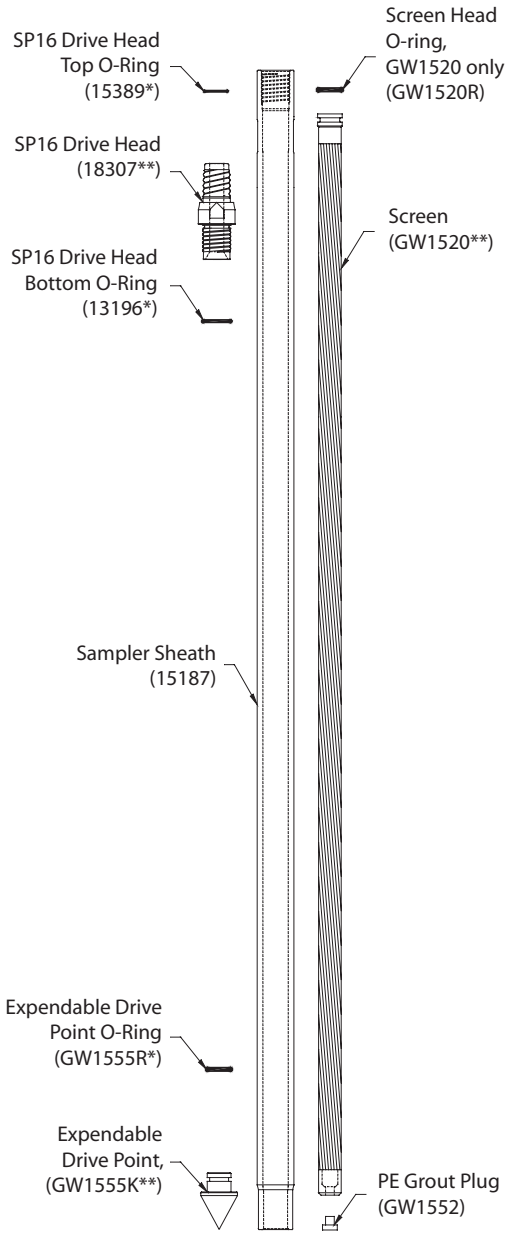
| **Grout Accessories** | **Part Number** |
|---|---|
| Grout Nozzle, for 0.375-inch OD tubing | GW1545 |
| High-Pressure Nylon Tubing, 0.375-inch OD / 0.25-inch ID, 100-ft. (30 m) | 11633 |
| Grout Machine, self-contained* | GS1000 |
| Grout System Accessories Package, 1.5-inch rods | GS1015 |

| **Groundwater Purging and Sampling Accessories** | **Part Number** |
|---|---|
| Polyethylene Tubing, 0.375-inch OD, 500 ft.* | TB25L |
| Check Valve Assembly, 0.375-inch OD Tubing* | GW4210 |
| Water Level Meter, 0.438-inch OD Probe, 100 ft. cable* | GW2000 |
| Mechanical Bladder Pump** | MB470 |
| Mini Bailer Assembly, stainless steel | GW41 |

| **Additional Tools** | **Part Number** |
|---|---|
| Adjustable Wrench, 6.0-inch | FA200 |
| Adjustable Wrench, 10.0-inch | FA201 |
| Pipe Wrenches | NA |

* See Appendix A for additional tooling options.

** Refer to the Standard Operating Procedure (SOP) for the Mechanical Bladder Pump (Technical Bulletin No. MK3013) for additional tooling needs.




* Included in the SP16 O-ring Service Kit (15844)
** Part numbers show most common tool configuration. See Appendix A for alternative parts.

**FIGURE 3.1**
**SP16 Sampler Parts**

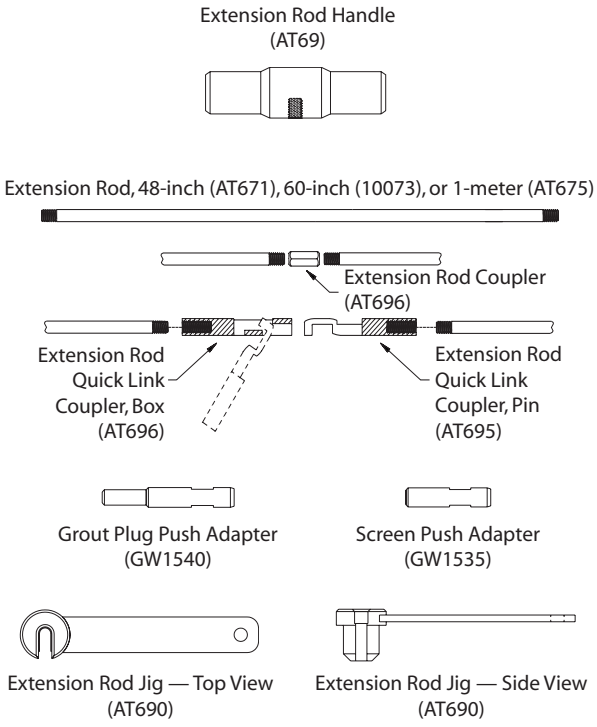



**FIGURE 3.2**
**Geoprobe® Extension Rods and Accessories**

## 4.0 OPERATION

### 4.1 Basic Operation

The SP16 sampler utilize a stainless steel or PVC screen which is encased in an alloy steel sampler sheath. An expendable drive point is placed in the lower end of the sheath while a drive head is attached to the top. O-rings on the drive head and expendable point provide a watertight sheath which keeps contaminants out of the system as the sampler is driven to depth.

Once the sampling interval is reached, extension rods equipped with a screen push adapter are inserted down the ID of the probe rods. The tool string is then retracted up to 44 inches (1118 mm) while the screen is held in place with the extension rods. The system is now ready for groundwater sampling. When sampling is complete, a removable plug in the bottom of the screen allows for grouting below the sampler as the tool string is retrieved.

### 4.2 Sampler Options

The Screen Point 15 and Screen Point 16 Groundwater Samplers are nearly identical. Subtle differences in the design of the SP16 sampler make it more durable than the earlier SP15 system. Operators of GH60-equipped machines should always utilize SP16 tooling. Operators of machines equipped with GH40 Series hammers may also choose SP16 tooling when sampling in difficult probing conditions.

A 1.75-inch OD Expendable Drive Point (17066K) and Disposable PVC Screen (16089) provide two useful options for the SP16 sampler. The 1.75-inch drive point may be used when soil conditions make it difficult to remove the sampler after driving to depth. The disposable PVC screen may be left downhole after sampling (when regulations permit) to eliminate the time required for screen decontamination.

### 4.3 Decontamination

In order to collect representative groundwater samples, all sampler parts must be thoroughly cleaned before and after each use. Scrub all metal parts using a stiff brush and a nonphosphate soap solution. Steam cleaning may be substituted for hand-washing if available. Rinse with distilled water and allow to air-dry before assembly.

### 4.4 SP16 Sampler Assembly (Figure 4.1)

Part numbers are listed for a standard SP16 sampler using 1.5-inch probe rods. Refer to Page 6 for screen and drive head alternatives.

1. Place an O-ring on a steel expendable drive point (GW1555K). Firmly seat the expendable point in the necked end of a sampler sheath (15187).

2. Install a PE Grout Plug (GW1552) in the bottom end of a Wire-wound Stainless Steel Screen (GW1520). Place a GW1520R O-ring in the groove on the top end of the screen.

3. Slide the screen inside of the sampler sheath with the grout plug toward the bottom of the sampler. Ensure that the expendable point was not displaced by the screen.

4. Install a bottom O-ring (13196) on a Drive Head (18307 or 15188). Thread the drive head into the sampler sheath using an adjustable wrench if necessary to ensure complete engagement of the threads. Attach a Drive Cap (12787 or 15590) to the top of the drive head.

   **NOTE:** The 18307 drive head should be used whenever possible as the smaller 0.5-inch ID provides a greater material cross-section for increased durability.

**Sampler assembly is complete.**

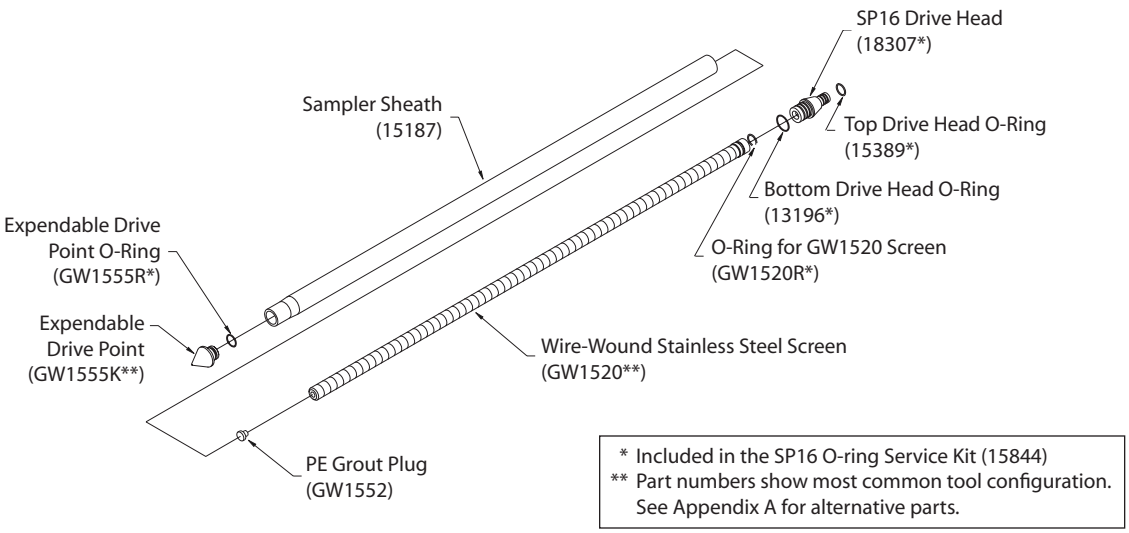



**FIGURE 4.1**
**Screen Point 16 Groundwater Sampler Assembly**

### 4.5 Advancing the SP16 Sampler

To provide adequate room for screen deployment with the Rod Grip Pull System, the probe derrick should be extended a little over halfway out of the carrier vehicle when positioning for operation.

**1.** Begin by placing the assembled sampler (Fig. 2.1.A) in the driving position beneath the hydraulic hammer of the direct push machine as shown in Figure 4.2.

**2.** Advance the sampler with the throttle control at slow speed for the first few feet to ensure that the sampler is aligned properly. Switch to fast speed for the remainder of the probe stroke.

**3.** Completely raise the hammer assembly. Remove the drive cap and place an O-ring in the top groove of the drive head. Distilled water may be used to lubricate the O-ring if needed.

Add a probe rod (length to be determined by operator) and reattach the drive cap to the rod string. Drive the sampler the entire length of the new rod with the throttle control at fast speed.

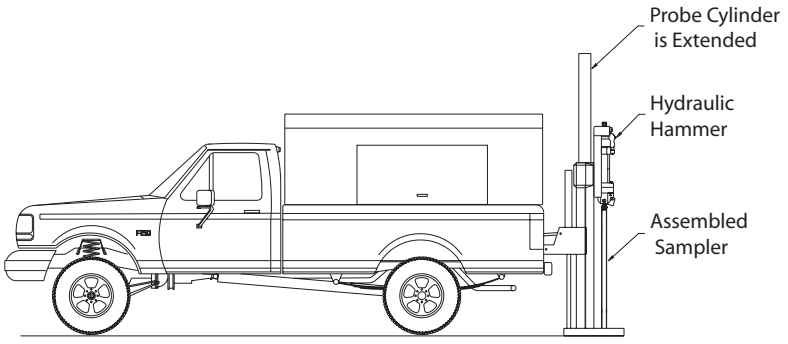



**FIGURE 4.2**
**Screen Point 16 Groundwater Sampler in Driving Position**

**4.** Repeat Step 3 until the desired sampling interval is reached. Approximately 12 inches (305 mm) of the last probe rod must extend above the ground surface to allow attachment of the puller assembly. A 12-inch (305 mm) rod may be added if the tool string is over-driven.

**5.** Remove the drive cap and retract the probe derrick away from the tool string.

### 4.6 Screen Deployment

1. Thread a screen push adapter (GW1535) on an extension rod of suitable length (AT671, 10073, or AT675). Attach a threaded coupler (AT68) to the other end of the extension rod. Lower the extension rod inside of the probe rod taking care not to drop it down the tool string. An extension rod jig (AT690) may be used to hold the rods.

2. Add extension rods until the adapter contacts the bottom of the screen. To speed up this step, it is recommended that Extension Rod Quick Links (AT695 and AT696) are used at every other rod joint.

3. Ensure that at least 48 inches (1219 mm) of extension rod protrudes from the probe rod. Thread an extension rod handle (AT69) on the top extension rod.

4. Maneuver the probe assembly into position for pulling.

5. Raise (pull) the tool string while physically holding the screen in place with the extension rods (Fig. 4.3.B). A slight knock with the extension rod string will help to dislodge the expendable point and start the screen moving inside the sheath.

   Raise the hammer and tool string about 44 inches (1118 cm) if using a GW1520 or GW1530 screen. At this point the screen head will contact the necked portion of the sampler sheath (Fig. 4.3.C.) and the extension rods will rise with the probe rods. Use care when deploying a PVC screen so as not to break the screen when it contacts the bottom of the sampler sheath.

   The Disposable Screen (16089) will extend completely out of the sheath if the tool string is raised more than 45 inches (1143 mm). Measure and mark this distance on the top extension rod to avoid losing the screen during deployment.

6. Remove the rod grip handle, lower the hammer assembly, and retract the probe derrick. Remove the top extension rod (with handle) and top probe rod. Finally, extract all extension rods.

7. Groundwater samples can now be collected with a mini-bailer, peristaltic or vacuum pump, tubing bottom check valve assembly, bladder pump, or other acceptable small diameter sampling device.

   When inserting tubing or a bladder pump down the rod string, ensure that it enters the screen interval. The leading end of the tubing or bladder pump will sometimes catch at the screen head giving the illusion that the bottom of the screen has been reached. An up-and-down motion combined with rotation helps move the tubing or bladder pump past the lip and into the screen.

### 4.7 Abandonment Grouting for GW1520 and GW1530 Screens

The SP16 Sampler can meet ASTM D 5299 requirements for abandoning environmental wells or borings when grouting is conducted properly. A removable grout plug makes it possible to deploy tubing through the bottom of GW1520 and GW1530 screens. A GS500 or GS1000 Grout Machine is then used to pump grout into the open probe hole as the sampler is withdrawn. The following procedure is presented as an example only and should be modified to satisfy local abandonment grouting regulations.

1. Maneuver the probe assembly into position for pulling. Attach the rod grip puller to the top probe rod. Raise the tool string approximately 4 to 6 inches (102 to 152 cm) to allow removal of the grout plug.

2. Thread the Grout Plug Push Adapter (GW1540) onto an extension rod. Insert the adapter and extension rod inside the probe rod string. Add extension rods until the adapter contacts the grout plug at the bottom of the screen. Attach the handle to the top extension rod. When the extension rods are slightly raised and lowered, a relatively soft rebound should be felt as the adapter contacts the grout plug. This is especially true when using a PVC screen.

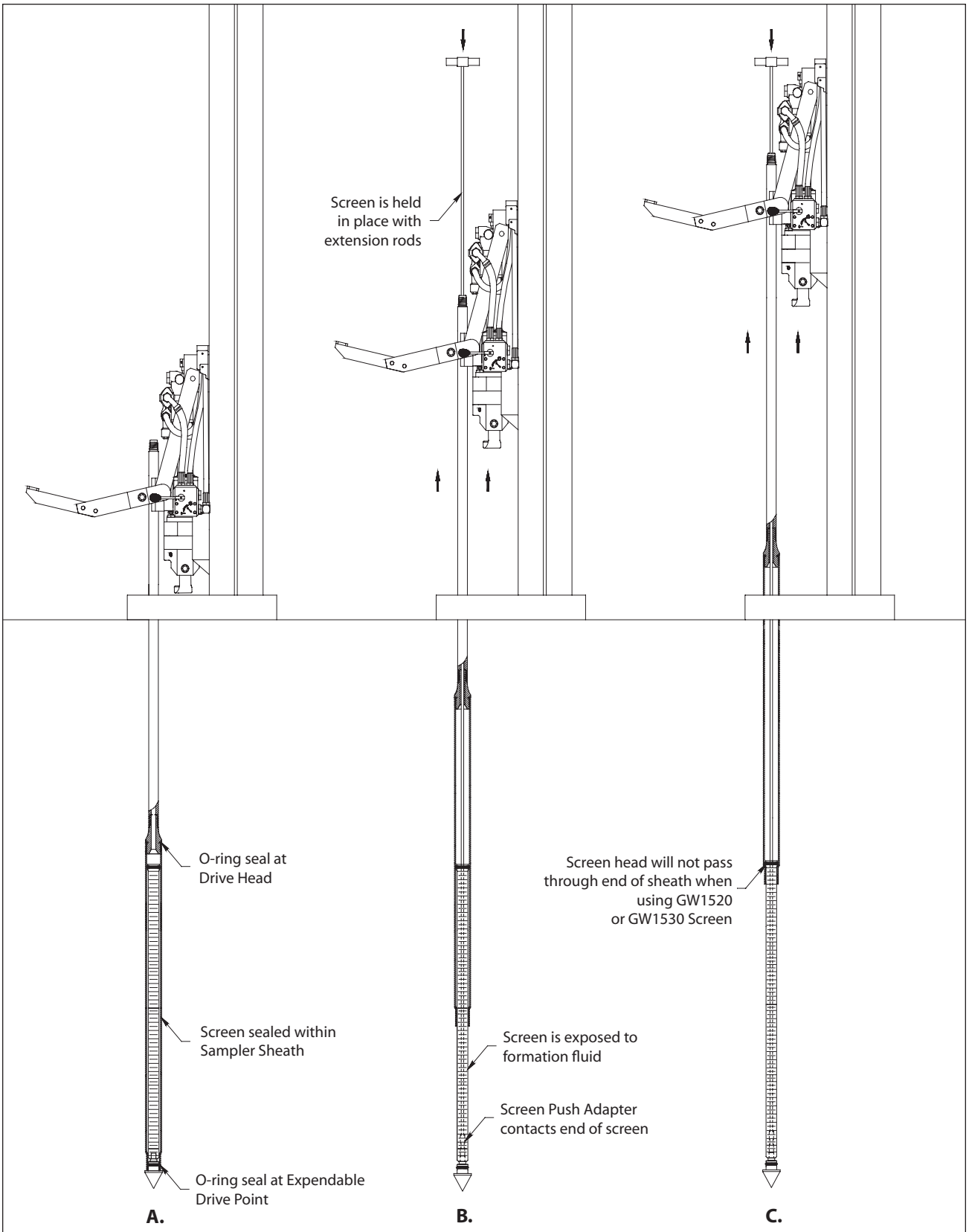



**FIGURE 4.3**
**Screen Deployment for SP16 Sampler**

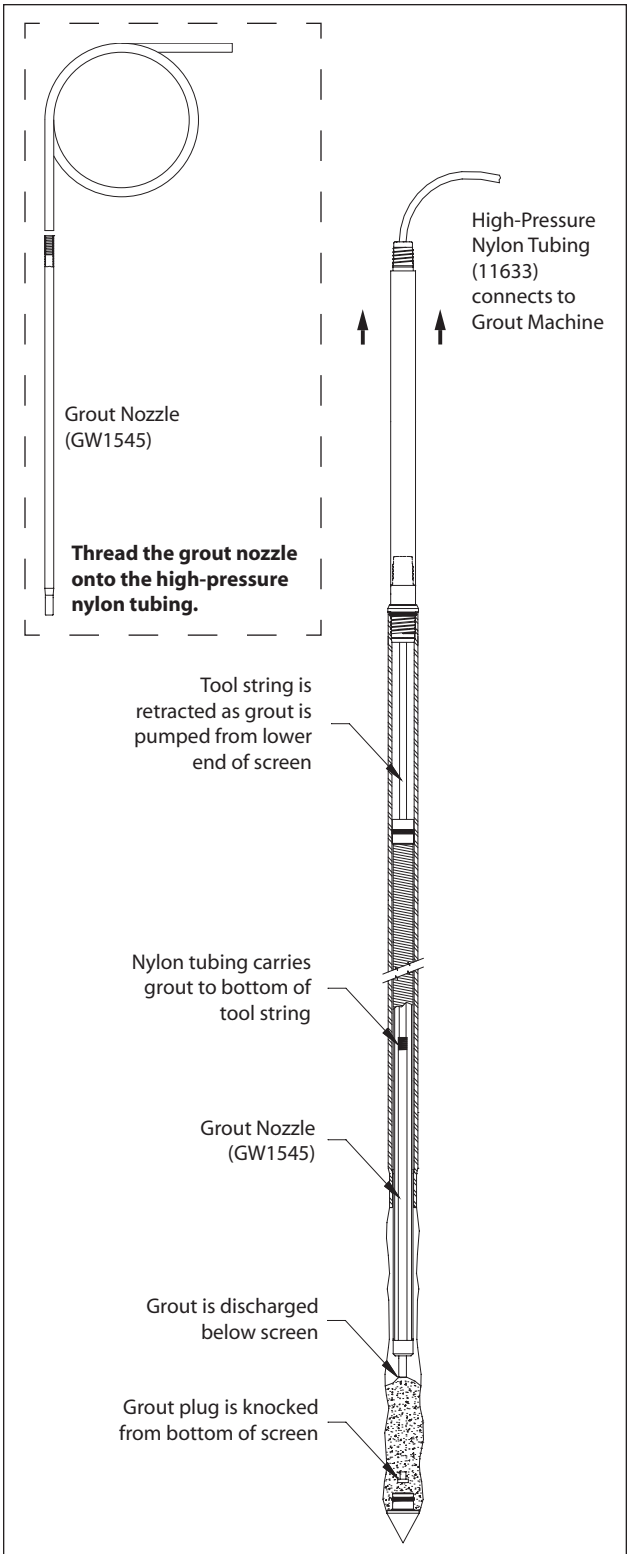



**FIGURE 4.4**
**Grouting the SP16 Sampler With a GW1520 or GW1530 Screen**

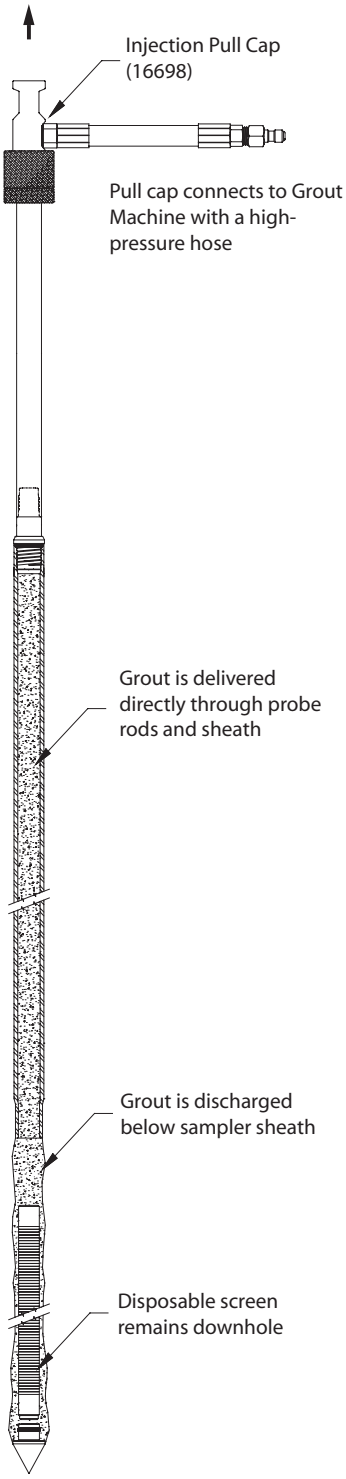



**FIGURE 4.5**
**Grouting the SP16 Sampler With a 16089 Disposable Screen**

3. Place a mark on the extension rod even with the top of the probe rod. Apply downward pressure on the extension rods and push the grout plug out of the screen. The mark placed on the extension rod should now be below the top of the probe rod. Remove all extension rods.

   **Note:** When working with a stainless steel screen, it may be necessary to raise and quickly lower the extension rods to jar the grout plug free. When the plug is successfully removed, a metal-on-metal sensation may be noted as the extension rods are gently "bounced" within the probe rods.

4. A Grout Nozzle (GW1545) is now connected to High-Pressure Nylon Tubing (11633) and inserted down through the probe rods to the bottom of the screen (Fig. 4.4). It may be necessary to pump a small amount of clean water through the tubing during deployment to jet out sediments that settled in the bottom of the screen. Resistance will sometimes be felt as the grout nozzle passes through the drive head. Rotate the tubing while moving it up-and-down to ensure that the nozzle has reached the bottom of the screen and is not hung up on the drive head.

   ***Note:*** *All probe rods remain strung on the tubing as the tool string is pulled. Provide extra tubing length to allow sufficient room to lay the rods on the ground as they are removed. An additional 20 feet is generally enough.*

5. Operate the grout pump while pulling the first rod with the rod grip pull system. Coordinate pumping and pulling rates so that grout fills the void left by the sampler. After pulling the first rod, release the rod grip handle, fully lower the hammer, and regrip the tool string. Unthread the top probe and slide it over the tubing placing it on the ground near the end of the tubing.

6. Repeat Step 5 until the sampler is retrieved. Do not bend or kink the tubing when pulling and laying out the probe rods. Sharp bends create weak spots in the tubing which may burst when pumping grout. Remember to operate the grout pump only when pulling the rod string. The probe hole is thus filled with grout from the bottom up as the rods are extracted.

7. Promptly clean all probe rods and sampler parts before the grout sets up and clogs the equipment.

### 4.8 Abandonment Grouting for the 16089 Disposable Screen

ASTM D 5299 requirements can also be met for the SP16 samplers when using the 16089 disposable screen. Because the screen remains downhole after sampling, the operator may choose either to deliver grout to the bottom of the tool string with nylon tubing or pump grout directly through the probe rods using an Injection Pull Cap (16698). A GS500 or GS1000 Grout Machine is needed to pump grout into the open probe hole as the sampler is withdrawn. The following procedure is presented as an example only and should be modified to satisfy local abandonment grouting regulations.

1. Maneuver the probe assembly into position for pulling with the rod grip puller.

2. Thread the screen push adapter onto an extension rod. Insert the adapter and extension rod inside the probe rod string. Add extension rods until the adapter contacts the bottom of the screen. Attach the handle to the top extension rod.

3. The disposable screen must be extended at least 46 inches (1168 mm) to clear the bottom of the sampler sheath. Considering the length of screen deployed in Section 4.7, determine the remaining distance required to fully extend the screen from the sheath. Mark this distance on the top extension rod.

4. Pull the tool string up to the mark on the top extension rod while holding the disposable screen in place.

   The screen is now fully deployed and the sampler is ready for abandonment grouting. Apply grout to the bottom of the tool string during retrieval using either flexible tubing (as described in Section 4.7) or an injection pull cap (Fig. 4.5). This section continues with a description of grouting with a pull cap.

5. Remove the rod grip handle and maneuver the probe assembly directly over the tool string. Thread an Injection Pull Cap (16698) onto the top probe rod and close the hammer pull latch over the top of the pull cap.

6. Connect the pull cap to a Geoprobe® grout machine using a high-pressure grout hose.

7. Operate the pump to fill the entire tool string with grout. When a sufficient volume has been pumped to fill the tool string, begin pulling the rods and sampler while continuing to operate the grout pump. Considering the known pump volume and sampler cross-section, time tooling withdrawal to slightly "overpump" grout into the subsurface. This will ensure that all voids are filled during sampler retrieval.

   The grouting process can lubricate the probe hole sufficiently to cause the tool string to slide back downhole when disconnected from the pull cap. Prevent this by withdrawing the tool string with the rod grip puller while maintaining a connection to the grout machine with the pull cap.

### 4.9 Retrieving the Screen Point 16 Sampler

If grouting is not required, the Screen Point 16 Sampler can be retrieved by pulling the probe rods as with most other Geoprobe® applications. The Rod Grip Pull System should be used for this process as it allows the operator to remove rods without completely releasing the tool string. This avoids having the probe rods fall back downhole when released during the pulling procedure. A standard Pull Cap (15164) may still be used if preferred. Refer to the Owner's Manual for your Geoprobe® direct push machine for specific instructions on pulling the tool string.

## 5.0 REFERENCES


American Society of Testing and Materials (ASTM), 2003. D6771-02 Standard Practice for Low-Flow Purging and Sampling for Wells and Devices Used for Ground-Water Quality Investigations. ASTM, West Conshocken, PA. (www.astm.org)

American Society of Testing and Materials (ASTM), 1993. ASTM 5299 *Standard Guide for Decommissioning of Groundwater Wells, Vadose Zone Monitoring Devices, Boreholes, and Other Devices for Environmental Activities.* ASTM West Conshohocken, PA. (www.astm.org)

Geoprobe Systems®, 2003, *Tools Catalog, V.6.*

Geoprobe Systems®, 2006, *Model MB470 Mechanical Bladder Pump Standard Operating Procedure (SOP), Technical Bulletin No. MK3013.*

Puls, Robert W., and Michael J. Barcelona, 1996. Ground Water Issue: Low-Flow (Minimal Drawdown) Ground Water Sampling Procedures. EPA/540/S-95/504. April.

U.S. Environmental Protection Agency (EPA), 2003. Environmental Technology Verification Report: Geoprobe Inc., Mechanical Bladder Pump Model MB470. Office of Research and Development, Washington, D.C. EPA/600R-03/086. August.

## Appendix A
## ALTERNATIVE PARTS

The following parts are available to meet unique soil conditions. See section 3.0 for a complete listing of the common tool configurations for the Geoprobe® Screen Point 16 Groundwater Sampler.

| **SP16 Sampler Parts and Accessories** | **Part Number** |
|---|---|
| SP16 Drive Head, 0.625-inch bore, 1.5-inch rods | 15188 |
| Expendable Drive Points, aluminum, 1.625-inch OD (Pkg. of 25) | GW1555ALK |
| Expendable Drive Points, steel, 1.75-inch OD (Pkg. of 25) | 17066K |
| Screen, PVC, 10-Slot | GW1530 |
| Screen, Disposable, PVC, 10-Slot | 16089 |

| **Groundwater Purging and Sampling Accessories** | **Part Number** |
|---|---|
| Polyethylene Tubing, 0.25-inch OD, 500 ft | TB17L |
| Polyethylene Tubing, 0.5-inch OD, 500 ft | TB37L |
| Polyethylene Tubing, 0.625-inch OD, 50 ft | TB50L |
| Check Valve Assembly, 0.25-inch OD Tubing | GW4240 |
| Check Valve Assembly, 0.5-inch OD Tubing | GW4220 |
| Check Valve Assembly, 0.625-inch OD Tubing | GW4230 |
| Water Level Meter, 0.375-inch OD Probe, 100-ft. cable | GW2001 |
| Water Level Meter, 0.438-inch OD Probe, 200-ft. cable | GW2002 |
| Water Level Meter, 0.375-inch OD Probe, 200-ft. cable | GW2003 |
| Water Level Meter, 0.438-inch OD Probe, 30-m cable | GW2005 |
| Water Level Meter, 0.438-inch OD Probe, 60-m cable | GW2007 |
| Water Level Meter, 0.375-inch OD Probe, 60-m cable | GE2008 |

| **Grouting Accessories** | **Part Number** |
|---|---|
| Grout Machine, auxiliary-powered | GS500 |

| **Probe Rods, Extension Rods, and Accessories** | **Part Number** |
|---|---|
| Probe Rod, 1.5-inch x 1-meter | 17899 |
| Probe Rod, 1.5-inch x 48-inch | 13359 |
| Drive Cap, 1.5-inch rods (for GH40 Series Hammer) | 15590 |
| Rod Grip Pull Handle, 1.5-inch Probe Rods (for GH40 Series Hammer) | GH1555 |
| Extension Rod, 48-inch | AT671 |
| Extension Rod, 1-meter | AT675 |

Equipment and tool specifications, including weights, dimensions, materials, and operating specifications included in this brochure are subject to change without notice. Where specifications are critical to your application, please consult Geoprobe Systems®.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**APPENDIX B**

Spectrum's Recommended Soil Sampling Procedure

And

USEPA Method 5035 Soil Sampling Procedure

**SPECTRUM ANALYTICAL, INC.**

***Featuring HANIBAL TECHNOLOGY***

# SAMPLE COLLECTION TECHNIQUES FOR VOC SOILS

## SPECTRUM TIP

The ratio of solvent to soil should be 1ml:1g. ***In all cases, the soil in the vial must be completely covered by the solvent.*** The most accurate way to achieve this criteria would be through the use of a field analytical balance.

When this option is not practical, Spectrum recommends the following procedure:

- Mark the bottom of the solvent meniscus on VOA vial with a permanent marker.
- Add sample to the VOA vial just **below** this meniscus mark.

### - WARNING -

USE CAUTION TO AVOID THE NEED TO RESAMPLE

- ***Don't collect too much soil.***

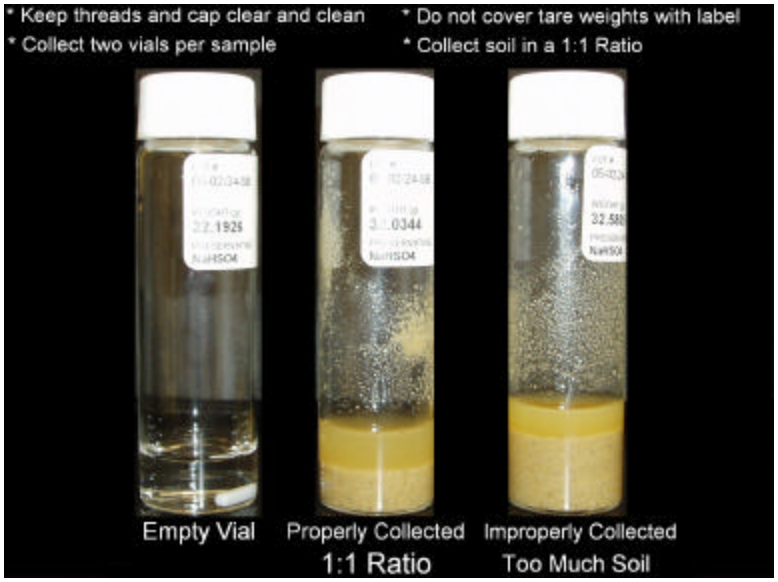
Too much soil may interfere with the solvents ability to extract the VOCs from the soil and may also impede the laboratories ability to analyze the sample. Excessive soil can be drawn up into the needle of the auto sampler used for analysis thereby clogging and damaging the instrument.

- ***Do clean soil/sediment from the glass threads on the vial.***

When collecting the sample it is important to insure that there is no soil or grit on the thread or in the cap.

- ***Don't over tighten the cap.***

Over tightening may prevent the vial from sealing correctly. If there is not an airtight seal, the sample, internal standard, and surrogate recoveries will be compromised thereby invaliding the data.




Collect all samples in a manner that minimizes sample handling and agitation.

**Low concentration soil samples (< 200 µg/Kg or < .2 mg/Kg)**

1. Samples should be collected in **duplicate** 40ml VOA vials containing 5ml of deionized water and a stir bar provided by Spectrum Analytical.
2. During collection approximately 5 grams of soil must be added to the pre-measured, pre-weighed water vial. All sediment must be removed from the glass threads of the vial to ensure an adequate seal.

*Samples collected in water must be analyzed or frozen between -7º to -15º C within 48 hours of collection. All samples collected in water will be frozen upon receipt at the laboratory until the time of analysis.*

**High concentration soil samples (> 200 µg/Kg or > .2 mg/Kg)**

1. Samples should be collected in 40ml VOA vials containing 15ml of purge and trap grade methanol provided by Spectrum Analytical.
2. During collection approximately 15 grams of soil must be added to the pre-measured, pre-weighed sodium bisulfate vial. All sediment must be removed from the glass threads of the vial to ensure an adequate seal.

- Please do not cover the tare weight with your sample identification label.
- Store samples on ice at 4°C until transport to the laboratory facility.
- An additional VOA vial (40ml) must be collected for screening and dry weight determination. This vial must not contain any sample preservation solution.

*In order to alleviate uncertainties regarding which sample collection technique to use, Spectrum Analytical recommends the collection of all soil samples in accordance with both low and high concentration techniques.*

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# CLOSED-SYSTEM PURGE-AND-TRAP AND EXTRACTION FOR VOLATILE ORGANICS IN SOIL AND WASTE SAMPLES

## 1.0 SCOPE AND APPLICATION

1.1 This method describes a closed-system purge-and-trap process for the analysis of volatile organic compounds (VOCs) in solid materials (e.g., soils, sediments, and solid waste). While the method is designed for use on samples containing low levels of VOCs, procedures are also provided for collecting and preparing solid samples containing high concentrations of VOCs and for oily wastes. For these high concentration and oily materials, sample collection and preparation are performed using the procedures described here, and sample introduction is performed using the aqueous purge-and-trap procedure in Method 5030. These procedures may be used in conjunction with any appropriate determinative gas chromatographic procedure, including, but not limited to, Methods 8015, 8021, and 8260. The following compounds are appropriate for this sample preparation technique:

| Compound | CAS No.[a] | Response | Stability |
|---|---|---|---|
| Acetone | 67-64-1 | ht | hs |
| Acetonitrile | 75-05-8 | pp | nd |
| Acrolein (Propenal) | 107-02-8 | pp | ms |
| Acrylonitrile | 107-13-1 | pp | hs |
| Allyl alcohol | 107-18-6 | ht | nd |
| Allyl chloride | 107-05-1 | c | ms |
| t-Amyl ethyl ether (TAEE) | 919-94-8 | c / ht | nd |
| t-Amyl methyl ether (TAME) | 994-05-8 | c / ht | hs |
| Benzene | 71-43-2 | c | hs |
| Benzyl chloride | 100-44-7 | c | nd |
| Bis(2-chloroethyl)sulfide | 505-60-2 | pp | nd |
| Bromoacetone | 598-31-2 | pp | nd |
| Bromochloromethane | 74-97-5 | c | hs |
| Bromodichloromethane | 75-27-4 | c | ms |
| Bromoform | 75-25-2 | c | hs |
| Bromomethane | 74-83-9 | c | hvs |
| *n*-Butanol | 71-36-3 | ht | nd |
| 2-Butanone (MEK) | 78-93-3 | pp | hvs |
| *t*-Butyl alcohol | 75-65-0 | ht | nd |
| Carbon disulfide | 75-15-0 | pp | hvs |
| Carbon tetrachloride | 56-23-5 | c | hvs |
| Chloral hydrate | 302-17-0 | pp | nd |
| Chlorobenzene | 108-90-7 | c | hvs |
| Chlorodibromomethane | 124-48-1 | c | nd |
| Chloroethane | 75-00-3 | c | ms |

(continued)

| Compound | CAS No.[a] | Response | Stability |
|---|---|---|---|
| 2-Chloroethanol | 107-07-3 | pp | nd |
| 2-Chloroethyl vinyl ether | 110-75-8 | c | ls |
| Chloroform | 67-66-3 | c | hs |
| Chloromethane | 74-87-3 | c | hvs |
| Chloroprene | 126-99-8 | c | nd |
| Crotonaldehyde | 4170-30-3 | pp | nd |
| 1,2-Dibromo-3-chloropropane | 96-12-8 | pp | ms |
| 1,2-Dibromoethane | 106-93-4 | c | hs |
| Dibromomethane | 74-95-3 | c | hs |
| 1,2-Dichlorobenzene | 95-50-1 | c | hs |
| 1,3-Dichlorobenzene | 541-73-1 | c | ms |
| 1,4-Dichlorobenzene | 106-46-7 | c | ms |
| *cis*-1,4-Dichloro-2-butene | 1476-11-5 | c | nd |
| *trans*-1,4-Dichloro-2-butene | 110-57-6 | pp | ls |
| Dichlorodifluoromethane | 75-71-8 | c | hs |
| 1,1-Dichloroethane | 75-34-3 | c | hs |
| 1,2-Dichloroethane | 107-06-2 | c | hs |
| 1,1-Dichloroethene | 75-35-4 | c | hvs |
| *cis*-1,2-Dichloroethene | 156-59-4 | c | hs |
| *trans*-1,2-Dichloroethene | 156-60-5 | c | ms |
| 1,2-Dichloropropane | 78-87-5 | c | hs |
| 1,3-Dichloro-2-propanol | 96-23-1 | pp | nd |
| *cis*-1,3-Dichloropropene | 10061-01-5 | c | ls |
| *trans*-1,3-Dichloropropene | 10061-02-6 | c | ls |
| 1,2,3,4-Diepoxybutane | 1464-53-5 | c | nd |
| Diethyl ether | 60-29-7 | c | nd |
| Diisopropyl ether (DIPE) | 108-20-3 | c / ht | hs |
| 1,4-Dioxane | 123-91-1 | pp | nd |
| Ethylbenzene | 100-41-4 | c | hvs |
| Ethylene oxide | 75-21-8 | pp | nd |
| Ethyl methacrylate | 97-63-2 | c | ms |
| Ethyl *tert*-butyl ether (ETBE) | 637-92-3 | c / ht | hs |
| Hexachlorobutadiene | 87-68-3 | c | ms |
| 2-Hexanone | 591-78-6 | pp | hvs |
| Iodomethane | 74-88-4 | c | nd |
| Isobutyl alcohol | 78-83-1 | ht / pp | nd |
| Isopropylbenzene | 98-82-8 | c | ms |
| Malononitrile | 109-77-3 | pp | nd |
| Methacrylonitrile | 126-98-7 | pp | hs |
| Methylene chloride | 75-09-2 | c | hs |
| Methyl methacrylate | 80-62-6 | c | ms |
| 4-Methyl-2-pentanone (MIBK) | 108-10-1 | pp | ms |
| Methyl *tert*-butyl ether (MTBE) | 1634-04-4 | c / ht | hs |
| Naphthalene | 91-20-3 | c | ms |
| Nitrobenzene | 98-95-3 | c | nd |

(continued)

| Compound | CAS No.[a] | Response | Stability |
|---|---|---|---|
| 2-Nitropropane | 79-46-9 | c | nd |
| N-Nitroso-di-*n*-butylamine | 924-16-3 | pp | nd |
| Paraldehyde | 123-63-7 | pp | nd |
| 2-Pentanone | 107-87-9 | pp | nd |
| 2-Picoline | 109-06-8 | pp | nd |
| 1-Propanol | 71-23-8 | ht / pp | nd |
| 2-Propanol | 67-63-0 | ht / pp | nd |
| â-Propiolactone | 57-57-8 | pp | nd |
| Propionitrile (ethyl cyanide) | 107-12-0 | ht | nd |
| *n*-Propylamine | 107-10-8 | c | nd |
| Styrene | 100-42-5 | c | hvs |
| 1,1,1,2-Tetrachloroethane | 630-20-6 | c | hs |
| 1,1,2,2-Tetrachloroethane | 79-34-5 | c | nd |
| Tetrachloroethene | 127-18-4 | c | ms |
| Toluene | 108-88-3 | c | hs |
| *o*-Toluidine | 95-53-4 | pp | nd |
| 1,2,4-Trichlorobenzene | 120-82-1 | c | hs |
| 1,1,1-Trichloroethane | 71-55-6 | c | ms |
| 1,1,2-Trichloroethane | 79-00-5 | c | hs |
| Trichloroethene | 79-01-6 | c | ms |
| Trichlorofluoromethane | 75-69-4 | c | ls |
| 1,2,3-Trichloropropane | 96-18-4 | c | ls |
| Vinyl acetate | 108-05-4 | c | ls |
| Vinyl chloride | 75-01-4 | c | hvs |
| *o*-Xylene | 95-47-6 | c | hvs |
| *m*-Xylene | 108-38-3 | c | hvs |
| *p*-Xylene | 106-42-3 | c | hvs |

[a] Chemical Abstract Service Registry Number

c = Adequate response by this technique
ht = Method analyte only when purged at 80°C
pp = Poor purging efficiency resulting in high Estimated Quantitation Limits
nd = Not determined
hs = High stability in preserved water samples (> 60 days). Longer holding times may be appropriate, see Appendix A, Table A.1 footnote and ref. 47 for additional information
ms = Medium stability in preserved water samples (15 - 60 days). Longer holding times may be appropriate, see Appendix A, Table A.1 footnote and ref. 47 for additional information
ls = Low stability in preserved water samples (< 14 days), analyses should be performed as soon as possible.
hvs = Highly variable stability in preserved water samples. Longer holding times may be appropriate, see Appendix A, Table A.1 footnote and ref. 47 for additional information.

1.2 The low soil method utilizes a hermetically-sealed sample vial, the seal of which is never broken from the time of sampling to the time of analysis. Since the sample is never exposed to the atmosphere after sampling, the losses of VOCs during sample transport, handling, and analysis are minimized. The applicable concentration range of the low soil method is dependent on the determinative method, matrix, and compound. However, it will generally fall in the 0.5 to 200 µg/kg range.

1.3 Procedures are included for preparing high concentration samples for purging by Method 5030. High concentration samples are those containing VOC levels of >200 µg/kg.

1.4 Procedures are also included for addressing oily wastes that are soluble in a water-miscible solvent. These samples are also purged using Method 5030.

1.5 This method can be used for 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, quantitation limits (by GC or GC/MS) are significantly higher because of poor purging efficiency. The purging efficiency can be improved for water soluble analytes, e.g. ketones and alcohols, when purging at an elevated temperature of 80°C as compared to 20° or 40°C.

1.6 This method, in conjunction with Method 8015 (GC/FID), may be used for the analysis of the aliphatic hydrocarbon fraction in the light ends of total petroleum hydrocarbons, e.g., gasoline. For the aromatic fraction (BTEX), use this method and Method 8021 (GC/PID). A total determinative analysis of gasoline fractions may be obtained using Method 8021 in series with Method 8015.

1.7 As with any preparative method for volatiles, samples should be screened to avoid contamination of the purge-and-trap system by samples that contain very high concentrations of purgeable material above the calibration range of the low concentration method. In addition, because the sealed sample container cannot be opened to remove a sample aliquot without compromising the integrity of the sample, multiple sample aliquots should be collected to allow for screening and reanalysis.

1.8 The closed-system purge-and-trap equipment employed for low concentration samples is not appropriate for soil samples preserved in the field with methanol. Such samples should be analyzed using Method 5030 (see the note in Sec. 8.2.2).

1.9 Analysts should consult the disclaimer statement at the front of the manual and the information in Chapter Two for guidance on the intended flexibility in the choice of methods, apparatus, materials, reagents, and supplies, and on the responsibilities of the analyst for demonstrating that the techniques employed are appropriate for the analytes of interest, in the matrix of interest, and at the levels of concern.

In addition, analysts and data users are advised that, except where explicitly specified in a regulation, the use of SW-846 methods is *not* mandatory in response to Federal testing requirements. The information contained in this method is provided by EPA as guidance to be used by the analyst and the regulated community in making judgments necessary to generate results that meet the data quality objectives for the intended application.

1.10 Use of this method is restricted to use by, or under supervision of, appropriately experienced and trained laboratory analysts. Each analyst must demonstrate the ability to generate acceptable results with this method.

## 2.0 SUMMARY OF METHOD

2.1 Low concentration soil method - generally applicable to soils and other solid samples with VOC concentrations in the range of 0.5 to 200 µg/kg (refer to Appendix A for additional information).

Volatile organic compounds (VOCs) are determined by collecting an approximately 5-g sample and shipping to the laboratory or appropriate analysis site by the various methods outlined in Appendix A. To ensure minimal loss of volatile constituents prior to analysis the entire sample vial is placed, unopened with an unpierced septum, into the instrument auto sampler device. Immediately before analysis, organic-free reagent water, surrogates, and internal standards (if applicable) are automatically added without opening the sample vial. The vial containing the sample is heated to 40°C and the volatiles purged into an appropriate trap using an inert gas combined with agitation of the sample. Purged components travel via a transfer line to a trap. When purging is complete, the trap is heated and backflushed with helium to desorb the trapped sample components into a gas chromatograph for analysis by an appropriate determinative method.

2.2 High concentration method - generally applicable to soils and other solid samples with VOC concentrations greater than 200 µg/kg (refer to Appendix A for additional information).

The sample introduction technique in Sec. 2.1 is not applicable to all samples, particularly those containing high concentrations (generally greater than 200 µg/kg) of VOCs which may overload either the volatile trapping material or exceed the working range of the determinative instrument system (e.g., GC/MS, GC/FID, GC/ELCD, etc.). In such instances, this method describes two sample collection options and the corresponding sample purging procedures.

2.2.1 The first option is to collect an appropriate sample volume in a pre-weighed vial with a septum-sealed screw-cap (see Sec 6) that contains a water-miscible organic solvent (e.g., methanol). At the time of analysis, an aliquot of the solvent is removed from the vial and diluted into water along with the internal standards and surrogates, then purged using Method 5030 and analyzed by an appropriate determinative method.

2.2.2 The second option is to collect a bulk sample in a VOA vial without the use of a chemical preservative. A portion of that sample is removed from the container in the laboratory and is dispersed in a water-miscible solvent to dissolve the volatile organic constituents. An aliquot of the solution is added to reagent water in a purge tube. Surrogates and internal standards (if applicable) are added to the solution, then purged using Method 5030, and analyzed by an appropriate determinative method. Because the procedure involves opening the vial and removing a portion of the soil, a significant amount of volatile constituents may be lost during handling. (See Appendix A, Sec. 5.1 for additional details)

NOTE: Surrogate compounds may either be spiked into the solvent at the time of extraction or the reagent water containing an aliquot of the extract prior to analysis. Since the surrogate recovery data from these two options provides assurances of either extraction or analytical efficiencies, the decision as to when the surrogates are added depends on what questions need to be answered for a given sample matrix and the intended uses of the data.

2.3 High concentration oily waste method - generally applicable to oily samples with VOC concentrations greater than 200 µg/kg that can be diluted in a water-miscible solvent.

Samples that are comprised of oils or samples that contain significant amounts of oil present additional analytical challenges. This procedure is generally appropriate for such samples when they are soluble in a water-miscible solvent.

2.3.1 After demonstrating that a test aliquot of the sample is soluble in methanol or polyethylene glycol (PEG), a separate aliquot of the sample is spiked with surrogates and diluted in the appropriate solvent. An aliquot of the solution is added to 5 mL of reagent water in a purge tube, taking care to ensure that a floating layer of oil is not present in the purge tube. Internal standards (if applicable) are added to the solution which is then purged using Method 5030 and analyzed by an appropriate determinative method.

NOTE: Surrogate compounds may either be spiked into the solvent at the time of extraction or the reagent water containing an aliquot of the extract prior to analysis. Since the surrogate recovery data from these two options provides assurances of either extraction or analytical efficiencies, the decision as to when the surrogates are added depends on what questions need to be answered for a given sample matrix and the intended uses of the data.

2.3.2 Samples that contain oily materials that are not soluble in water-miscible solvents must be prepared according to Method 3585.

## 3.0 DEFINITIONS

Refer to Chapter One for a listing of applicable quality assurance/quality control (QA/QC) definitions.

## 4.0 INTERFERENCES

4.1 Impurities in the purge gas and from organic compounds out-gassing from the plumbing ahead of the trap account for the majority of contamination problems. The analytical system must be demonstrated to be free from contamination under the conditions of the analysis by running method blanks. The use of non-polytetrafluoroethylene (non-PTFE) plastic coating, non-PTFE thread sealants, or flow controllers with rubber components in the purging device must be avoided, since such materials out-gas organic compounds which can be concentrated in the trap during the purge operation. These compounds can result in interferences or false positives in the determinative step.

4.2 Samples can be contaminated by diffusion of volatile organics (particularly methylene chloride and fluorocarbons) through the septum seal of the sample vial during shipment and storage. A trip blank prepared from an appropriate organic-free matrix and sample container, and carried through sampling and handling protocols, serves as a check on such contamination.

4.3 Contamination by carryover can occur whenever high-concentration and low-concentration samples are analyzed in sequence. Where practical, samples with unusually high concentrations of analytes should be followed by an analysis of organic-free reagent water to check for cross-contamination. If the target compounds present in an unusually concentrated sample are also found to be present in the subsequent samples, the analyst must demonstrate that the compounds are not due to carryover. Conversely, if those target compounds are not present in the subsequent sample, then the analysis of organic-free reagent water is not necessary.

4.4 The laboratory where volatile analysis is performed should be completely free of solvents. Special precautions must be taken when analyzing for methylene chloride. The analytical and sample storage area should be isolated from all atmospheric sources of methylene chloride, otherwise random background levels will result. Since methylene chloride will permeate through PTFE tubing, all GC carrier gas lines and purge gas plumbing should be constructed of stainless steel or copper tubing. Laboratory workers' clothing previously exposed to methylene chloride fumes during common liquid/liquid extraction procedures can contribute to sample contamination. The presence of other organic solvents in the laboratory where volatile organics are analyzed can also lead to random background levels and the same precautions must be taken.

## 5.0 SAFETY

This method does not address all safety issues associated with its use. The laboratory is responsible for maintaining a safe work environment and a current awareness file of OSHA regulations regarding the safe handling of the chemicals included in this method. A reference file of material safety data sheets (MSDSs) should be available to all personnel involved in these analyses.

## 6.0 EQUIPMENT AND SUPPLIES

### 6.1 Sample containers

The specific sample containers required will depend on the purge-and-trap system to be employed (see Sec. 6.2). Several systems are commercially available. Some systems employ 40-mL clear vials with a special frit and equipped with two PTFE-faced silicone septa. Other systems permit the use of any good quality glass vial that is large enough to contain at least 5 g of soil or solid material and at least 10 mL of water and that can be sealed with a screw-cap containing a PTFE-faced silicone septum. Consult the purge-and-trap system manufacturer's instructions regarding the suitable specific vials, septa, caps, and mechanical agitation devices. Additional information on sample containers can be found in Appendix A, Secs. 1.6, 3.0, 7.0 and 8.0.

### 6.2 Purge-and-trap system

The purge-and-trap system consists of a unit that automatically adds water, surrogates, and internal standards (if applicable) to a vial containing the sample, purges the VOCs using an inert gas stream while agitating the contents of the vial, and also traps the released VOCs for subsequent desorption into the gas chromatograph. Such systems are commercially available from several sources and shall meet the following specifications.

6.2.1 The purging device should be capable of accepting a vial sufficiently large enough to contain a 5-g soil sample plus a magnetic stirring bar and 10 mL of water. The device must be capable of heating a soil vial to 40°C and holding it at that temperature while the inert purge gas is allowed to pass through the sample. The device should also be capable of introducing at least 5 mL of organic-free reagent water into the sample vial while trapping the displaced headspace vapors. It must also be capable of agitating the sealed sample during purging, (e.g., using a magnetic stirring bar added to the vial prior to sample collection, sonication, or other means). The analytes being purged must be quantitatively transferred to an absorber trap. The trap must be capable of transferring the absorbed VOCs to the gas chromatograph (see 6.2.2).

NOTE: The equipment used to develop this method was a Dynatech PTA-30 W/S Autosampler. This device was subsequently sold to Varian, and is now available as the Archon Purge and Trap Autosampler. See the Disclaimer at the front of this manual for guidance on the use of alternative equipment.

6.2.2 A variety of traps and trapping materials may be employed with this method. The choice of trapping material may depend on the analytes of interest. Whichever trap is employed, it must demonstrate sufficient adsorption and desorption characteristics to meet the quantitation limits of all desired target analytes for a given project and the QC requirements in Method 8000 and the determinative method. The most difficult analytes are generally the gases, especially dichlorodifluoromethane. The trap must be capable of desorbing the late eluting target analytes.

NOTE: Check the responses of the brominated compounds when using alternative charcoal traps (especially Vocarb 4000, Supelco, Inc., Bellefonte, PA), as some degradation has been noted when higher desorption temperatures (especially above 240 - 250°C) are employed. 2-Chloroethyl vinyl ether is degraded on Vocarb 4000 but performs adequately when Vocarb 3000 (Supelco, Inc., Bellefonte, PA) is used. The primary criterion, as stated above, is that all target analytes meet the sensitivity requirements for a given project.

6.2.2.1 The trap used to develop this method was 25 cm long, with an inside diameter of 0.105 inches, and was packed with Carbopack/Carbosieve (Supelco, Inc., Bellefonte, PA).

6.2.2.2 The standard trap used in other EPA purge-and-trap methods is also acceptable. That trap is 25 cm long and has an inside diameter of at least 0.105 in. Starting from the inlet, the trap contains the equal amounts of the adsorbents listed below. It is recommended that 1.0 cm of methyl silicone-coated packing (35/60 mesh, Davison, grade 15 or equivalent) be inserted at the inlet to extend the life of the trap. If the analysis of dichlorodifluoromethane or other fluorocarbons of similar volatility is not required, then the charcoal can be eliminated and the polymer increased to fill 2/3 of the trap. If only compounds boiling above 35°C are to be analyzed, both the silica gel and charcoal can be eliminated and the polymer increased to fill the entire trap.

6.2.2.2.1 2,6-Diphenylene oxide polymer - 60/80 mesh, chromatographic grade (Tenax GC or equivalent).

6.2.2.2.2 Methyl silicone packing - OV-1 (3%) on Chromosorb-W, 60/80 mesh or equivalent.

6.2.2.2.3 Coconut charcoal - Prepare from Barnebey Cheney, CA-580-26, or equivalent, by crushing through 26 mesh screen.

6.2.2.3 Trapping materials other than those listed above also may be employed, provided that they meet the specifications as noted above.

6.2.3 The desorber for the trap must be capable of rapidly heating the trap to the temperature recommended by the trap material manufacturer, prior to the beginning of the flow of desorption gas. Several commercial desorbers (purge-and-trap units) are available.

6.3 Syringe and syringe valves

6.3.1 25-mL glass hypodermic syringes with Luer-Lok (or equivalent) tip (other sizes are acceptable depending on sample volume used).

6.3.2 2-way syringe valves with Luer ends.

6.3.3 25-µL micro syringe with a 2-inch x 0.006-inch ID, 22° bevel needle (Hamilton #702N or equivalent).

6.3.4 Micro syringes - 10-, 100-µL.

6.3.5 Syringes - 0.5-, 1.0-, and 5-mL, gas-tight with shut-off valve.

6.4 Miscellaneous

6.4.1 Glass vials

6.4.1.1 60-mL, septum-sealed, to collect samples for screening, moisture determination.

6.4.1.2 40-mL, screw-cap, PTFE lined, septum-sealed. Examine each vial prior to use to ensure that the vial has a flat, uniform sealing surface.

6.4.2 Top-loading balance - Capable of accurately weighing to 0.01 g.

6.4.3 Glass scintillation vials - 20-mL, with screw-caps and PTFE liners, or glass culture tubes with screw-caps and PTFE liners, for dilution of oily waste samples.

6.4.4 Volumetric flasks - Class A, 10-mL and 100-mL, with ground-glass stoppers.

6.4.5 2-mL glass vials, for GC autosampler - Used for oily waste samples extracted with methanol or PEG.

6.4.6 Spatula, stainless steel - narrow enough to fit into a sample vial.

6.4.7 Disposable Pasteur pipettes.

6.4.8 Magnetic stirring bars - PTFE- or glass-coated, of the appropriate size to fit the sample vials. Consult manufacturer's recommendation for specific stirring bars. Stirring bars may be reused, provided that they are thoroughly cleaned between uses. Consult the manufacturers of the purging device and the stirring bars for suggested cleaning procedures.

6.5 Field sampling equipment

6.5.1 Purge-and-trap soil sampler - Model 3780PT (Associated Design and Manufacturing Company, Alexandria, VA), or equivalent.

6.5.2 EnCore™ sampler - (En Novative Technologies, Inc., Green Bay, WI), or equivalent.

6.5.3 Terra Core™ sampler - (En Novative Technologies, Inc., Green Bay, WI), or equivalent.

6.5.4 EasyDraw™ syringe and PowerStop™ handle - (US Oil Company, Inc., Kimberly, WI), or equivalent.

6.5.5 Alternatively, disposable plastic syringes with a barrel smaller than the neck of the soil vial may be used to collect the sample. The syringe end of the barrel is cut off prior to sampling. One syringe is needed for each sample aliquot to be collected.

6.5.4 Portable balance - For field use, capable of weighing to 0.01 g.

6.5.5 Balance weights - Balances employed in the field should be checked against an appropriate reference weight at least once daily, prior to weighing any samples, or as described in the sampling plan. The specific weights used will depend on the total weight of the sample container, sample, stirring bar, reagent water added, cap, and septum.

6.5.6 Additional types of field sampling equipment and accessories are described in Appendix A, Secs. 1.6 and 7.0.

## 7.0 REAGENTS AND STANDARDS

7.1 Organic-free reagent water - All references to water in this method refer to organic-free reagent water, as defined in Chapter One.

7.2 Methanol, $CH_3OH$ - purge-and-trap quality or equivalent. Store away from other solvents.

7.3 Polyethylene glycol (PEG), $H(OCH_2CH_2)_nOH$ - free of interferences at the detection limit of the target analytes.

7.4 Low concentration sample preservative

7.4.1 For determination as to whether sample preservation is necessary and for selection of appropriate preservation options, see Appendix A, Secs. 1.2, 1.3, 3.0 and 8.0.

7.4.2 Sodium bisulfate, $NaHSO_4$ - ACS reagent grade or equivalent.

7.4.3 The preservative, if necessary, should be added to the vial prior to shipment to the field, and must be present in the vial prior to adding the sample.

7.5 See the determinative method and Method 5000 for guidance on internal standards and surrogates to be employed in this procedure. The recommended surrogates are 4-bromofluorobenzene, 1,2-dichloroethane-$d_4$, and toluene-$d_8$. Other compounds may be used as surrogates, depending upon the analysis requirements and the specific target analytes. The recommended internal standards are chlorobenzene-$d_5$, 1,4-dichlorobenzene-$d_4$, and fluorobenzene. Other compounds may be used as internal standards as long as they have retention times similar to the target analytes being detected.

## 8.0 SAMPLE COLLECTION, PRESERVATION, AND STORAGE

Refer to the introductory material in this chapter, Organic Analytes, Sec. 4.1, and Appendix A for general sample collection information. The low concentration portion of this method employs sample vials that are filled and weighed in the field and never opened during the analytical process.

As a result, sampling personnel should be equipped with a portable balance capable of weighing to 0.01 g.

8.1 Preparation of sample vials

The specific preparation procedures for sample vials depend on the expected concentration range of the sample, with separate preparation procedures for low concentration soil samples and high concentration soil and solid waste samples. Sample vials should be prepared in a fixed laboratory or other controlled environment, sealed, and shipped to the field location. Gloves should be worn during the preparation steps. More detailed information on additional options for the preparation of sample vials can be found in Appendix A, Secs. 3.0, 7.0, and 8.0.

8.1.1 Low concentration soil samples

The following steps apply to the preparation of vials used in the collection of low concentration soil samples to be analyzed by the closed-system purge-and-trap equipment described in this method.

8.1.1.1 Add a clean magnetic stirring bar to each clean vial. If the purge-and-trap device (Sec. 6.2) employs a means of stirring the sample other than a magnetic stirrer (e.g., sonication or other mechanical means), then the stir bar is omitted.

8.1.1.2 Add preservative, if necessary, (See Appendix A, Secs. 1.2, 1.3, 3.0 and 8.0) to each vial. The preservative is added to each vial prior to shipping the vial to the field. Add approximately 1 g of sodium bisulfate to each vial. If samples markedly smaller or larger than 5 g are to be collected, adjust the amount of preservative added to correspond to approximately 0.2 g of preservative for each 1 g of sample. Enough sodium bisulfate should be present to ensure a sample pH of $\leq$2.

8.1.1.3 Add 5 mL of organic-free reagent water to each vial. The water and the preservative will form an acid solution that will reduce or eliminate the majority of the biological activity in the sample, thereby preventing biodegradation of the volatile target analytes.

8.1.1.4 Seal the vial with the screw-cap and septum seal. If the double-ended, fritted, vials are used, seal both ends as recommended by the manufacturer.

8.1.1.5 Affix a label to each vial. This eliminates the need to label the vials in the field and assures that the tare weight of the vial includes the label. (The weight of any markings added to the label in the field is negligible).

8.1.1.6 Weigh the prepared vial to the nearest 0.01 g, record the tare weight, and write it on the label.

8.1.1.7 Because volatile organics will partition into the headspace of the vial from the aqueous solution and will be lost when the vial is opened, surrogates, matrix spikes, and internal standards (if applicable) should only be added to the vials after the sample has been added to the vial. These standards should be introduced back in the laboratory, either manually by puncturing the septum with a small-gauge needle or automatically by the sample introduction system, just prior to analysis.

8.1.2 High concentration soil samples collected without a preservative

When high concentration samples are collected without a preservative, a variety of sample containers may be employed, including 60-mL glass vials with septum seals (see Sec. 6.4). More detailed information on additional options for the preparation of sample vials can be found in Appendix A, Secs. 3.0, 7.0, and 8.0.

8.1.3 High concentration soil samples collected and preserved in the field

The following steps apply to the preparation of vials used in the collection of high concentration soil samples to be preserved in the field with methanol and analyzed by the aqueous purge-and-trap equipment described in Method 5030. See the water-miscible solvent dilution effect information in Sec. 11.5 and Method 8000 for guidance on correcting results for data reporting purposes. More detailed information on additional options for the preparation of sample vials can be found in Appendix A, Secs. 3.0, 7.0, and 8.0.

8.1.3.1 Add 10 mL of methanol to each vial.

8.1.3.2 Seal the vial with the screw-cap and septum seal.

8.1.3.3 Affix a label to each vial. This eliminates the need to label the vials in the field and assures that the tare weight of the vial includes the label. (The weight of any markings added to the label in the field is negligible).

8.1.3.4 Weigh the prepared vial to the nearest 0.01 g, record the tare weight, and write it on the label.

NOTE: Vials containing methanol should be weighed a second time on the day that they are to be used. Vials found to have lost methanol (reduction in weight of >0.01 g) should not be used for sample collection.

8.1.3.5 Surrogates, internal standards and matrix spikes (if applicable) should be added to the sample after it is returned to the laboratory and prior to analysis.

8.1.4 Oily waste samples

When oily waste samples are known to be soluble in methanol or PEG, sample vials may be prepared as described in Sec. 8.1.3, using the appropriate solvent. However, when the solubility of the waste is unknown, the sample should be collected without the use of a preservative, in a vial such as that described in Sec. 8.1.2.

8.2 Sample collection

Collect the sample according to the procedures outlined in the sampling plan. As with any sampling procedure for volatiles, care must be taken to minimize the disturbance of the sample in order to minimize the loss of the volatile components. Several techniques may be used to transfer a sample to the relatively narrow opening of the low concentration soil vial. These include devices such as the EnCore™ sampler, the Purge-and-Trap Soil Sampler ™, or any other sampling device listed in Sec. 6.5, or equivalent. Always wear gloves whenever handling the tared sample vials. More detailed information and additional sample collection options can be found in Appendix A, Sec. 7.0.

8.2.1 Low concentration soil samples

8.2.1.1 Volatile organic compounds (VOCs) are determined by collecting an approximately 5-g sample and shipping to the laboratory or appropriate analysis site by the various methods outlined in Appendix A. Using an appropriate sample collection device, collect approximately 5 g of sample as soon as possible after the surface of the soil or other solid material has been exposed to the atmosphere: generally within a few minutes at most. Carefully wipe the exterior of the sample collection device with a clean cloth or towel.

8.2.1.2 Using the sample collection device, add about 5 g (2 - 3 cm) of soil to the sample vial containing the preservative solution or other preservation options as discussed in Appendix A. Quickly brush any soil off the vial threads and immediately seal the vial with the septum and screw-cap. Store samples on ice at 4°C. Alternatively, samples can be collected into an empty vial or vial containing reagent water (with or without preservative) and stored frozen at < -7°C.

NOTE: Soil samples that contain carbonate minerals (either from natural sources or applied as an amendment) may effervesce upon contact with the acidic preservative solution option in the low concentration sample vial. If the amount of gas generated is very small (i.e., several mL), any loss of volatiles as a result of such effervescence may be minimal if the vial is sealed quickly. However, if larger amounts of gas are generated, not only may the sample lose a significant amount of analyte, but the gas pressure may shatter the vial if the sample vial is sealed. Therefore, when samples are known or suspected to contain high levels of carbonates, a test sample should be collected, added to a vial, and checked for effervescence. If a rapid or vigorous reaction occurs, discard the sample and collect low concentration samples in vials without chemical preservation.

8.2.1.3 When practical, use a portable balance to weigh the sealed vial containing the sample to ensure that 5.0 ± 0.5 g of sample were added. The balance should be calibrated in the field using an appropriate weight for the sample containers employed (Sec. 6.5.5). Record the weight of the sealed vial containing the sample to the nearest 0.01 g.

8.2.1.4 Alternatively, collect several trial samples with plastic syringes. Weigh each trial sample and note the length of the soil column in the syringe. Use these data to determine the length of soil in the syringe that corresponds to 5.0 ± 0.5 g. Discard each trial sample.

8.2.1.5 As with the collection of aqueous samples for volatiles, collect at least two replicate samples. This will allow the laboratory an additional sample for reanalysis, if needed. The second sample should be taken from the same soil stratum or the same section of the solid waste being sampled, and within close proximity to the location from which the original sample was collected.

8.2.1.6 In addition, since the soil vial cannot be opened without compromising the integrity of the sample, at least one additional aliquot of sample must be collected for screening, moisture determination, and high concentration analysis (if necessary). This third aliquot may be collected in a 60-mL glass vial or a third 40-mL soil sample vial. However, this third vial must *not* contain the sample preservative solution, as an aliquot will be used to determine % moisture. If high concentration samples are collected in

vials containing methanol, then two additional aliquots should be collected, one for high concentration analysis collected in a vial containing methanol, and another for the moisture determination in a vial without either methanol or the low concentration aqueous preservative solution.

8.2.1.7 If samples are known or expected to contain target analytes over a wide range of concentrations, thereby requiring the analyses of multiple sample aliquots, it may be advisable and practical to take an additional sample aliquot in a low concentration soil vial containing the preservative, but collecting only 1-2 g instead of the 5 g collected in Sec. 8.2.1.1. This aliquot may be used for those analytes that exceed the instrument calibration range in the 5-g analysis.

8.2.1.8 The EnCore™ sampler has not been thoroughly evaluated by EPA as a sample storage device. While preliminary results indicate that storage in the EnCore™ device may be appropriate for up to 48 hours, samples collected in this device should be transferred to the soil sample vials as soon as possible, or analyzed within 48 hours.

8.2.1.9 The collection of low concentration soil samples in vials that contain methanol is not appropriate for samples analyzed with the closed-system purge-and-trap equipment described in this method (see Sec. 8.2.2).

8.2.2 High concentration soil samples preserved in the field

The collection of soil samples in vials that contain methanol has been suggested by some as a combined preservation and extraction procedure. However, this procedure is not appropriate for use with the low concentration soil procedure described in this method.

NOTE: The use of methanol preservation has not been formally evaluated by EPA and analysts must be aware of three potential problems. First, the use of methanol as a preservative and extraction solvent introduces a significant dilution factor that will raise the method quantitation limit beyond the operating range of the low concentration direct purge-and-trap procedure (0.5-200 µg/kg). The exact dilution factor will depend on the masses of solvent and sample, but generally exceeds 100, and may make it difficult to demonstrate compliance with regulatory limits or action levels for some analytes. Because the analytes of interest are volatile, the methanol extract cannot be concentrated to overcome the dilution problem. Thus, for samples of unknown composition, it may still be necessary to collect an aliquot for analysis by this closed-system procedure and another aliquot preserved in methanol and analyzed by other procedures. Secondly, solid samples with a significant moisture content (>10%) that are extracted prior to analysis in a water miscible solvent such as methanol are diluted by the total volume of the solvent/water mixture. (see Sec. 11.5 and Method 8000) The final problem is that the addition of methanol to the sample is likely to cause the sample to fail the ignitability characteristic, or cause it to become a listed waste, thereby requiring the unused sample volume to be managed as a hazardous waste.

8.2.2.1 When samples are known to contain volatiles at concentrations high enough that the dilution factor will not preclude obtaining results within the calibration range of the appropriate determinative method, a sample may be collected and immediately placed in a sample vial containing purge-and-trap grade methanol.

8.2.2.2 Using an appropriate sample collection device, collect approximately 5 g of sample as soon as possible after the surface of the soil or other solid material has been exposed to the atmosphere: generally within a few minutes at most. Carefully wipe the exterior of the sample collection device with a clean cloth or towel.

8.2.2.3 Using the sample collection device, add about 5 g (2 - 3 cm) of soil to the vial containing 10 mL of methanol. Quickly brush any soil off the vial threads and immediately seal the vial with the septum and screw-cap. Store samples on ice at 4 °C.

8.2.2.4 When practical, use a portable balance to weigh the sealed vial containing the sample to ensure that 5.0 ± 0.5 g of sample were added. The balance should be calibrated in the field using an appropriate weight for the sample containers employed (Sec. 6.5.5). Record the weight of the sealed vial containing the sample to the nearest 0.01 g.

8.2.2.5 Alternatively, collect several trial samples with plastic syringes. Weigh each trial sample and note the length of the soil column in the syringe. Use these data to determine the length of soil in the syringe that corresponds to 5.0 ± 0.5 g. Discard each trial sample.

8.2.2.6 Other sample weights and volumes of methanol may be employed, provided that the analyst can demonstrate that the sensitivity of the overall analytical procedure is appropriate for the intended application.

8.2.2.7 The collection of at least one additional sample aliquot is required for the determination of the moisture content, as described in Sec. 6.2.1.6. Samples collected in methanol should be shipped as described in Sec. 6.3, and must be clearly labeled as containing methanol, so that the samples are not analyzed using the closed-system purge-and-trap equipment described in this procedure.

8.2.3 High concentration sample <u>not</u> preserved in the field

The collection of high concentration bulk samples, i.e., wastes containing percent level concentrations, that are not preserved in the field generally follows similar procedures as for the other types of samples described in Secs. 8.2.1 and 8.2.2, with the obvious exception that the sample vials contain neither the aqueous preservative solution nor methanol. However, when field preservation is not employed, it is better to collect a larger volume sample, filling the sample container as full as practical in order to minimize the headspace. Such collection procedures generally do not require the collection of a separate aliquot for moisture determination, but it may be advisable to collect a second sample aliquot for screening purposes, in order to minimize the loss of volatiles in either aliquot.

8.2.4 Oily waste samples

The collection procedures for oily samples depend on knowledge of the waste and its solubility in methanol or other solvents.

8.2.4.1 When an oily waste is <u>known</u> to be soluble in methanol or PEG, the sample may be collected in a vial containing such a solvent (see Sec. 8.1.4), using procedures similar to those described in Sec. 8.2.2.

8.2.4.2 When the solubility of the oily waste is <u>not</u> known, the sample should either be collected in a vial without a preservative, as described in Sec. 8.2.3, or the

solubility of a trial sample should be tested in the field, using a vial containing solvent. If the trial sample is soluble in the solvent, then collect the oily waste sample as described in Sec. 8.2.2. Otherwise, collect an unpreserved sample as described in Sec. 8.2.3.

8.3 Sample handling and shipment

All samples for volatiles analysis should be cooled to approximately 4°C, packed in appropriate containers, and shipped to the laboratory on ice, as described in the sampling plan. See Appendix A, Secs. 3.0, 7.0, and 8.0 for additional sample handling options.

8.4 Sample storage

8.4.1 Once in the laboratory, store samples at the recommended temperature until analysis (refer to Appendix A, Secs. 3.0 and 7.4 for additional sample storage information). The sample storage area should be free of organic solvent vapors.

8.4.2 All samples should be analyzed as soon as practical, and within the designated holding time from collection. Samples not analyzed within the designated holding time must be noted and the data are considered minimum values.

8.4.3 When the low concentration samples are strongly alkaline or highly calcareous in nature, the sodium bisulfate preservative solution may not be strong enough to reduce the pH of the soil/water solution to below 2. Therefore, when low concentration soils to be sampled are known or suspected to be strongly alkaline or highly calcareous, additional steps may be required to preserve the samples. Such steps include: addition of larger amounts of the sodium bisulfate preservative to non-calcareous samples, storage of low concentration samples at <-7°C (taking care not to fill the vials so full that the expansion of the water in the vial breaks the vial), or significantly reducing the maximum holding time for low concentration soil samples. Whichever steps are employed, they should be clearly described in the sampling and QA project plans and distributed to both the field and laboratory personnel. See Sec. 8.2.1.2 for additional information.

8.4.4 See Appendix A, Secs. 3.0, 7.0, and 8.0 for additional sample storage options.

## 9.0 QUALITY CONTROL

9.1 Refer to Chapter One for guidance on quality assurance (QA) and quality control (QC) protocols and Method 5000 for sample preparation QC procedures. Each laboratory should maintain a formal quality assurance program. The laboratory should also maintain records to document the quality of the data generated. All data sheets and quality control data should be maintained for reference or inspection. When inconsistencies exist between QC guidelines, method-specific QC criteria take precedence over both technique-specific criteria and those criteria given in Chapter One, and technique-specific QC criteria take precedence over the criteria in Chapter One.

9.2 Before processing any samples, the analyst should demonstrate through the analysis of an organic-free reagent water method blank that all glassware and reagents are interference free. Each time a set of samples is extracted, or there is a change in reagents, a method blank should be processed as a safeguard against chronic laboratory contamination. The blank samples should be carried through all stages of the sample preparation and measurement.

9.3 Initial demonstration of proficiency - Each laboratory must demonstrate initial proficiency with each sample preparation and determinative method combination it utilizes, by generating data of acceptable accuracy and precision for target analytes in a clean matrix. The laboratory must also repeat this demonstration whenever new staff are trained or significant changes in instrumentation are made. See the Quality Control Section of Methods 5000 and 8000 for information on how to accomplish this demonstration.

9.4 Sample quality control for preparation and analysis - See the Quality Control Section of Method 5000 and Method 8000 for procedures to follow to demonstrate acceptable continuing performance on each set of samples to be analyzed. These include the method blank, either a matrix spike/matrix spike duplicate or a matrix spike and duplicate sample analysis, a laboratory control sample (LCS), and the addition of surrogates to each sample and QC sample.

9.5 It is recommended that the laboratory adopt additional quality assurance practices for use with this method. The specific practices that are most productive depend upon the needs of the laboratory and the nature of the samples. Whenever possible, the laboratory should analyze standard reference materials and participate in relevant performance evaluation studies.

9.6 The laboratory should have quality control procedures to make sure that sample integrity is not compromised during the sample collection and sample handling process, e.g., making sure that septa and vial caps do not leak, etc. (See Appendix A, Secs. 1.6 and 7.1.1) In addition, it would be advisable for the laboratory to monitor the internal standard's (IS) area counts for the low concentration samples, since leaks attributed to a poor seal with the vial caps and septa will be evident by low IS area counts. Sample containers and data results for instances where low IS area counts are observed and leaks are suspected, should be discarded.

## 10.0 CALIBRATION AND STANDARDIZATION

Refer to the appropriate determinative method for calibration and standardization procedures.

## 11.0 PROCEDURE

This section describes procedures for sample screening, the low concentration soil method, the high concentration soil method, and the procedure for oily waste samples. High concentration samples are to be introduced into the GC system using Method 5030. Oily waste samples are to be introduced into the GC system using Method 5030 if they are soluble in a water-miscible solvent, or using Method 3585 if they are not.

### 11.1 Sample screening

11.1.1 It is highly recommended that all samples be screened prior to the purge-and-trap GC or GC/MS analysis. Samples may contain higher than expected quantities of purgeable organics that will contaminate the purge-and-trap system, thereby requiring extensive cleanup and instrument maintenance. The screening data are used to determine which is the appropriate sample preparation procedure for the particular sample, the low concentration closed-system direct purge-and-trap method (Sec. 11.2), the high concentration (methanol extraction) method (Sec. 11.3), or the nonaqueous liquid (oily waste) methanol or PEG dilution procedure (Sec. 11.4).

11.1.2 The analyst may employ any appropriate screening technique. Three suggested screening techniques employing SW-846 methods are:

11.1.2.1 Automated headspace (Method 5021) using a gas chromatograph (GC) equipped with an appropriate detector,

11.1.2.2 Screening with a portable photoionization detector (PID) (Method 3815) or,

11.1.2.3 Extraction of the sample with hexadecane (Method 3820) and analysis of the extract on a GC equipped with a FID and/or an ECD.

11.1.3 The analyst may inject a calibration standard containing the analytes of interest at a concentration equivalent to the upper limit of the calibration range of the low concentration soil method. The results from this standard may be used to determine when the screening results approach the upper limit of the low concentration soil method. There are no linearity or other performance criteria associated with the injection of such a standard, and other approaches may be employed to estimate sample concentrations.

11.1.4 Use the low concentration closed-system purge-and-trap method (Sec. 11.2) if the estimated concentration from the screening procedure falls within the calibration range of the selected determinative method. If the concentration exceeds the calibration range of the low concentration soil method, then use either the high concentration soil method (Sec. 11.3), or the oily waste method (Sec. 11.4).

11.2 Low concentration soil method (Approximate concentration range of 0.5 to 200 µg/kg - the concentration range is dependent upon the determinative method and the sensitivity of each analyte.)

11.2.1 Initial set-up

Prior to using this introduction technique for any GC or GC/MS method, the system must be calibrated. General calibration procedures are discussed in Method 8000, while the determinative methods and Method 5000 provide specific information on calibration and preparation of standards. Normally, external standard calibration is preferred for the GC methods (non-MS detection) because of possible interference problems with internal standards. If interferences are not a problem, or when a GC/MS method is used, internal standard calibration may be employed.

11.2.1.1 Assemble a purge-and-trap device that meets the specification in Sec. 6.2 and that is connected to a gas chromatograph or a gas chromatograph/mass spectrometer system.

11.2.1.2 Before initial use, a Carbopack/Carbosieve trap should be conditioned overnight at 245°C by baking out with an inert gas flow of at least 20 mL/minute. If other trapping materials are substituted for the Carbopack/Carbosieve, follow the manufacturers recommendations for conditioning. Vent the trap effluent to the hood, not to the analytical column. Prior to daily use, the trap should be conditioned by baking for 10 minutes at 245°C. The trap may be vented to the analytical column during daily conditioning; however, the column must be run through the temperature program prior to analysis of samples.

11.2.1.3 If the standard trap in Sec. 6.2.2.2 is employed, prior to initial use, the trap should be conditioned overnight at 180°C by baking out with an inert gas flow of at least 20 mL/min, or according to the manufacturer's recommendations. Vent the trap effluent to the hood, not to the analytical column. Prior to daily use, the trap should be

conditioned by baking for 10 min at 180°C. The trap may be vented to the analytical column during daily conditioning; however, the column must be run through the temperature program prior to analysis of samples.

11.2.1.4 Establish the purge-and-trap instrument operating conditions. Adjust the instrument to inject 5 mL of water, to heat the sample to 40°C, and to hold the sample at 40°C for 1.5 minutes before commencing the purge process, or as recommended by the instrument manufacturer.

11.2.1.5 Prepare a minimum of five initial calibration standards containing all the analytes of interest and surrogates, as described in Method 8000, and following the instrument manufacturer's instructions. The calibration standards are prepared in organic-free reagent water. The volume of organic-free reagent water used for calibration must be the same volume used for sample analysis (normally 5 mL added to the vial before shipping it to the field plus the organic-free reagent water added by the instrument). When the sodium bisulfate preservation technique is used, the calibration standards should also contain approximately the same amount of the sodium bisulfate preservative as the sample (e.g., ~1 g), as the presence of the preservative will affect the purging efficiencies of the analytes. The internal standard solution must be added automatically, by the instrument, in the same fashion as used for the samples. Place the soil vial containing the solution in the instrument carousel. In order to calibrate the surrogates using standards at five concentrations, it may be necessary to disable the automatic addition of surrogates to each vial containing a calibration standard (consult the manufacturer's instructions). Prior to purging, heat the sample vial to 40°C for 1.5 minutes, or as recommended by the manufacturer.

11.2.1.6 Carry out the purge-and-trap procedure as outlined in Secs. 11.2.3. to 11.2.5.

11.2.1.7 Calculate calibration factors (CF) or response factors (RF) for each analyte of interest using the procedures described in Method 8000. Calculate the average CF (external standards) or RF (internal standards) for each compound, as described in Method 8000. Evaluate the linearity of the calibration data, or choose another calibration model, as described in Method 8000 and the specific determinative method.

11.2.1.8 For GC/MS analysis, a system performance check must be made before this calibration curve is used (see Method 8260). If the purge-and-trap procedure is used with Method 8021, evaluate the response for the following four compounds: chloromethane; 1,1-dichloroethane; bromoform; and 1,1,2,2-tetrachloroethane. They are used to check for proper purge flow and to check for degradation caused by contaminated lines or active sites in the system.

11.2.1.8.1 Chloromethane is the most likely compound to be lost if the purge flow is too fast.

11.2.1.8.2 Bromoform 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.

11.2.1.8.3 Tetrachloroethane and 1,1-dichloroethane are degraded by contaminated transfer lines in purge-and-trap systems and/or active sites in trapping materials.

11.2.1.9 When analyzing for very late eluting compounds with Method 8021 (i.e., hexachlorobutadiene, 1,2,3-trichlorobenzene, etc.), cross-contamination and memory effects from a high concentration sample or even the standard are a common problem. Extra rinsing of the purge vessel after analysis normally corrects this. The newer purge-and-trap systems often overcome this problem with better bake-out of the system following the purge-and-trap process. Also, the charcoal traps retain less moisture and decrease the problem.

11.2.2 Calibration verification (see appropriate determinative method)

Refer to Method 8000 for details on calibration verification. A single standard near the mid-point of calibration range is used for verification. This standard should also contain approximately 1 g of sodium bisulfate if the samples are also preserved in this manner.

11.2.3 Sample purge-and-trap

This method is designed for a 5-g sample size, but smaller sample sizes may be used. Consult the instrument manufacturer's instructions regarding larger sample sizes, in order to avoid clogging of the purging apparatus. The soil vial is hermetically sealed at the sampling site, and MUST remain so in order to guarantee the integrity of the sample. Gloves must be worn when handling the sample vial since the vial has been tared. If any soil is noted on the exterior of the vial or cap, it must be carefully removed prior to weighing. Weigh the vial and contents to the nearest 0.01 g, even if the sample weight was determined in the field, and record this weight. This second weighing provides a check on the field sampling procedures and provides additional assurance that the reported sample weight is accurate. Data users should be advised on significant discrepancies between the field and laboratory weights.

11.2.3.1 Remove the sample vial from storage and allow it to warm to room temperature. Shake the vial gently, to ensure that the contents move freely and that stirring will be effective. Place the sample vial in the instrument carousel according to the manufacturer's instructions.

11.2.3.2 Without disturbing the hermetic seal on the sample vial, add 5 mL of organic-free reagent water, the internal standards, and the surrogate compounds. This is carried out using the automated sampler. Other volumes of organic-free reagent water may be used, however, it is imperative that all samples, blanks, and calibration standards have exactly the same final volume of organic-free reagent water. Prior to purging, heat the sample vial to 40°C for 1.5 minutes, or as described by the manufacturer.

11.2.3.3 For the sample selected for matrix spiking, add the matrix spiking solution described in the Reagents Section of Method 5000, either manually, or automatically, following the manufacturer's instructions. The concentration of the spiking solution and the amount added should be established as described in the Quality Control Section of Method 8000.

11.2.3.4 Purge the sample with helium or another inert gas at a flow rate of up to 40 mL/minute (the flow rate may vary from 20 to 40 mL/min, depending on the target analyte group) for the appropriate purge time (usually 11 minutes) while the sample is being agitated with the magnetic stirring bar or other mechanical means. The purged analytes are allowed to flow out of the vial through a glass-lined transfer line to a trap packed with suitable sorbent materials.

11.2.4 Sample desorption

11.2.4.1 Non-cryogenic interface - After the purge, place the purge-and-trap system in the desorb mode and preheat the trap to 245°C without a flow of desorption gas. Start the flow of desorption gas at 10 mL/minute for about four minutes (1.5 min is normally adequate for analytes in Method 8015). Begin the temperature program of the gas chromatograph and start data acquisition.

11.2.4.2 Cryogenic interface - After the purge, place the purge-and-trap system in the desorb mode, make sure that the cryogenic interface is at -150°C or lower, and rapidly heat the trap to 245°C while backflushing with an inert gas at 4 mL/minute for about 5 minutes (1.5 min is normally adequate for analytes in Method 8015). At the end of the 5-minute desorption cycle, rapidly heat the cryogenic trap to 250°C. Begin the temperature program of the gas chromatograph and start the data acquisition.

11.2.5 Trap reconditioning

After desorbing the sample, recondition the trap by returning the purge-and-trap system to the purge mode. Maintain the trap temperature at 245°C (or other temperature recommended by the manufacturer of the trap packing materials). After approximately 10 minutes, turn off the trap heater and halt the purge flow through the trap. When the trap is cool, the next sample can be analyzed.

11.2.6 Data interpretation

Perform qualitative and quantitative analysis following the guidance given in the determinative method and Method 8000. If the concentration of any target analyte exceeds the calibration range of the instrument, it will be necessary to reanalyze the sample by the high concentration method. Such reanalyses need only address those analytes for which the concentration exceeded the calibration range of the low concentration method. Alternatively, if a sample aliquot of 1-2 g was also collected (see Sec. 8.2.1.7), it may be practical to analyze that aliquot for the analytes that exceeded the instrument calibration range in the 5-g analysis. If results are to be corrected for moisture content, proceed to Sec. 11.5.

11.3 High concentration method for soil samples with concentrations generally greater than 200 µg/kg.

The high concentration method for soil is based on a solvent extraction. A solid sample is either extracted or diluted, depending on sample solubility in a water-miscible solvent. An aliquot of the extract is added to organic-free reagent water containing, if applicable, internal and matrix spiking standards, purged according to Method 5030, and analyzed by an appropriate determinative method. Wastes that are insoluble in methanol (i.e., petroleum and coke wastes) are diluted with hexadecane (see Sec. 11.3.8).

NOTE: Surrogate compounds may either be spiked into the solvent at the time of extraction or the reagent water containing an aliquot of the extract prior to analysis. Since the surrogate recovery data from these two options provides assurances of either extraction or analytical efficiencies, the decision as to when the surrogates are added depends on what questions need to be answered for a given sample matrix and the intended uses of the data.

The specific sample preparation steps depend on whether or not the sample was preserved in the field. Samples that were not preserved in the field are prepared using the steps below,

beginning at Sec. 11.3.1. If solvent preservation was employed in the field, then the preparation begins with Sec. 11.3.4.

11.3.1 When the high concentration sample is not preserved in the field, the sample consists of the entire contents of the sample container. Do not discard any supernatant liquids. Whenever practical, mix the contents of the sample container by shaking or other mechanical means without opening the vial. When shaking is not practical, quickly mix the contents of the vial with a narrow metal spatula and immediately reseal the vial.

11.3.2 If the sample is from an unknown source, perform a solubility test preferably using a sample container reserved for the % moisture determination before proceeding. Remove several grams of material from the sample container. If the sample material is obtained from a vial dedicated for analysis, quickly reseal the container to minimize the loss of volatiles. Weigh 1-g aliquots of the sample into several test tubes or other suitable containers. Add 10 mL of methanol to the first tube, 10 mL of PEG to the second, and 10 mL of hexadecane to the third. Swirl the sample and determine if it is soluble in the solvent. Once the solubility has been evaluated, discard these test solutions. If the sample is soluble in either methanol or PEG, proceed with Sec. 11.3.3. If the sample is only soluble in hexadecane, proceed with Sec. 11.3.8.

11.3.3 For soil and solid waste samples that are soluble in methanol, add 9.0 mL of methanol and 1.0 mL of the surrogate spiking solution, or 10.0 mL of methanol without surrogates to a tared 20-mL vial. Using a top-loading balance, weigh 5 g (wet weight) of sample into the vial. Quickly cap the vial and reweigh the vial. Record the weight to 0.1 g. See Appendix A, Sec. 6.2.1 for methanol contact time information. If the sample was not soluble in methanol, but was soluble in PEG, employ the same procedure described above, but use 9.0 or 10.0 mL of PEG in place of the methanol. Proceed with Sec. 11.3.5.

NOTE: The steps in Secs. 11.3.1, 11.3.2, and 11.3.3 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.

11.3.4 For soil and solid waste samples that were collected in methanol or PEG (see Sec. 8.2.2), weigh the vial to 0.1 g as a check on the weight recorded in the field. If desired, add the surrogate spiking solution to the vial by injecting it through the septum, and proceed with Sec. 11.3.5. See Appendix A, Sec. A.6.2.1 for methanol contact time information.

11.3.5 Pipet approximately 1 mL of the extract from either Sec. 11.3.3 or 11.3.4 into a GC vial for storage, using a disposable pipet, and seal the vial. The remainder of the extract may be discarded. Add approximately 1 mL of methanol or PEG to a separate GC vial for use as the method blank for each set of samples extracted with the same solvent.

11.3.6 The extracts must be stored at 4°C in the dark, prior to analysis. Add an appropriate aliquot of the extract (based on the approximate sample concentration as noted in the table below) to 5.0 mL of organic-free reagent water containing if applicable, surrogates, internal standards, and matrix spike compounds, and analyze by Method 5030 in conjunction with the appropriate determinative method. Proceed to the Procedure Section in Method 5030 and follow the procedure for purging high concentration samples.

QUANTITY OF METHANOL EXTRACT REQUIRED FOR ANALYSIS OF HIGH CONCENTRATION SOILS/SEDIMENTS

| Approximate Concentration Range | Volume of Methanol Extract[a] |
|---|---|
| 500 - 10,000 µg/kg | 100 µL |
| 1,000 - 20,000 µg/kg | 50 µL |
| 5,000 - 100,000 µg/kg | 10 µL |
| 25,000 - 500,000 µg/kg | 100 µL of 1/50 dilution[b] |

Calculate appropriate dilution factor for concentrations exceeding those in this table.

[a] The volume of methanol added to 5 mL of water being purged should be kept constant. Therefore, add to the 5-mL syringe whatever volume of methanol is necessary to maintain a total volume of 100 µL of methanol.

[b] Dilute an aliquot of the methanol extract and then take 100 µL for analysis.

11.3.7 If results are to be reported using a correction factor for moisture content, determine the moisture content of a separate aliquot of the sample, using the procedure in Sec. 11.5, after the sample extract has been transferred to a GC vial and the vial sealed.

11.3.8 For solids that are not soluble in methanol or PEG (including those samples consisting primarily of petroleum or coking waste) dilute or extract the sample with hexadecane using the procedures in the Procedure Section of Method 3585.

11.4 High concentration method for oily waste samples

This procedure for the analysis of oily waste samples involves the dilution of the sample in methanol or PEG. However, care must be taken to avoid introducing any of the floating oil layer into the instrument. A portion of the diluted sample is then added to 5.0 mL of organic-free reagent water, purged according to Method 5030, and analyzed using an appropriate determinative method.

NOTE: Surrogate compounds may either be spiked into the solvent at the time of extraction or the reagent water containing an aliquot of the extract prior to analysis. Since the surrogate recovery data from these two options provides assurances of either extraction or analytical efficiencies, the decision as to when the surrogates are added depends on what questions need to be answered for a given sample matrix and the intended uses of the data.

For oily samples that are not soluble in methanol or PEG (including those samples consisting primarily of petroleum or coking waste), dilute or extract with hexadecane using the procedures in the Procedure Section of Method 3585.

The specific sample preparation steps depend on whether or not the sample was preserved in the field. Samples that were not preserved in the field are prepared using the steps below, beginning at Sec. 11.4.1. If methanol preservation was employed in the field, then the preparation begins with Sec. 11.4.3.

11.4.1 If the waste was not preserved in the field and it is soluble in methanol or PEG, weigh 1 g (wet weight) of the sample into a tared 10-mL volumetric flask, a tared scintillation vial, or a tared culture tube. If a vial or tube is used instead of a volumetric flask, it must be calibrated prior to use. This operation must be performed prior to opening the sample vial and weighing out the aliquot for analysis.

11.4.1.1 To calibrate the vessel, pipet 10.0 mL of methanol or PEG into the vial or tube and mark the bottom of the meniscus.

11.4.1.2 Discard this solvent, and proceed with weighing out the 1-g sample aliquot.

11.4.2 Quickly add 1.0 mL of surrogate spiking solution, if desired, to the flask, vial, or tube, and dilute to 10.0 mL with the appropriate solvent (methanol or PEG). Swirl the vial to mix the contents. See Appendix A, Sec. 6.2.1 for methanol contact time information.

11.4.3 If the sample was collected in the field in a vial containing methanol or PEG, weigh the vial to 0.1 g as a check on the weight recorded in the field. If desired, add the surrogate spiking solution to the vial by injecting it through the septum. Swirl the vial to mix the contents and proceed with Sec. 11.4.4. See Appendix A, Sec. 6.2.1 for methanol contact time information.

11.4.4 Regardless of how the sample was collected, the target analytes are extracted into the solvent along with the majority of the oily waste (i.e., some of the oil may still be floating on the surface). If oil is floating on the surface, transfer 1 to 2 mL of the extract to a clean GC vial using a Pasteur pipet. Ensure that no oil is transferred to the vial.

11.4.5 Add 10 - 50 µL of the methanol extract to 5 mL of organic-free reagent water containing if applicable, surrogates and internal standards, followed by purge-and-trap analysis, using Method 5030.

11.4.6 If necessary, prepare a matrix spike sample by adding 10 - 50 µL of the matrix spike standard dissolved in methanol to a 1-g aliquot of the oily waste. Shake the vial to disperse the matrix spike solution throughout the oil. Then add 10 mL of extraction solvent and proceed with the extraction and analysis, as described in Secs. 11.4.2 - 11.4.5. Calculate the recovery of the spiked analytes as described in Method 8000. If the recovery is not within the acceptance limits for the application, use the hexadecane dilution technique in the Procedure Section of Method 3585.

11.5 Determination of % moisture

If results are to be reported using a correction factor for moisture content, it is necessary to determine the moisture content of the sample. Also note that solid samples with a significant moisture content (>10%) that are extracted prior to analysis in a water miscible solvent such as methanol are diluted by the total volume of the solvent/water mixture. (Ref. 51) In order to report this type of sample result on an "as received" basis, the detected concentration needs to be corrected by the solvent/water dilution factor. See Method 8000 for an explanation and the applicable calculations.

NOTE: It is highly recommended that the moisture content determination only be made after the analyst has determined that no sample aliquots will be taken from the 60-mL vial for high concentration analysis. This is to minimize loss of volatiles and to avoid sample contamination from the laboratory atmosphere. There is no holding time associated with the moisture content determination. Thus, this determination can be made any time prior to reporting the sample results, as long as the vial containing the additional sample has remained sealed and properly stored.

11.5.1 Weigh 5-10 g of the sample from the 60-mL VOA vial into a tared crucible.

11.5.2 Dry this aliquot overnight at 105°C. Allow to cool in a desiccator before weighing. Calculate the % moisture as follows:

$$\% \text{ moisture} = \frac{\text{g of sample} - \text{g of dry sample}}{\text{g of sample}} \times 100$$

WARNING: The drying oven should be contained in a hood or vented. Significant laboratory contamination may result from a heavily contaminated hazardous waste sample.

## 12.0 DATA ANALYSIS AND CALCULATIONS

There are no calculations explicitly associated with this extraction procedure. See the appropriate determinative method and Method 8000 for calculation of final sample results.

## 13.0 METHOD PERFORMANCE

13.1 Single laboratory accuracy and precision data were obtained for the method analytes in three soil matrices, sand, a soil collected 10 feet below the surface of a hazardous landfill, called the C-Horizon, and a surface garden soil. Each sample was fortified with the analytes at a concentration of 20 ng/5 g, which is equivalent to 4 µg/kg. These data are listed in tables found in Method 8260.

13.2 Single laboratory accuracy and precision data were obtained for certain method analytes when extracting oily liquid using methanol as the extraction solvent. The data are presented in a table in Method 8260. The compounds were spiked into three portions of an oily liquid (taken from a waste site) following the procedure for matrix spiking described in Sec. 7.4. This represents a worst case set of data based on recovery data from many sources of oily liquid.

## 14.0 POLLUTION PREVENTION

14.1 Pollution prevention encompasses any technique that reduces or eliminates the quantity and/or toxicity of waste at the point of generation. Numerous opportunities for pollution prevention exist in laboratory operation. The EPA has established a preferred hierarchy of environmental management techniques that places pollution prevention as the management option of first choice. Whenever feasible, laboratory personnel should use pollution prevention techniques to address their waste generation. When wastes cannot be feasibly reduced at the source, the Agency recommends recycling as the next best option.

14.2 For information about pollution prevention that may be applicable to laboratories and research institutions consult *Less is Better: Laboratory Chemical Management for Waste*

*Reduction* available from the American Chemical Society's Department of Government Relations and Science Policy, 1155 16th St., N.W. Washington, D.C. 20036.

## 15.0 WASTE MANAGEMENT

The Environmental Protection Agency requires that laboratory waste management practices be conducted consistent with all applicable rules and regulations. The Agency urges laboratories to protect the air, water, and land by minimizing and controlling all releases from hoods and bench operations, complying with the letter and spirit of any sewer discharge permits and regulations, and by complying with all solid and hazardous waste regulations, particularly the hazardous waste identification rules and land disposal restrictions. For further information on waste management, consult *The Waste Management Manual for Laboratory Personnel* available from the American Chemical Society at the address listed in Sec. 12.2.

## 16.0 REFERENCES


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# APPENDIX A

# THE COLLECTION AND PRESERVATION OF AQUEOUS AND SOLID SAMPLES FOR VOLATILE ORGANIC COMPOUND (VOC) ANALYSIS

# FOREWORD

The information provided in this Appendix is based on EPA's evaluation of currently available data and technology as applied to the most appropriate sample handling and preservation procedures in order to minimize the loss of volatile organic compounds (VOCs) during the collection and analysis of aqueous and solid materials, such as groundwater, wastewater, soils, solid waste, or sediments. These procedures are designed to minimize the losses of VOCs through the two most common mechanisms, volatilization and biodegradation. The intended users of this Appendix guidance are those individuals and organizations involved in the collection and preparation of samples for VOC analyses during the characterization of solid materials under the Resource Conservation and Recovery Act (RCRA). The procedures and techniques described in this Appendix are not presented in any preferential order nor do they represent EPA requirements, but rather they are intended solely as guidance and should be selected and utilized based on the stated project-specific data quality objectives.

This Method 5035 Appendix was developed under the direction of Mr. Barry Lesnik, U.S. EPA, Office of Solid Waste (OSW), Methods Team in collaboration with Mr. David Payne, U.S. EPA, Region 5, Mr. Alan Hewitt, U.S. ACE CRREL, and the SW-846 Organic Methods Workgroup Members. The Methods Team is the focal point within OSW for expertise in analytical chemistry and characteristic testing methodologies, environmental sampling and monitoring, and quality assurance. The Methods Team provides technical support to other OSW Divisions, EPA Program Offices and Regions, state regulatory agencies, and the regulated community.

# DISCLAIMER

The U.S. Environmental Protection Agency's Office of Solid Waste (EPA or the Agency) has prepared this Method 5035 Appendix to provide guidance to those individuals involved in the collection and preparation of samples for volatile organic compounds (VOCs) analysis during the characterization of aqueous and solid materials under the Resource Conservation and Recovery Act (RCRA). This Appendix provides guidance for selecting an appropriate sample collection and preservation technique that may be suitable for VOC analyses in order to meet the data quality requirements or objectives for the intended use of the results.

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## Tables

## A.1.0 PURPOSE AND OVERVIEW

This Appendix provides guidance in sample collection and preservation procedures that may be suitable for use during the characterization of volatile organic compounds (VOCs) in solid materials, such as soils, solid wastes, or sediments and aqueous samples or leachates from solid matrices.

### A.1.1 What are VOCs?

VOCs are a class of organic compounds that includes low molecular weight aromatics, hydrocarbons, halogenated hydrocarbons, ketones, acetates, nitriles, acrylates, ethers, and sulfides with sufficiently low boiling points to give them appreciable vapor pressures at 1 atmosphere of pressure. Although EPA has never defined a strict boiling point cut-off for this compound class, most VOCs of concern to EPA have boiling points below 150 °C, while some members of this class may have boiling points as high as 200 °C.

The solubilities of the individual VOCs in water vary widely, from insoluble to soluble, with many of the oxygenated compounds (ketones and ethers) at the soluble end of the range and the hydrocarbons and substituted hydrocarbons at the insoluble end of the range.

Given that water may be present to varying degrees in such solid materials of environmental significance as soils, solid wastes, and sediments, the water solubility of an individual VOC may in fact control its "solubility" in solid samples.

### A.1.2 What is sample preservation?

The sample collection procedures described in EPA analytical methods are designed to ensure that at the time of analysis, the chemical composition of the small volume of material collected from the parent bulk material is representative of the chemical composition of the original material. Considerations regarding sample support and sampling design (discussed in Chapter Nine of the SW-846 manual) ensure that the physical aspects of sample collection (e.g., sample volume and orientation, numbers and distribution of samples) produce data estimates that are representative of the bulk material subject to regulatory decision-making, perhaps millions of gallons a day of discharged wastewater, or thousands of kilograms of solid material. Once collected, a sample should be maintained in a manner that preserves the relationship between it and the bulk material, e.g., the chemical composition of the sample should not change by virtue of being collected. Maintaining that relationship between the sample and the bulk material is referred to as sample preservation.

Several types of sample preservation are employed in EPA methods. The most common method of preservation is to cool the sample to 4 ± 2 °C. Cooling may be applied to many types of sample matrices, including water, soil, sediments, and solid wastes. The temperature of 4 ± 2 °C is used because it represents the temperature at which pure water exhibits its maximum density, hence its minimum volume. However, if aqueous samples are cooled below 0 °C, the water expands significantly as it freezes and may crack the sample container.

By lowering the temperature of the sample, many of the physical, chemical, and biological processes that may cause environmental contaminants to leave the sample (e.g., loss of volatiles to the air) or be transformed into other compounds (e.g., chemical breakdown or biodegradation) are greatly slowed. However, even if the rates of biodegradation are reduced by physical preservation, many environmental matrices of interest contain large numbers of microorganisms that may break down contaminants. Examples include wastewaters from sewage

treatment, surface waters, and surface soil. In these types of matrices, simply reducing the rate at which biodegradation occurs may not be enough to maintain the condition of the original sample.

The most practical way in which to reduce this biological activity in aqueous samples is through the use of chemical preservatives that act as biocides. Historically, this has included preservatives such as sodium bisulfate or hydrochloric acid to adjust the pH for aqueous samples to less than pH 2, at which point, virtually all biological activity ceases.

Adjusting the pH of a solid sample such as a soil, sediment, or solid waste presents a number of other difficulties. In particular, samples containing carbonates should not be acidified due to the potential for effervescence which may result in loss of volatile compounds. Precautions should also be taken when preserving by acidification since certain compounds within the olefins, ketones, esters, ethers, and sulfides classes may react under low pH conditions and possibly not be representative of the material as sampled. Additionally, acidification of solid wastes may evolve toxic gases that may be harmful to field and laboratory personnel. **It is therefore recommended that when collecting wastes of unknown composition, preliminary screening and characterization of potential sample contents should be performed prior to use of acidification as a means to chemically preserve samples designated for determinative analyses.**

Sample collection and preservation procedures should be carefully selected in order to minimize VOC losses prior to sample preparation and determination in the laboratory. Although this guidance discusses some traditional approaches to VOC sample collection and preservation, its main purpose is to provide guidance regarding newer approaches, such as freezing the samples, which may particularly decrease VOC loss in some materials. For additional information regarding the challenges associated with collecting and handling VOC samples, recommended reading includes the "Standard Guide for Sampling Waste and Solids for Volatile Organic Compounds" (ASTM D 4547-98), published by the American Society for Testing and Materials (ASTM). (Ref. 15)

Currently, **it is recommended that VOC solid samples are to be collected, while maintaining a closed-system approach to prevent constituent losses, using an appropriate coring device and immediately transferred to the VOA vial to be used for analysis and should be stored for no longer than 48 hours at 4 ± 2°C prior to analysis or preservation.** Longer storage times at 4 ± 2°C may be appropriate if it can be demonstrated that the VOC concentrations are not adversely affected or that the data generated at the time of sample analysis meets the project-specific data quality objectives. Extended sample storage, up to 14 days from sample collection, may be obtained by either physical or chemical preservation techniques as noted in this Appendix guidance. These preservation techniques can be initiated at the time of sample collection or after arrival in a laboratory. Refer to Table A.1 for a summary of the recommended preservation techniques and analytical holding times.

A.1.3 Do all VOA samples need to be chemically preserved?

No. Only samples that contain analytes that are subject to biological degradation prior to analysis need to be preserved. Samples where aromatic hydrocarbons are target analytes, which are most subject to biological degradation, need to be preserved, unless they are to be analyzed immediately on-site, even if other VOA compound classes are present. Preservation may be inappropriate for highly reactive compounds, e.g., styrene, vinyl chloride, since it may accelerate loss by polymerization or other rapid chemical reaction. Samples for which chlorinated aliphatic hydrocarbons are the only target analytes generally do not need to be preserved. However, all aqueous samples containing free chlorine must be preserved with a dechlorinating agent in order to prevent formation of trihalomethanes and other possible chemical reactions.

A.1.4 Who is the intended audience for this Appendix?

VOCs are frequently Resource Conservation and Recovery Act (RCRA) Program analytes of concern, and thus waste management decisions are often based on characterization of the VOC levels. The intended users of this Appendix guidance are those individuals involved in any way in the collection and preparation of samples for VOC analysis during the characterization of solid materials under RCRA. This may include:

- field sampling personnel
- laboratory analysts
- environmental project managers, whether at a facility regulated under RCRA, or working for a regulatory agency
- Federal, state, and local regulators with oversight responsibilities for sample collection activities
- quality assurance personnel
- data quality assessors.

A.1.5 What does this guidance *not* cover?

This Appendix does *not* provide detailed guidance regarding sampling design or the actual steps in sample preparation and VOC determination in the laboratory. For such guidance, users of this manual should refer to Chapter Nine of SW-846 and the preparation and determinative methods that are selected for analysis as part of the planning process in order to meet the intended data quality objectives.

A.1.6 What equipment is needed?

The site-specific Sampling and Analysis Plan should clearly list the required sample collection equipment necessary to ensure that the loss of volatile constituents will be minimized during the sample collection process. As with all environmental sampling applications, the analytical data usability and representativeness will be affected by improper sample collection techniques. Sampling personnel will be responsible for ensuring that VOA vials are sealed properly using a septum of sufficient thickness without any punctures. The improper vial sealing (i.e., due to excess sample retained on the vial threads) and tightening of caps are the primary factors in the loss of volatiles due to sample collection activities. Care should also be exercised in the selection of approved pre-cleaned and certified VOA vials absent of burrs on the glass. Procedures should be in place for the selection and appropriate use of sample collection devices (i.e., bailer, coring tool, etc.) along with the required decontamination measures. It is also recommended to store one trip blank per cooler when collecting volatile samples in order to assess possible field induced contamination.

A.1.7 How is the guidance organized?

This Appendix is organized as follows:

**Section A.2.0 - Project Planning** -- Provides an overview of the data quality objectives (DQOs) process as related to the suggested project planning activities prior to sample collection.

**Section A.3.0 - Aqueous Sample Matrices and Volatile Organic Compounds** – Outlines the appropriate sampling and preservation strategy for aqueous sample matrices.

**Section A.4.0 - Solid Materials/Cohesive Soils and Volatile Organic Compounds** -- Describes the two most common mechanisms (volatilization and biodegradation) for potential VOC losses during the sample collection process.

**Section A.5.0 - History of Practices in the Sampling and Preparation of Solid Materials for VOC Analysis** – Provides a summary of the common historical VOC loss mechanisms and discusses the improvements and new developments in sample collection techniques.

**Section A.6.0 - Overview of Vapor Partitioning and Methanol Extraction Technologies** – Discusses the two most commonly used methods for the laboratory preparation of soils for VOC analysis.

**Section A.7.0 - Sample Collection** – Describes the sample collection and storage process for various solid matrices.

**Section A.8.0 - Approaches to Sample Preparation** -- Provides examples of several sample preparation techniques that may be appropriate based on the intended use of the data.

**Section A.9.0 - Summary of Findings** – Lists the key highlights as discussed in Sections A.2.0 through A.8.0.

**Section A.10.0 - References**

## A.2.0 PROJECT PLANNING

The EPA requires that a systematic planning process such as, but not limited to, the Data Quality Objectives (DQOs) Process be used for all EPA environmental data collection activities. Systematic Planning is necessary to define the type, quantity, and quality of data a decision maker needs before collecting or generating environmental data. As part of the DQO process, questions such as "what are the possible sample matrices?," "why is the sample being collected?," and "what are the appropriate analytical methods?" can be answered based on the intended use of the data. The Systematic Planning process should also include the preparation of a Quality Assurance Project Plan (QAPP) along with a site-specific Sampling and Analysis Plan (SAP) prior to any sample collection activities. Refer to *Guidance for the Data Quality Objectives Process* (G-4) (August 2000, EPA/600/R-96/055), *Guidance for Quality Assurance Project Plans* (G-5) (February 1998, EPA/600/R-98/018) and Chapter Nine of SW-846 for guidance on how to perform the DQO process and planning guidance associated with RCRA waste sampling and analysis.

During the project planning period it is important to stress to all interested parties that any samples identified as a result of the planning process must be representative of the material subject to investigation, and that each sample handling activity can affect sample integrity and representativeness up through analysis (e.g., VOCs can be lost if samples are not appropriately collected and preserved [See Sec. A.1.3]).

The EPA encourages the use of a performance-based measurement system (PBMS) during selection of sample collection and preparation approaches. The EPA defines PBMS as "a set of processes wherein the data quality needs, mandates or limitations of a program or project are specified, and serve as criteria for selecting appropriate methods to meet those needs in a cost effective manner." The PBMS process permits the use of any appropriate method that demonstrates the ability to meet established criteria while complying with specified data quality needs. In addition, analysts must generate initial and continuing method performance data that demonstrate that the selected approaches were appropriate. Implementation of PBMS does not negate the need for or use of standard or consensus methods. It only eliminates the mandate that they be used exclusively. The following are typical items that should be considered during selection of approaches to VOC sample collection and preservation:

1. VOC concentration range.

2. VOC constituents of interest.

3. Physical characteristics of material, i.e., water content and particle size distribution.

4. Chemical and biological characteristics of material, i.e., acid/base properties, chlorine residual, carbonate content, and microbial activity.

5. Compatibility with selected preparation method.

6. Holding time constraints.

7. Data quality requirements.

## A.3.0 AQUEOUS SAMPLE MATRICES AND VOLATILE ORGANIC COMPOUNDS

All environmental aqueous samples are physically preserved at 4 ± 2°C immediately after collection in order to improve the overall VOC stability prior to analysis. This preservation process alone has been shown to be effective in preventing the degradation of most constituents for up to seven days from the sample collection date. Depending on the project required VOC constituents, an aqueous sample stability or holding time period can be extended to fourteen days with the use of chemical preservatives such as sodium bisulfate or hydrochloric acid. The chemical preservatives act as acidifying agents to lower the sample pH and thereby inhibit microbial activity which may cause biological degradation of aromatic hydrocarbons. However, since reactive compounds such as 2-chloroethyl vinyl ether are unstable at low pHs, if these analytes are to be determined, the collection of a second set of samples without acid preservatives is necessary. In addition, aqueous samples containing methyl *tert*-butyl ether and other fuel oxygenate ethers should not be acidified if high temperature sample preparative methods (Methods 5021, 5030, 5032) are used. (Refs 48,49) (NOTE: if the aromatic constituents such as benzene, toluene, ethylbenzene, and xylenes (BTEX) are among the analytes of interest, acidification is required for biologically active samples because it has been demonstrated that losses can occur within four hours of sample collection).

The presence of free chlorine in aqueous samples must be monitored and controlled in order to prevent the possible formation of trihalomethanes and reaction with certain compounds such as styrene after sample collection. Therefore, samples containing residual chlorine should be treated with a 10% sodium thiosulfate solution or ascorbic acid prior to acidification in order to reduce the chlorine to unreactive chloride.

Details of procedures and protocols for sample collection must be identified in an approved sampling plan. Aqueous samples for volatile constituents should be collected in vials or containers specifically designed to prevent loss of analytes. In most cases, containers should be provided by the laboratory conducting the analysis. If chemical preservation is required and the laboratory has not pre-preserved the containers, add the appropriate preservative prior to sample collection. Store empty VOC containers on ice in order to reduce potential volatilization while they are being filled. During the sample collection process do not rinse the container before filling and take care to minimize sample overflow that may dilute the preservative. The container should be filled until the water sample forms a positive meniscus at the brim. At this point the container is capped immediately to prevent bubbles and headspace. After the sample has been collected and the container capped, the formation of bubbles can be verified by inverting and lightly tapping the side of the container. Sometimes it is not possible to collect a sample without air bubbles, particularly if the water is aerated. In these cases, the field personnel should record the problem and account for the probable cause. (NOTE: dechlorinating agents should not be mixed with the acid preservative prior to sample collection).

During transport and prior to analysis, samples should be stored in a cooler or refrigerator maintained at 4 ± 2°C and care should be taken to prevent freezing of the sample and possible container breakage. The sampling plan should indicate how sample shipment will occur along with method of packaging, shipping, and the time schedule relative to sample collection and analytical holding times. Refer to Table A.1 for a summary of the recommended preservation techniques and analytical holding times.

A large number of water VOC sample holding time and stability studies have been performed to determine the degree of degradation which may occur at a variety of concentrations, preservation, and storage conditions. Data from these studies have been reviewed by the Oak Ridge National Laboratory (ORNL) in order to develop an approach for assessing the data

confidence from analyses completed beyond the regulatory holding time of 14 days. This approach is based on methodology, referred to as "Practical Reporting Times," that were developed by ORNL in the early 1990's, and described in a summary report listed in Ref. 47. Users may find the data provided in Tables 2 and 3 of this referenced report to be helpful in estimating the post-holding time degradation of VOCs in water and for determining the potential data impact from analyses completed beyond the required holding time. However, the user is cautioned that the holding times provided in this report are estimations based on actual analytical data, and the true values are relative to the on-site sample matrix conditions. See the footnote following Table A.1 regarding holding time extensions.

### A.3.1 Alternative Considerations for Sample Holding Time Criteria

Recognition that holding times for environmental contaminants are analyte-specific and highly variable is not new. (Refs. 52,53,54). Environmental contaminants may be short-lived, destroyed by preservation, or highly resistant to degradation. Understanding and applying historical knowledge (Table A.1) can be important and valuable. (Ref. 55) Therefore, we encourage consideration of alternative holding times for several reasons:

1. Project planning,

2. Performance based data review processes,

3. Analytical method selection,

4. Streamlined verification of unexpected or suspect analytical results, and

5. Design of alternative quality control procedures.

Specific examples of how to implement the information incorporated in Table A.1 include the following: During project/systematic planning, field measurement or quick-turn-around analyses must be identified as critical if particular contaminants of concern for a project are easily lost or destroyed. Currently, data review guidelines suggest samples analyzed within 2 weeks of collection be accepted as uniformly reliable, and analyses completed >2 weeks after sample collection are uniformly assessed as unacceptably uncertain. This review judgement is not technically defensible. Many of the most common contaminant decision drivers listed in Table A.1 are important, because they are stable over time, e.g., chlorinated solvents. For these contaminants, cooperative Inter-Agency research has demonstrated no significant change in results from analyses performed at 30 days, often as long as 96 days, after collection and preservation. *NOTE: this extension assumes preservation of samples as identified in Table A.1.* In addition, longer holding times than those specified in Table A.1 may be appropriate if it can be demonstrated that the reported VOC concentrations are not adversely affected from preservation, storage and analyses performed outside the recommended holding times.

The resistance to degradation of these frequent environmental drivers offers additional process improvement opportunities. Utilization of a second VOA sample analyzed beyond the recommended holding time is a mechanism to verify or independently determine unexpected results or correct laboratory errors that cannot be addressed within the current 2-week window. With no significant loss of confidence in the results, this approach eliminates the schedule delays and expense of sampling crew mobilization.

In addition, the use of site-specific performance evaluation material is recognized as a high confidence mechanism to ensure reliability of project data. However, the historical perception of short shelf-life for volatile organics in water eliminates implementation of this approach as a viable

quality control/quality assurance system component for water monitoring programs. Table1 and the associated references contain documentation of appropriate analytes and procedures to develop and implement these alternatives.

**Table A.1**
**Recommended VOC Sample Preservation Techniques and Holding Times**

| Sample Matrix | Preservative | Holding Time | Comment |
|---|---|---|---|
| Aqueous Samples With No Residual Chlorine Present | Cool to 4 ± 2°C. | 7 days | If MTBE and other fuel oxygenate ethers are present and a high temperature sample preparative method is to be used, do not acid preserve the samples. If aromatic and biologically active compounds are analytes of interest, acid preservation is necessary and the holding time is extended to 14 days. |
| Aqueous Samples With No Residual Chlorine Present | Cool to 4 ± 2°C and adjust pH to less than 2 with HCl or solid $NaHSO_4$. | 14 days[1] | Reactive compounds such as 2-chloroethylvinyl ether readily break down under acidic conditions. If these types of compounds are analytes of interest, collect a second set of samples without acid preservatives and analyze as soon as possible. |
| Aqueous Samples With Residual Chlorine Present | Collect sample in a pre-preserved container containing either 25 mg ascorbic acid or 3 mg of sodium thiosulfate per 40-mL of chlorinated sample volume containing less than 5 mg/L of residual chlorine. Cool to 4 ± 2°C. | 7 days | Samples containing greater than 5 mg/L of residual chlorine may require additional amounts of dechlorinating agents. If MTBE and other fuel oxygenate ethers are present and a high temperature sample preparative method is to be used, do not acid preserve the samples. If aromatic and biologically active compounds are analytes of interest, acid preservation is necessary and the holding time is extended to 14 days. |
| Aqueous Samples With Residual Chlorine Present | Collect sample in a pre-preserved container containing either 25 mg ascorbic acid or 3 mg of sodium thiosulfate per 40-mL of chlorinated sample volume containing less than 5 mg/L of residual chlorine. Cool to 4 ± 2°C and adjust pH to less than 2 with HCl or solid $NaHSO_4$ | 14 days[1] | Samples containing greater than 5 mg/L of residual chlorine may require additional amounts of dechlorinating agents. Reactive compounds such as 2-chloroethylvinyl ether readily break down under acidic conditions. If these types of compounds are analytes of interest, collect a second set of samples without acid preservatives and analyze as soon as possible. Caution: never add acid preservative directly to a dechlorinating agent prior to sample collection. |
| Solid Samples[2] | Sample is extruded into an empty sealed vial and frozen on-site to < -7°C. | 14 days[1] | Sample vials should not be frozen below -20°C due to potential problems with vial seals and the loss of constituents upon sample thawing. |

**Table A.1** (Continued)

| Sample Matrix | Preservative | Holding Time[1] | Comment |
|---|---|---|---|
| Solid Samples[2] | Sample is extruded into an empty sealed vial and cooled to 4 ± 2°C for no more than 48 hours then frozen to < -7°C upon laboratory receipt. | 14 days[1] | Analysis must be completed within 48 hours if samples are not frozen prior to the expiration of the 48 hour period. Sample vials should not be frozen below -20°C due to potential problems with vial seals and the loss of constituents upon sample thawing. |
| | Sample is extruded into an empty sealed vial and cooled to 4 ± 2°C for no more than 48 hours then preserved with methanol upon laboratory receipt. | 14 days[1] | Analysis must be completed within 48 hours if samples are not preserved with methanol prior to the expiration of the 48 hour period. |
| | Sample is extruded into an empty sealed vial and cooled to 4 ± 2°C. | 48 hours | |
| | Cool to 4 ± 2°C the coring tool used as a transport device | 48 hours | The holding time may be extended to 14 days if the sample is extruded to a sealed vial and either frozen to < -7°C or chemically preserved. Coring tools should not be frozen below -20°C due to potential problems with tool seals and the loss of constituents upon sample thawing. |
| | Freeze to < -7°C the coring tool used as a transport device | 48 hours | The holding time may be extended to 14 days if the sample is extruded to a sealed vial and either frozen to < -7°C or chemically preserved. Coring tools should not be frozen below -20°C due to potential problems with tool seals and the loss of constituents upon sample thawing. |
| | Sample is extruded into a vial containing reagent water and frozen on-site to < -7°C. | 14 days[1] | Sample vials should not be frozen below -20°C due to potential problems with vial seals and the loss of constituents upon sample thawing. |
| | Sample is extruded into a vial containing reagent water and cooled to 4 ± 2°C for 48 hours or less then frozen to < -7°C upon laboratory receipt. | 14 days[1] | Analysis must be completed within 48 hours if samples are not frozen prior to the expiration of the 48 hour period. Sample vials should not be frozen below -20°C due to potential problems with vial seals and the loss of constituents upon sample thawing. |

**Table A.1** (Continued)

| Sample Matrix | Preservative | Holding Time[1] | Comment |
|---|---|---|---|
| Solid Samples[2] | Sample is extruded into a vial containing reagent water and 1 g $NaHSO_4$ and cooled to 4 ± 2°C. | 14 days[1] | Reactive compounds such as 2-chloroethylvinyl ether readily break down under acidic conditions. If these types of compounds are analytes of interest, collect a second set of samples without acid preservatives and analyze as soon as possible. |
| | Sample is extruded into a vial containing methanol and cooled to 4 ± 2°C. | 14 days[1] | Additional methanol extract storage time beyond 14 days may be acceptable if the desired VOC constituent stability can be demonstrated from appropriate performance data. |

[1] A longer holding time may be appropriate if it can be demonstrated that the reported VOC concentrations are not adversely affected from preservation, storage and analyses performed outside the recommended holding times.

[2] For biologically active soils, immediate chemical or freezing preservation is necessary due to the rapid loss of BTEX compounds within the first 48 hours of sample collection.

During the selection of VOC sample collection and preservation approaches, it is important to understand the mechanisms of VOC loss inherent to solid materials and VOCs. In general, uncontrolled losses occur through both volatilization and biodegradation. However, for some compounds, e.g., vinyl chloride, acrylonitrile, 2-chloroethylvinyl ether, and styrene, rapid losses can occur through chemical reaction, as well. (Ref. 46)

In most solid materials, VOCs coexist in gaseous and liquid phases, as well as sorbed to the solid particles. The molecular diffusion coefficients of VOCs in the gaseous phase are high enough to allow for the immediate volatilization of those VOCs from a freshly exposed sample surface, resulting in a loss to the surrounding atmosphere. If the sample matrix is porous, these losses will continue as VOCs below the surface diffuse outward. Furthermore, once the gaseous phase is lost, the dynamic equilibrium between the gaseous phase and the liquid and sorbed VOC phases will result in rapid transformations of the liquid and sorbed VOCs to the gaseous phase, where they can continue to be lost to the atmosphere. (Ref. 4) Thus, the primary goal of preservation is to minimize or eliminate the loss of the compounds of concern through direct volatilization to the atmosphere.

The biodegradation of VOCs usually involves compound loss by biological processes mediated by naturally-occurring micro- and macro-organisms found in solid environmental samples such as soils and sediments. Aerobic processes are often of greatest concern, but anaerobic organisms in some sediments and soils can also result in significant losses of VOCs. Biodegradation may be of concern in waste samples, particularly those that may have been stored outdoors.

Most soil sample collection procedures involve intrusive sampling operations that can create or enhance aerobic conditions within a sample. Aerobic conditions can occur by disaggregation of the particles in the solid, or by simple exposure of the sample to air (e.g., collection of a sediment sample from under standing water). Soil samples should be collected immediately or as soon as practical after exposure of the soil during such activities as tank removal or excavation in order to minimize VOC losses from uncontrolled aerobic processes. Unless precautions as noted in this Appendix are employed, aerobic conditions will then persist during handling and storage of the sample.

The rate of biodegradation is dependent on several factors, including the indigenous microbes, the chemical properties of the individual VOC, the total VOC concentration, the chemical properties of the solid matrix, and temperature. In general, the biodegradation mechanism for soil VOCs is not as large a source of determinate error as volatilization. Volatilization losses of an order of magnitude can occur in minutes to hours, whereas losses of a similar magnitude due to biodegradation usually take days to weeks.

Biodegradation is compound selective whereby, under aerobic conditions, the biological mechanisms favor the degradation of aromatic hydrocarbons over the loss of halogenated (chlorinated) hydrocarbons. (Refs. 1,2,4) Aromatic hydrocarbons such as benzene, toluene, ethyl benzene, and xylenes (collectively referred to as BTEX) can be lost in days from samples stored at $4 \pm 2^\circ$ C, while losses of chlorinated hydrocarbons by biodegradation over the same period can be relatively insignificant. Major benzene and toluene biodegradation losses (50% or more) have been observed when soils are stored at room temperature ($22^\circ$C) for five (5) days and near complete concentration reduction when stored for fourteen (14) days at $4^\circ$C. (Refs. 1,2,4,6,11,17) For extremely biologically active soils this can occur in less than five days. (Ref. 11)

Due to the above mechanisms, attempts are made from the beginning to maintain sample integrity and representativeness. In doing so, approaches often use various combinations of chemical (e.g., methanol) and physical (e.g., freezing) preservation procedures and collection (e.g., single transfer to air-tight vial) and storage practices (e.g., holding times) to minimize VOC loss. Some of these approaches are presented within this guidance.

## A.5.0 HISTORY OF PRACTICES IN THE SAMPLING AND PREPARATION OF SOLID MATERIALS FOR VOC ANALYSIS

### A.5.1 Traditional Practices

Over the past 20 years, solid samples obtained for VOC analysis were collected using a spatula -type device to completely fill a container for transfer off-site before the introduction of certain preparation steps and analysis within a 14-day holding time. VOC sampling procedures recommended the use of clean stainless steel utensils to completely fill either 40-mL to 250-mL glass containers. The containers were then closed with polytetrafluoroethylene (PTFE)-lined caps. Sample containers were stored in coolers at 4 ± 2° C and shipped to field or off-site support laboratories for subsampling (usually with 1 to 5 g aliquots) and subsequent analysis. The common holding time for these bulk soil samples, held at 4 ± 2°C, was 14 days.

During the 1990s, research efforts demonstrated that the above VOC bulk sampling procedure is inaccurate and produces VOC results that are biased low. (Refs. 3,8,10,16,30,31,32,33,34) The studies showed that bulk samples can lose 90% or more of their VOC content prior to analytical measurement. (Refs. 3,8,10,16,29,31,32,33) Reasons identified for these losses include:

1. Volatilization from exposure of the solid surface near the time of collection. (Refs. 3,8)

2. Volatilization from intermediate storage containers (e.g., core barrel liners, plastic bags, etc.). (Refs. 4,10,13,17,30)

3. Volatilization from disaggregation of the solid during collection. (Refs. 3,8)

4. Volatilization from failed seals on the PTFE-lined caps of the bottles or volatile organic analyte (VOA) vials (can be caused by soiling of cap and bottle ring closures during filling of containers). (Refs. 3,8)

5. Volatilization during laboratory subsampling of the bulk samples. (Refs. 3,8)

6. Biodegradation (principally of aromatic hydrocarbons, especially benzene and toluene) during storage (probably hastened by disaggregation of soils during sampling). (Refs. 3,8,11)

7. Reaction of chemically reactive compounds during sample storage.

8. Pressure changes during sample collection and transport.

### A.5.2 Improvement of Sample Collection Techniques

Due to concerns regarding the loss of VOCs, particularly in samples containing low concentrations of VOCs (<200 µg/kg) during traditional sampling practices, the scientific community investigated other approaches to VOC sample collection and preparation. A closed-system purge-and-trap technique was developed and tested for the analysis of low-level concentrations of VOCs in solids. The methanol extraction option for high concentrations (>200 µg/kg) and oily wastes remained unchanged. The Office of Solid Waste promulgated Method 5035 as part of Update III to the Third Edition of SW-846 on June 13, 1997. (Ref. 38) As an active participant in these studies in conjunction with OSW, the American Society for Testing and Materials (ASTM) published the

"Standard Guide for Sampling Waste and Soils for Volatile Organic Compounds" (ASTM D 4547-98). (Ref. 15)

These documents include the immediate in-field transfer of the sample (by a coring tool of 2 to 5 g capacity) into a tared VOA vial (of 22 to 40 mL capacity) that contains acidified reagent water (most often acidified by 1g $NaHSO_4$ per 5g of soil) so that a vapor partitioning preparation procedure (see Sec. A.6.0 of this Appendix) can be performed by the laboratory on the sample without reopening the vial. A second in-field transfer to a tared VOA vial containing 5 to 10 mLs of methanol is used for VOC soil concentrations larger than 200 µg/kg.

Another technique described is the immediate in-field collection and maximum 48 hour storage in an air-tight coring device/container (such as the EnCore™ sampler) so that the laboratory preservation and preparation procedures described for the closed-system purge-and-trap (Method 5035) or headspace (Method 5021) can be performed. (Ref. 39) (An EnCore™ sampler is a device that can be used for both sample collection and as the sample transport and storage device. See Sec. A.8.0)

Both documents recommend similar approaches to sample preservation and preparation in order to minimize VOC loss and address the collection of cohesive solids whereby a coring tool collects a relatively undisturbed sample by compression, and then extrudes the sample into an appropriate VOA vial. The documents also provide guidance for the collection of cemented materials and non-cohesive materials (e.g., dry sand, mixtures of gravel and fines) and collectively address factors that must be considered when selecting the most appropriate approach for VOC sample preservation and preparation, including expected concentrations of VOCs (high versus low). A screening method for determining whether a sample contains high or low concentrations of VOCs (Method 3815) is available for making these determinations on-site. (Ref. 40)

A.5.3 New Developments

Since the publication of the new VOA sampling techniques for solids, the scientific community has continued to investigate additional techniques to further improve sample collection and preservation to minimize VOC loss. For example, studies were conducted regarding the freezing of samples without the use of chemical preservatives (see Sec. A.8.0), use of "empty VOA vials," and more information was gained regarding acidification of samples, as discussed below. (Refs. 4,19)

Current practice recommends the use of $NaHSO_4$ to acidify reagent water in VOA vials prior to addition of the sample when preservation is necessary. (Ref.1) This acidification is one means used to minimize loss of VOC due to biodegradation. However, acidification is not recommended for solids or aqueous samples with significant levels of carbonates, because the acidification can cause effervescence and the loss of VOCs. In 1998 and 1999, other adverse effects of acid preservation of soils were discovered i.e., chemical breakdown of certain classes of compounds. Additionally, certain VOC components such as 2-chloroethylvinyl ether are lost by the acidification. An artifact is sometimes observed for acetone in that acidification of certain soils may cause the formation of acetone. (Refs. 4,37)

The approaches recommended in Sec. A.8.0 of this guidance incorporate the new developments in solid sample preparation for VOC analysis.

## A.6.0 OVERVIEW OF VAPOR PARTITIONING AND METHANOL EXTRACTION TECHNOLOGIES

Vapor partitioning and methanol extraction are the two most commonly used methods for the laboratory preparation of soils for VOC analysis. This section briefly discusses these two procedures, and their relative advantages and disadvantages. For further information, ASTM D 4547-98 (Ref. 15) discusses the merits of vapor partitioning relative to the use of methanol extraction; and Method 5035 relates concerns regarding the use of methanol.

Selection of the preparation technology should be made during the systematic planning process prior to sample collection given that the selection will dictate subsequent sample collection and preservation practices. One technology may be preferred based on the project data quality objectives and target analytes, and the sample collection and handling approaches need to be compatible with the chosen technology.

Each preparation technology involves use of a VOA vial for sample collection and transport. Approaches for preparation of the vials (with and without preservatives), often based on the technology to be used, will be discussed in a section to follow.

### A.6.1 Vapor Partitioning

One means of vapor partitioning involves the direct analysis of a sample by purge-and-trap (Method 5035). This technique is routinely used for the analysis of volatiles in environmental samples and is considered more sensitive than the headspace technique. By purging samples at higher temperatures, higher molecular weight compounds can be detected. However, the purge-and-trap technique requires more time for sample preparation.

Another means of vapor partitioning involves the direct analysis of a sample by equilibrium headspace (Method 5021). This technique is most suited for the analysis of very light molecular weight volatiles in samples that can be efficiently partitioned into the headspace gas volume from the liquid or solid matrix sample. Higher boiling point volatiles are not detected with this technique due to their low partition rate in the gas headspace volume. In addition, the technique is generally less sensitive than purge-and-trap, however, it is preferred for the analysis of gases, highly water-soluble compounds, and very light molecular weight volatiles which may not be analyzed using the purge-and-trap technique.

For both vapor partitioning techniques, the vapor is removed for analysis without opening the container. Heat and water are usually used to assist in the direct partitioning of VOCs from the solid matrix. Vapor partitioning is applicable to VOC soil concentrations of 2 to 200 ppb. Methods 5021 or 5035 commonly require 2- to 5-g soil aliquots collected in individual 20- to 40-mL VOA vials, depending on the specific instrumentation used in the selected purge-and-trap or headspace method. Only one analysis per VOA vial can be done using purge-and-trap or headspace (Methods 5035 or 5021).

Vapor partitioning can offer lower detection limits than methanol extraction because no dilution is involved. In addition, there are no organic solvent interferences and no use of regulated organic solvents (e.g., methanol), which requires special handling and disposal practices. Use of methanol may generate a flammable waste that is hazardous based on the ignitability characteristic (40 CFR § 261.21) or a listed waste (40 CFR § 261, Appendix VII).

### A.6.2 Methanol Extraction

Methanol extraction involves the extraction of VOCs from a sample with methanol, and the subsequent transfer of an aliquot of the extract to water (dilution) for either purge-and-trap or headspace analysis. After extraction with methanol (anywhere from 1:1 methanol to soil to a 10:1 methanol to soil ratio); the extract typically receives a 50-fold dilution. Methanol extracts must be diluted to minimize adverse effects of methanol on analytical instrumentation. However, solid samples with a significant moisture content (>10%) that are extracted prior to analysis in a water miscible solvent such as methanol are diluted by the total volume of the solvent/water mixture. (Ref. 51) The total mixture volume can only be calculated based on the sample moisture present as determined by the % moisture determination. Therefore, in order to report results for samples containing significant moisture contents on an "as received" basis, the detected concentration needs to be corrected by the solvent/water dilution factor. See Method 8000 for an explanation and the applicable calculations.

One advantage of a methanol extract is it may be tested more than once. Methanol extracts of soil are applicable to a wide range of high to low concentrations, e.g., 50 ppb to several ppm. Once a methanol extract is obtained, it can be stored at 4 ± 2 °C for two weeks, and sufficient volume is present for multiple VOC determinations. Additional extract storage time beyond two weeks may be acceptable if the desired VOC constituent stability can be demonstrated from appropriate performance data.

As noted above, concerns exist regarding the use of methanol extraction. The information to follow provides recent observations regarding the use of methanol for VOC analysis.

#### A.6.2.1 Contact Time Effect

Methanol extraction can provide more robust, larger or accurate values for VOCs when compared to vapor partitioning results. (Refs. 5,9,16,27,29,30,32,33,41,42) However, methanol extract results tend to increase with time as the sample contact time increases. (Refs 27,33) State agencies implementing methanol extraction for soil VOCs often require either a minimum contact time of one day, or the soil is to be sonicated for 20 to 30 minutes at 40° C with the methanol prior to analytical measurement of VOCs. The actual contact time should be sufficient enough to efficiently extract all VOC constituents of interest and to allow for the complete breakdown of agglomerated solid materials.

Particularly volatile VOCs (e.g., benzene, dichloroethene) in sandy soils are not expected to show this effect of contact time. The less volatile VOCs (e.g., xylenes) in an organic rich soil or clay can be expected to demonstrate higher results with increased contact time. (Refs. 5,9,27,33)

#### A.6.2.2 Safety and Hazardous Waste Generation Concerns

A primary disadvantage of methanol extraction is that it poses hazards to personnel due to its toxicity and flammability. Finally, the addition of methanol to a sample is likely to cause the sample to fail the ignitability characteristic or to become a listed waste, thereby making the unused sample volume a hazardous waste.

## A.7.0 SAMPLE COLLECTION

### A.7.1 Collection of Samples for Analysis

After a fresh surface of the solid material is exposed to the atmosphere, the subsample collection process should be completed in the least amount of time in order to minimize the loss of VOCs due to volatilization. Removing a subsample from a material should be done with the least amount of disruption (disaggregation) as possible. Additionally, rough trimming of the sampling location's surface layers should be considered if the material may have already lost VOCs (been exposed for more than a couple of minutes) or if it may be contaminated by other waste, different soil strata, or vegetation. Removal of surface layers can be accomplished by scraping the surface using a clean spatula, scoop, knife, or shovel. (Refs. 15,51)

#### A.7.1.1 Subsampling of Cohesive Granular but Uncemented Materials Using Devices Designed to Obtain a Sample Appropriate for Analysis

Subsamples of the appropriate size for analysis should be collected using a metal or rigid plastic coring tool. For example, coring tools for the purpose of transferring a subsample can be made from disposable plastic syringes by cutting off the tapered front end and removing the rubber cap from the plunger or can be purchased as either plastic or stainless steel coring devices. These smaller coring devices help to maintain the sample structure during collection and transfer to the VOA vial as do their larger counterparts used to retrieve subsurface materials. When inserting a clean coring tool into a fresh surface for sample collection, air should not be trapped behind the sample. If air is trapped, it could either pass through the sampled material causing VOCs to be lost or cause the sample to be pushed prematurely from the coring tool. The commercially available EasyDraw Syringe™ and Powerstop Handle™ and Terra Core™ sampler coring devices are designed to prevent headspace air above the sample contents. For greater ease in pushing into the solid matrix, the front edge of these tools can be sharpened. The optimum diameter of the coring tool depends on the following: size of the opening on the collection vial or bottle (tool should fit inside mouth), dimensions of the original sample, particle size of the solid materials (e.g., gravel-size particles would require larger samplers), and volume of sample required for analysis. For example when a 5-g subsample of soil is specified, only a single 3-$cm^3$ volume of soil has to be collected (assuming the soil has density of 1.7 $g/cm^3$). Larger subsample masses or more subsample increments are preferred as the heterogeneity of the material increases. After an undisturbed sample has been obtained by pushing the barrel of the coring tool into a freshly exposed surface and then removing the corer once filled, the exterior of the barrel should be quickly wiped with a clean disposable towel. The next step varies, depending on whether the coring device is used for sample storage and transfer or solely for transfer. If the coring tool is used as a storage container, cap the open end after ensuring that the sealing surfaces are cleaned. If the device is to be solely used for collection and not for storage, immediately extrude the sample into a VOA vial or bottle by gently pushing the plunger. The volume of material collected should not cause excessive stress on the coring tool during intrusion into the material, or be so large that the sample easily falls apart during extrusion. Obtaining and transferring a sample should be done rapidly (<10 seconds) to reduce volatilization losses. If the vial or bottle contains methanol or another liquid, it should be held at an angle when extruding the sample into the container to minimize splashing. Just before capping, a visual inspection of the lip and threads of the sample vessel should be made, and any foreign debris should be removed with a clean towel, allowing an airtight seal to form.

A.7.1.2 Devices that Can be Used for Subsampling a Cemented Material

The material requiring sampling may be so hard that even metal coring tools cannot penetrate it. Subsamples of such materials can be collected by fragmenting a larger portion of the material using a clean chisel to generate aggregate(s) of a size that can be placed into a VOA vial or bottle. When transferring the aggregate(s), precautions must be taken to prevent compromising the sealing surfaces and threads of the container. Losses of VOCs by using this procedure are dependent on the location of the contaminant relative to the surface of the material being sampled. Therefore, caution should be taken in the interpretation of the data obtained from materials that fit this description. As a last resort when this task can not be performed onsite, a large sample can be collected in a vapor-tight container and transported to the laboratory for subsampling. Collecting, fragmenting, and adding the sample to a container should be accomplished as quickly as possible.

A.7.1.3 Devices that Can be Used for Subsampling a Non-cohesive Granular Material

As a last resort, gravel, or a mixture of gravel and fines that can not be easily obtained or transferred using coring tools, can be quickly sampled using a stainless steel spatula or scoop. If the collection vial or bottle contains methanol or an aqueous solution, samples should be transferred with minimal splashing and without the spatula or scoop contacting the liquid contents. For some solids, a wide-bottom funnel or similar channeling device may be necessary to facilitate transfer to the container and prevent compromising of the sealing surfaces of the container. Caution should be taken in the interpretation of the data obtained from materials that fit this description. Losses of VOCs are likely because the nature of the sampling method and the noncohesive nature of the material expose more surface area to the atmosphere than other types of samples. During the sampling process, noncohesive materials also allow for the separation of coarser materials from fines, which can skew the concentration data if the different particle sizes, which have different surface areas, are not properly represented in the sample.

A.7.2 Use of the EnCore™ Sampler (or Equivalent) for Sample Transport and Storage

The EnCore™ sampler is a sampling device that can be used as both a simultaneous coring tool for cohesive soils and a transport device to a support laboratory (field or off-site). The EnCore™ sampler is intended to be a combined sampler-storage device for soils until a receiving laboratory can initiate either immediate VOC analysis, or preserve extruded soil aliquots for later VOC analysis. It is meant to be disposed after use. The commercially available device is constructed of an inert composite polymer. It uses a coring/storage chamber to collect either a 5 g or 25 g sample of cohesive soils. It has a press-on cap with hermetically vapor tight seal and locking arm mechanism. It also has a vapor tight plunger for the nondisruptive extrusion of the sample into an appropriate container for VOC analysis of soil.

An individual disposable EnCore™ sampler (or equivalent) is needed for each soil aliquot collected for vapor partitioning or methanol sample preparation. Upon soil sample collection, the EnCore™ sampler is stored at 4 ± 2°C until laboratory receipt within 48 hours. Upon laboratory receipt, soil aliquots are extruded to appropriate tared and prepared VOA vials.

Validation data have been provided to support use of the EnCore™ sampler for VOC concentrations in soil between 5 and 10 ppm, for two (2) sandy soils, with a 2-day holding time at 4 ± 2°C. Preliminary data (Ref. 25) demonstrate an effective 2-day (48-hour) holding time at 4 ±

2°C for three sandy soil types with VOC concentrations at 100 ppb (benzene and toluene at 300 ppb), as well as an effective 1 or 2 week holding time at -12°C (freezing temperature). Recent published work (Ref. 43) neither definitively supports or shows the EnCore™ device to be ineffective for sample storage at these preservation temperatures. Soils stored in the EnCore™ device for 2 calendar days at 4 ± 2°C are subject to loss of BTEX compounds by biodegradation if the soil is an aerated, biologically active soil (e.g., garden soil) (Ref. 24), but this BTEX loss is eliminated for up to 48 hours under freezing conditions. (Ref. 2)

Further details on the EnCore™ sampler can be found in ASTM D4547-98 (Ref. 15) or other publications.

A.7.3 Concerns Regarding Use of Core Barrel Liners

One geotechnical technique for retrieval of bulk soil from subsurface regions is ring-lined barrel samples. Core barrel liners fit snugly within a corer and can be constructed of steel or brass (which is inert to VOCs). Cylindrical cores of subsurface soil can be obtained anywhere from 1 to 4 inches in diameter of varying lengths in feet.

Core barrel liners have been used as both a sample collection and storage device for VOC soil samples. Upon retrieval with subsurface soil, the core barrel liner (brass) is covered on both ends with a thin sheet of PTFE or with aluminum foil. Plastic caps are pressed over the ends to hold the PTFE/aluminum in place. The core barrel liner sample is maintained at 4 ± 2°C during shipment and storage at a laboratory. Sample preparation for VOC analysis is initiated by opening the core barrel coverings and sub-sampling the soil with a coring tool for analyses by either the vapor partitioning or methanol extraction options.

Experimental work has demonstrated that the core barrel transport and storage procedure is ineffective for a 2-day storage and holding time. (Refs. 4,10,13,16, 36) PTFE coverings (0.02 mm and 0.05 mm thickness) and aluminum foil will not prevent losses of 30-90% for certain volatile compounds (dichloroethene, benzene and trichloroethene). Therefore, the core barrel liners should be used as sample collection and transfer devices only with the least amount of elapsed time as possible prior to sample preparation.

A.7.4 After Collection -- Sample Handling and Storage

A.7.4.1 Holding Times

Published holding times should be followed, unless performance data can be produced to support longer time periods.

This guidance assumes a 48-hour holding time, unpreserved at 4 ± 2°C, between sample collection and analysis or preservation of VOC soil aliquots in VOA vials. Most validation data provided to support or justify an approach listed the holding time as 48 hours. The 48-hour holding time results for VOC in soil can provide average recoveries of 80% or more. However, recoveries from samples stored for 5 days are less successful. Little data exists on the impact of holding times between 48 hours and 5 days.

Implementing a 48-hour holding time can be difficult when transporting VOC soil samples (via overnight air carrier) from the field to an off-site support laboratory. All interested parties i.e., field and laboratory personnel need to be cognizant that the 48 hour holding time begins **from the time of sample collection**. If the VOC analysis cannot be completed prior to the expiration of the initial 48 hour period, other

preservation measures (i.e., freezing, chemical preservation, and methanol extraction) are required in order to extend the analysis holding time to 14 days from the time of sample collection.

A.7.5 Quality Control

Quality control checks to be employed during field sampling activities should include the collection, preparation, and analysis of the various QC samples discussed below:

Note: The exact specifications and need for the following QC samples should be outlined in the project planning documents.

1. Field duplicate: A field duplicate may be prepared at a frequency of one per day per matrix. The field duplicate is an independent sample which is collected as close as possible to the same point in time and space as the primary field sample. Field duplicates are used to estimate the reproducibility (precision) of the sampling process.

2. Trip blank: Trip blanks should be prepared at a frequency of one per day of sampling during which samples will be collected for VOCs. Trip blanks are prepared using reagent water (see Chapter One for definition) prior to the site visit at the time sample containers and kits are transported to the site. The trip blank will accompany the field samples throughout all sample collection and transport operations. This blank will not be opened during sampling activities and will be used to assess sample contamination originating from sample transport, shipping, or site conditions.

3. Field blank: A field blank conversely is prepared on-site during the sample collection activities using the same reagent water source used to prepare the trip blank. The field blank should be collected and preserved in the same manner as the environmental samples. The results from this analysis are used to assess sample contamination originating predominantly from field sampling conditions.

4. Equipment rinsate: An equipment rinsate blank should be collected from sample collection devices used for each distinct sample matrix. The equipment blanks are obtained either prior to or during sample collection activities. The results from these analyses are used to assess possible sample contamination from sampling equipment.

5. Temperature blank: A temperature blank prepared with a water-filled vial or a suitable thermometer, should be included with each cooler of samples designated for transport. Upon sample receipt, the laboratory will use the temperature blank or thermometer to determine the internal temperature of each cooler. Acceptable temperatures are $4 \pm 2\,^{\circ}C$ for refrigerated aqueous and solid samples and $< -7\,^{\circ}C$ for frozen solid samples.

6. Matrix spike and matrix spike duplicate: Additional sample aliquots should be collected when matrix spike and matrix spike duplicate analyses are required. Matrix spikes are aliquots of environmental samples to which known concentrations of certain target analytes have been added before sample manipulation from the preparation, cleanup, and determinative procedures have been implemented. The matrix spike analysis is used to assess the performance of the method by measuring the effects of interferences caused by

the sample matrix and reflects the accuracy of the method for the particular matrix in question.

7.6 Interferences / Artifacts of Analysis

When aqueous and solid samples are acidified it can lead to losses of highly reactive compounds such as 2-chloroethylvinyl ether through chemical reaction. Additionally, acidification of certain soils with sodium bisulfate may produce a false positive acetone artifact of 100-200 ppb, or more. (Refs. 4,37) Furthermore, *meta*- and *para*-xylene co-elute on most analytical columns and need to be reported as an isomeric pair. Acid preservation of samples to be analyzed for methyl *tert*-butyl ether (MTBE) should be avoided because use of a high temperature sample preparation method (Methods 5021, 5030, or 5032) can cause degradation of the MTBE to *tert*-butyl alcohol (TBA) during the high temperature sample preparation step. (Refs. 49,50)

Since aqueous samples containing residual chlorine must be dechlorinated to prevent the formation of trihalomethanes and other chlorinated compounds, the sample should be added to the dechlorinating agent prior to acid preservation. The addition of sodium thiosulfate to an acidified sample will generate sulfur dioxide which may interfere with the determination of gaseous VOC constituents of interest.

The project chemist should research and review historical data pertaining to the use of VOCs at the site under investigation. If previous data indicates that tetrachloroethylene or trichloroethylene were used at the site and their daughter products dichloroethene and dichloroethane are present, then vinyl chloride may also be present. In this scenario acid preservation would not be appropriate due to the reactive nature of vinyl chloride.

If the sampling location is known to contain polymers that were manufactured from monomers, then both vinyl chloride or styrene could be present. For this situation, due to the potential for reactive compounds present, acid preservation would not be necessary.

Pre-testing of a representative soil sample, prior to collection, with acid or bisulfate may show effervescence if carbonaceous materials are present. If bubbling occurs during chemical preservation, samples should not be collected with acid or bisulfate preservative. If the soil sample is a loamy material or contains a high proportion of decayed matter then acid preservation may generate acetone as a byproduct. The sampling personnel should examine and pre-test the soils to be collected prior to actual collection in order to make the proper determination for the correct preservation technique.

The laboratory should fully document whenever sample matrix interferences are suspected and can be attributed to poor analytical method quality control data. It is also important for the laboratory area where volatile analyses are performed to be completely free of solvents. Special precautions must be taken for the analysis of methylene chloride, since random background levels will result if the analytical and storage areas are not isolated from all sources of atmospheric methylene chloride.

## A.8.0 APPROACHES TO SAMPLE PREPARATI0N

This section provides examples of approaches to sample preparation that include prepared vials (e.g., chemical preservation approaches) and use of empty vials (other means such as freezing used for preservation). Complete validation data is not available for all approaches. Analysts are responsible for showing that any given approach is appropriate for the intended use of the data.

Typically, as part of these procedures, a cohesive soil subsample (2 to 5g) from a freshly exposed sampling trench, geotechnical coring device/probe, etc., using a coring tool such as a cut-off syringe or purchased device (e.g., EasyDraw Syringe™ and Powerstop Handle™ or EnCore™), is extruded immediately to either a tared empty VOA vial or to a tared prepared VOA vial. Precautions with handling tared vials i.e., not applying additional labels, markings, and seals are necessary to ensure an accurate sample weight. Once filled with sample, the VOA vials are then capped (with PTFE-lined septa) until VOC sample preparation. Three or more replicate VOA vials (e.g., two for vapor partitioning and additional ones for any matrix spike QC analysis) are utilized by either technique, as well as one more soil aliquot for a percent moisture determination. One coring tool (disposable or reusable) can be used at each soil sampling location by providing co-located cores for the replicate VOA vials. The same coring tool can be used to collect an additional co-located soil for the percent moisture determination typically required by the laboratory preparation procedures. If the coring tool can be properly capped to prevent moisture loss, the coring tool can be used as a storage container for percent moisture. Note: should freezing be used as a means to preserve samples in the field, the aliquot reserved for percent moisture determination should not be frozen.

The preparation of samples for VOC analysis can be initiated either in the field at the time of collection using the prepared VOA vials, or at either an on- or off-site support laboratory using either the empty VOA vials (note the manual puncture of septa to introduce reagent water prior to analysis is not recommended) or a coring tool (e.g., the EnCore™ sampler) that can also serve as a sample transport device. A separate EnCore™ sampler is required for each replicate VOA vial used for VOC analysis.

When determining VOCs over the complete concentration range of ppb to several ppm, four (4) or more VOA vials may be required for each sampling point. For example, at least one VOA vial is necessary for methanol extraction when selected to analyze high VOC concentrations, while at least two vials are necessary for when vapor partitioning is to be used because low VOC concentrations (<200 ppb) are expected. A fourth VOA vial may be necessary for percent moisture determination so that VOC concentrations can be corrected for moisture content and/or methanol dilution factor, if required. A set of replicates for a single investigative soil sample are often composed of the following:

1. Two (2) 40-mL VOA vials for direct vapor partitioning measurement. These are needed for the most sensitive measurements - one is kept in reserve for any necessary repeat analysis. The upper concentration value of the vapor partitioning method's calibration range limits the usability of these direct measurements.

2. One (1) 40-mL VOA vial for methanol extraction of soil aliquot prior to vapor partitioning. Once a methanol extract is obtained, an aliquot of this extract is diluted fifty-fold (50) or more with water and is tested by vapor partitioning as a water matrix. The 50-fold dilution is necessary to minimize interferences in vapor partitioning measurements of water matrices. Methanol extracts have no

upper limit of measured VOC concentration since the extract can be sub-aliquoted for different dilutions.

3. One (1) 60-mL VOA vial for any percent moisture determination to report VOC results on a moisture corrected basis, if necessary. Also note that solid samples with a significant moisture content (>10%) that are extracted prior to analysis in a water miscible solvent such as methanol are diluted by the total volume of the solvent/water mixture. (Ref. 51) The total mixture volume can only be calculated based on the sample moisture present as determined by the % moisture determination. Therefore, in order to report this type of sample result on an "as received" basis, the detected concentration needs to be corrected by the solvent/water dilution factor. See Method 8000 for an explanation and the applicable calculations. The physical preservation (4 ± 2°C) of this vial is not as critical as for the VOC analytes in soil.

4. VOA vials for any QC audits such as duplicates, matrix spikes, etc.

Please note that a VOA vial should always be collected for methanol extraction unless it is known in advance that VOCs will not exceed the upper usable concentration values for direct vapor partitioning measurements.

Before presenting the different approaches using empty or prepared vials, a discussion is included regarding the study of the preservation of soils by freezing. As noted, this was studied using empty VOA vials. Some of the empty VOA vial approaches that follow in Sec. A.8.2 use freezing as a preservative.

A.8.1 Overview of Empty Vial Technique

Hewitt (Refs. 2, 4, 7,10) and Ricker (Refs. 19, 20, 21) independently developed “empty vial” techniques. Using a coring tool (Hewitt’s cut-off syringe or Ricker’s commercially available syringe and 5- to 13-g sample) a 5-g aliquot of undisturbed soil is transferred to a tared empty VOA vial and capped with a PTFE-lined septa (PTFE of 0.25 mm thickness). The two "empty vial" techniques were evaluated using methanol extraction (Method 5035) measurements.

The sealed vial with the soil aliquot is maintained either frozen (< -7°C), or at 4 ± 2°C until laboratory receipt and analysis. Multiple VOA vials can be collected, as necessary based on the sample preparation technique to be used. Sample vials should not be frozen below -20°C due to potential problems with vial seals and the loss of constituents upon sample thawing.

Upon laboratory receipt of VOA vials maintained at 4 ± 2°C (within 48 hours of sample collection), one “empty VOA vial” is selected for methanol extraction and the methanol reagent is added through the septum using a glass syringe equipped with a 23-gage Luer Lock needle. The methanol is mixed with the soil and any pressure can be relieved by cracking the VOA vial’s cap once. The methanol extract, stored at 4 ± 2 °C or less, has a shelf life of up to two weeks. Upon laboratory receipt of frozen VOC samples, a vial may be thawed and methanol added through the septa as described above.

To determine VOCs by vapor partitioning, “empty VOA vials” should have 10 mLs of reagent water added, either through the septa liner by a laboratory’s automated sampler at the time of analysis, or be present in the vial prior to sample collection (see Sec. A.8.3) when Method 5035 is used. For VOC samples maintained at 4 ± 2°C this must be done within 48 hours of sample collection. Experimental work by En Novative Technologies, Inc., and Hewitt (Refs. 44, 45) indicates that VOCs are slowly lost through the pierced septa after reagent water is manually added to an

"empty VOA vial," prior to Method 5035 purge and trap measurements. To avoid any clogging of the needle of an automated purge-and-trap system, reagent water or the sodium bisulfate solution can be present in the VOA vial (Sec. A.8.3) prior to sample collection, thereby, allowing the soil/solid to be dispersed prior to the purge-and-trap analysis.

Ricker and Hewitt in their experimental work demonstrated that the empty VOA vial, with a suitable PTFE-lined septa cap, has integrity for several days. Significant VOC losses do not occur at 4 ± 2 °C through the septum of the sealed VOA vial. A 48-hour holding time for soils, at 4 ± 2 °C storage of samples, has been found effective with the “empty VOA vial” for most target VOCs studied, except for aromatic compounds in biologically active, aerated garden soils (Refs. 2, 20). Hewitt studied freezing of soils (< -7 °C) as a preservative for soils, in conjunction with the “empty VOA vial” technique and found it effective for all target VOCs studied, including aromatic compounds, so long as freezing starts at the time of collection.

When soils are maintained at 4 ± 2°C for 48 hours until freezing starts, the same condition or stability is found for the VOCs except for benzene in biologically active soil. Use of freezing at the time of lab receipt of empty VOA vials can therefore simplify sample handling of soil materials. ASTM D 4547-98 (Ref. 15) and Method 5035 briefly mention freezing, but do not endorse it because data were not available at the time of their publication to support preservation by freezing. With this approach, chemical preservatives are not needed. VOA vials, maintained at < -7 °C, need only be thawed on the day of analysis, whether it be by vapor partitioning or by methanol extraction.

### A.8.2 Preservation Approaches Using Empty VOA Vials

This section provides five examples of approaches to sample preparation using empty VOA vials -- no preservatives or solutions are added to the vials.

#### A.8.2.1 Preservation by freezing (< -7 °C)

Upon collection, the soil is added to replicate empty vials and frozen at < -7 °C until thawed for analysis. The design of newer vials makes it possible to freeze the contents in an upright position, however, it may be advisable to place the vials on their side during the freezing process to prevent breakage. Freezing has been found effective to preserve both aromatic and chlorinated hydrocarbon VOCs in soil for two weeks at all VOC concentrations studied. (Refs. 2,4,11) Sample vials should not be frozen below -20 °C due to potential problems with vial seals and the loss of constituents upon sample thawing.

The on- or off-site support laboratory thaws a VOA vial when needed and either adds 5 or 10 mLs of methanol through the PTFE-lined septum using a 23-gage Luer lock syringe for methanol extraction and preservation. (Refs. 4,21,22) Addition of 5-10 mLs of water to the vial through the septum should not be performed, since this technique will create a punctured septum capable of producing VOC losses prior to purge-and-trap analysis.

This technique is unpopular for vapor partitioning because a prepared VOA vial with reagent water fits the operations of Methods 5021/5035 better than the empty VOA vial.

This technique can be undesirable when soil samples are transported to a support laboratory because dry ice, gel packs or salt-ice mixtures can be required to

maintain sub-zero temperature conditions during shipment. This technique has merit when freezers are available at a field site or on a sampling vessel.

A.8.2.2 Refrigerate VOA vials at 4 ± 2°C for 48 hours or less, then preserve by freezing at < -7°C upon laboratory receipt

Upon laboratory receipt, replicate soil VOA vials are frozen (< -7°C) then thawed as needed for preparation by methanol extraction, or if possible by vapor partitioning. Sample vials should not be frozen below -20°C due to potential problems with vial seals and the loss of constituents upon sample thawing. The 48-hour time period prior to freezing is practical and can be supported by the studies:

1. The chlorinated hydrocarbon volatiles that were studied have been found to be stable for two weeks at 4°C, with dichloroethene isomers not being as stable as other chlorinated compounds studied. (Refs. 1,2,4,7,11,16)

2. For spiked (at 5 ppm) typical soils, aromatic hydrocarbons demonstrate major losses at room temperature (22°C) after 5 days of storage. (Refs. 1,2,4) When these soil types are stored at 4°C, major losses occur between 10 and 14 days for aromatic hydrocarbons (e.g., benzene) spiked at 5 ppm. (Refs. 1,2,4) When these soil types are spiked at 30-40 ppb with aromatic hydrocarbons, major losses for benzene and toluene occur at 3-5 days of storage. (Refs. 2,4)

3. Aromatic hydrocarbons (such as benzene or toluene) when spiked into biologically active soil (aerated garden soil or fertilized soil) and stored at 4°C demonstrate losses of 20-30% within 48 hours. (Refs. 2,16,17,19,20). Limited disruption sampling techniques in conjunction with a maximum holding time of 48 hours can minimize this loss, but not eliminate it. Soils containing manure exhibited a major loss of aromatic hydrocarbons within one day while soil sterilization eliminated this loss. (Ref. 16)

4. Observed losses of aromatic or dichloroethene volatile compounds in soil, stored at 4°C, cease when soil is frozen at < -7°C. (Refs. 2,4).

A.8.2.3 Refrigerate VOA vials at 4 ± 2°C for 48 hours or less, then preserve with methanol upon laboratory receipt

Upon laboratory receipt, the volume of methanol necessary for methanol extraction sample preparation is added to one of the replicate VOA vials through the PTFE-lined septum cap, using a 23-gage needle on a Luer lock syringe. Methanol will preserve VOCs in soil for 2 weeks if stored at 4 ± 2°C. See Sec. A.8.2.2 above for discussion on initial ≤48-hour transport at 4 ± 2°C. Certain PTFE-lined septa caps were found to be effective seals for 10 days prior to the addition of methanol. (Refs. 19,20,21)

When methanol is added through the septum cap to a soil aliquot core in an empty VOA vial, the mixture is swirled to provide contact with the soil and methanol, to wet the head space, and dissolve gaseous and sorbed VOC compounds into the methanol. At this point, there can be a pressure build-up within the vial that can be removed by cracking the VOA vial cap and immediately resealing it. (Ref. 4) There is believed not to be significant VOC loss so long as the methanol remains in contact with

the soil material. The methanol extraction efficiency can be improved by sonicating and heating the mixture at 40°C for 30 minutes followed by centrifuging and transferring the supernatant to a disposable, screw-top glass centrifuge tube. (Ref. 33)

A.8.2.4 Refrigerate VOA vials at 4 ± 2°C for 48 hours or less and complete VOC analysis (Method 5021/5035) within 48 hours

VOC sample preparation by vapor partitioning is completed within 48 hours from sample collection. See Secs. A.8.2.2 and A.8.2.3 above for further details.

A.8.2.5 Refrigerate/freeze coring tool used as transport device for 48 hours or less (Refs. 15,26)

Each replicate soil aliquot is collected by a suitable coring device, (e.g., EnCore™) that is used as a transport device to the laboratory. Upon laboratory receipt, soil aliquots from each replicate transport device are extruded into individual empty or prepared tared VOA vials as noted in Secs. A.8.2.2 to A.8.2.4. Upon cap closure, the vial is weighed again and the wet sample weight is determined by difference.

For spiked soils characteristic of a waste site, some VOC losses were observed in 2 days for soils stored at 4 ± 2°C and losses continued further at a 5-day and 12-day storage time period. Losses during the first 2 days for aromatics and dichloroethene, were equivalent to the empty vial techniques as noted in Sec. A.8.2.2. (Ref. 4) Also, sampling of TCE contaminated soil showed reasonable agreement between the EnCore™ and cut-off syringe/empty vial techniques. (Ref. 4) Significant losses after 2 days at 4°C have been observed for the EnCore™ for biologically active soils. (Refs.16,24).

The EnCore™ sampler has been systematically evaluated for three sandy soil types (at high VOC concentrations (5 -10 ppm) and at low VOC concentrations (100 ppb). (Refs. 22,23,24,25). The EnCore™ was effective as a 2-day transport device when stored at 4 ± 2°C, for the above studies, and storage could be extended from 1 week to 12 days further under freezing conditions (< -7°C) during the low VOC concentration study. (Ref. 25) The EnCore™ was ineffective for one soil type using high concentration spikes, because the soil was non-cohesive (dry clumped sand) - any coring device could be ineffective. (Refs. 15,22) The three soils exhibited little biodegradation of aromatic hydrocarbons discussed above.

For the original EnCore™ of stainless steel construction, it was found to be the only sampling/storage device that was as effective as the original single vial technique (Dynatech vial of January 1995 draft Method 5035). (Ref. 16)

A.8.3 Preservation Approaches Using Prepared VOA Vials

This section provides four examples of preservation approaches using prepared VOA vials. During sample collection, a coring tool is used to extrude the collected sample into a VOA vial containing methanol. Co-located soil cores are extruded into replicate VOA vials containing reagent water, or reagent water acidified with 1 g $NaHSO_4$ per 5 mLs water.

Coordination between field and laboratory personnel is required so specific vials and reagents are consistent with laboratory instrumentation and reagents. Vials with reagents, and any magnetic stirring bars (e.g., for Method 5035) need be tared prior to field use. If prepared VOA vials contain methanol or water they must be tared with the septum caps and the added reagent. Once

methanol or water reagent is added, a meniscus level of the liquid in the VOA vial can be marked. This allows field personnel to note any apparent liquid loss (especially methanol) during shipment to the field. If field personnel are concerned with reagent weight loss during shipment to the field and return, individual vials can be periodically weighed after initial tare or after addition of cored soil aliquot.

A.8.3.1 Collection with reagent water, preservation by freezing (< -7°C) and analysis by vapor partitioning

Extrude collected soil from a coring device into a VOA vial containing 5 mLs water (Method 5035), turn vial on its side and freeze contents. It may be problematical to freeze 10 mLs of water in the 22 ml vial used for Method 5021. Maintain at < -7°C until thawed for analysis. Sample vials should not be frozen below -20°C due to potential problems with vial seals and the loss of constituents upon sample thawing. Few published data exist to validate this preservation technique, but its effectiveness is inferred from Sec. A.8.2.1, and should be demonstrated by appropriate performance data results. (Ref. 28)

A.8.3.2 Collection with reagent water, preservation by refrigeration at 4 ± 2°C for 48 hours or less and immediate laboratory analysis or freezing storage at < -7°C for subsequent vapor partitioning

Sample is collected as in Sec. A.8.3.1 but transported to the laboratory within 48 hours at 4 ± 2 °C for:

1. Immediate analysis by vapor partitioning within 48 hours of sample collection.

2. Freezing at < -7°C upon laboratory receipt for vapor partitioning analysis within 2 weeks from sample collection. Sample vials should not be frozen below -20°C due to potential problems with vial seals and the loss of constituents upon sample thawing.

One investigator has found that a spiked hazardous waste site soil provided the same results one week after freezing with water as the initial spiked soil results. (Ref 28). Another investigator used headspace techniques with soil added to 10 mLs of reagent water to develop justification for certain variables discussed in Sec. A.8.3.2 for the initial 48-hour holding time. (Refs. 6,12) Sec. A.8.2.2 should be consulted for biodegradation effects for aromatic hydrocarbons. This technique allows the laboratory to observe the dispersion of soils in water and take any corrective action prior to purge-and-trap analysis. This technique is also most consistent with automated purge-and-trap samplers where stirring occurs prior to the purge cycle.

A.8.3.3 Collection with 5 mLs of water and 1 g of $NaHSO_4$ and analysis by vapor partitioning

Extrude collected soil from a coring device into a VOA vial containing 5 mLs of reagent water and 1 g of $NaHSO_4$ for vapor partitioning by Method 5035. For a spiked soil, $NaHSO_4$ was found to preserve the aromatic hydrocarbons at room temperature for more than 2 weeks. (Refs.1,11) The same soil showed major losses of aromatic hydrocarbons (5-10 ppm) when stored at room temperature for 5 days or at 4 ± 2°C after 10 days when no $NaHSO_4$ was present. (Ref. 1) The studied chlorinated hydrocarbons demonstrated insignificant losses during these storage conditions. The

use of $NaHSO_4$ with sample acidification to pH 2 or less eliminates the biodegradation of the important aromatic hydrocarbon volatile compounds.

1 g of $NaHSO_4$ will acidify 5 g of soil with an alkaline content (as $CaCO_3$) of 5%. It is insufficient to neutralize a soil with an alkaline content of 10%. This technique has been found to be somewhat problematic since publication of Method 5035. Carbonaceous soils cause effervescence of the acidic soil slurry with loss of volatiles and even cause failure of the septa VOA vial cap or even the VOA vial itself. Upon acidification, certain soils exhibit a false positive acetone artifact of 100-200 ppb, or more. (Refs. 4,37) The $NaHSO_4$ corrosive vapors may cause increased purge-and-trap maintenance by laboratories due to creation of active sites on the trapping material. A very few target compounds such as styrene, vinyl chloride, and 2-chloroethylvinyl ether react under acidic conditions and are not detected. Note that the sodium chloride matrix modifying reagent of Method 5021 was found to be as effective as $NaHSO_4$ for inhibiting biodegradation of aromatic hydrocarbons in soil and may be more advantageous to use with calcareous soils, since the inhibitory agent is not dependent on the concentration of hydrogen ion present. (Ref. 1)

A.8.3.4 Collection and preservation with methanol at 4 ± 2° C

Extrude collected soil from a coring device into a VOA vial containing 5 -10 mLs of methanol. Larger volumes of methanol may be used if compositing of soils is required. Methanol preservation is effective for 2 weeks if stored at 4 ± 2°C. Also, one investigator has found methanol preservation of a sand spiked with gasoline to be effective when traditional techniques were ineffective. (Ref. 36)

## A.9.0 SUMMARY OF FINDINGS

1. An aqueous sample holding time period can be extended to fourteen days with the use of chemical preservatives such as sodium bisulfate or hydrochloric acid, however, since reactive compounds such as 2-chloroethylvinyl ether are unstable at low pHs, if these types of analytes are to be determined, the collection of a second set of samples without acid preservatives is necessary. Aqueous samples containing methyl *tert*-butyl ether and other fuel oxygenate ethers should not be acidified if high temperature sample preparative methods (Methods 5021, 5030, 5032) are used. (Refs. 49,50) (Sec. A.3.0)

2. The solid material to be characterized should be sampled with limited disruption (e.g., by a coring device for cohesive soils) and single transfer to an air tight VOA vial (PTFE-lined septa cap) that will be used for storage and preparation for VOC analysis. (Sec. A.7.1)

3. Data have been published or presented to validate different storage devices, procedures, preservative reagents and techniques for the VOC analysis of aqueous and solid samples. A wide range of recovery results have been observed. Acceptable devices, procedures, preservatives, and techniques should provide an average recovery of greater than 80% for important volatile contaminants such as benzene, dichloro- and trichloroethanes/ethenes. A recovery of 80% may be difficult for gaseous VOC contaminants such as vinyl chloride and chloroethane; however, the acceptability of a procedure should not be solely based on the less volatile VOCs such as chlorobenzene, xylenes, and trimethyl benzene. (Secs. A.2.0, A.6.0, A.7.0 and A.8.0)

4. VOCs in solids can be successfully sampled using coring tools (usually 5-g aliquots but can be 2 to 25 g) if the material is cohesive. Sampling procedures are not available to prevent VOC loss during sampling of non-cohesive soil material (dry sand, gravel, liquid sediment) or cemented material. (Secs. A.4.0 and A.7.0)

5. The following two techniques have been found accurate (minimal VOC loss) for preparation of soils for VOC analysis; however, they are not without problems:

   a. Soil is added to empty VOA vials at time of collection and is frozen at < -7°C until thawed for analysis. Validation data have not been provided yet, but it is believed that a prepared VOA vial with reagent water only is also acceptable for low concentration VOC in soil (<200 ppb) if frozen at < -7°C at time of collection.

   b. Soil is added to a prepared VOA vial, with methanol reagent, at time of collection and stored at 4 ± 2°C until time of analysis. This is applicable only to VOC in soil concentrations greater than 50 ppb. (Sec. A.6.2) (See comments below regarding use of methanol.)

6. The following techniques have been found to be the most practical, currently available alternatives for preparation of soil for VOC analysis. Validation data are not available to fully support their use for all types of soil or to fully differentiate them in accuracy relative to each other. The techniques rely on transport of sealed VOA vials or coring tools, at 4 ± 2°C, to a support laboratory within 48

hours where they are preserved/stored appropriately or immediately tested for VOCs. As more validation data and experience occur with time, their relative worth will become more apparent. The techniques listed below are superior to the traditional procedures of ten years ago.

a. Soil is added to tared replicate "empty VOA vials" at time of collection, preserved, refrigerated at 4 ± 2°C until laboratory receipt within 48 hours, and then preserved by freezing (< -7°C). Individual vials are thawed prior to sample preparation within 14 days of collection. A thawed vial must be processed within 24 hours by either screening using methanol extraction or analysis by vapor partitioning. At time of laboratory receipt, laboratories have the option of immediately testing a soil by vapor partitioning where the required reagent water is added through the PTFE-lined septa cap using the automated instrument sampling devices after weighing an "empty VOA vial" and obtaining wet sample weight by difference. In addition at time of laboratory receipt, laboratories have the option of immediately preparing a soil for methanol extraction by weighing an "empty VOA vial," obtaining the wet sample weight by difference, then adding methanol reagent through the PTFE-lined septa cap using a 23-gage needle on a Luer lock syringe. The sample-methanol mixture is shaken for 15 seconds to wet the vial's head space. The vial cap is opened once to vent pressure and then closed for the extraction process. (Sec. A.8.0)

b. For carbonate-containing soils (or soils suspected as such), ASTM D4547-98 (Ref. 15) provides for adding 2 to 5 g of soil (using coring tool) to tared, replicate prepared VOA vial containing 5 mLs of reagent water. Prepared VOA vials are maintained at 4 ± 2°C until laboratory receipt within 48 hours, and immediately tested for VOCs by vapor partitioning. This approach offers the advantage of mixing and dispersing the soil into the water and to observe any problematic samples prior to vapor partitioning analysis. Alternatively, the reagent water prepared VOA vials may be preserved by freezing (< -7°C) by placing vials in horizontal position. This technique is an alternative or fall-back from the prepared VOA vial with acidified reagent water; however, little or no data are available to validate its use. (Sec. A.5.3)

c. Soil is collected in replicate "Coring Tool Used as Transport Device" (e.g., the EnCore™ sampler), maintained at 4 ± 2 °C until laboratory receipt within 48 hours, then extruded into individual "Empty VOA Vials" for preservation by freezing (< -7°C) or into prepared VOA vials for immediate analysis by vapor partitioning or for sample preparation by methanol extraction. (Sec. A.7.2)

d. For known non-carbonate soils, a coring tool soil aliquot for BTEX type VOC analysis is added to a tared prepared VOA vial containing 5 mLs reagent water acidified with 1g $NaHSO_4$. The prepared VOA vial is maintained at 4 ± 2°C for BTEX testing by vapor partitioning within 14 days of sample collection. Acidified reagent water has been problematic when applied to a wide range of soil types for a large analyte list; however, it is effective for the volatile BTEX compounds in known non-carbonate soils. It is a specialized, preservation technique that minimizes aromatic VOC losses from biodegradation at 4 ± 2 °C.

Acetone artifacts are sometimes observed in soil samples preserved with $NaHSO_4$.

7. Use of a prepared VOA vial with acidified ($NaHSO_4$) reagent water is not recommended as a primary preservation technique for all soil types and a broad VOC analyte list. This technique is applicable to volatile aromatic hydrocarbons in soils known not to contain carbonates as discussed above.

8. A longer holding time may be appropriate if it can be demonstrated that the reported VOC concentrations are not adversely affected from storage and analyses performed outside the recommended holding times.

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# APPENDIX C

Soil Vapor Sampling Guidance

# FINAL

# Guidance for Evaluating Soil Vapor Intrusion in the State of New York

October 2006

Prepared by:

**NEW YORK STATE DEPARTMENT OF HEALTH**
Center for Environmental Health
Bureau of Environmental Exposure Investigation

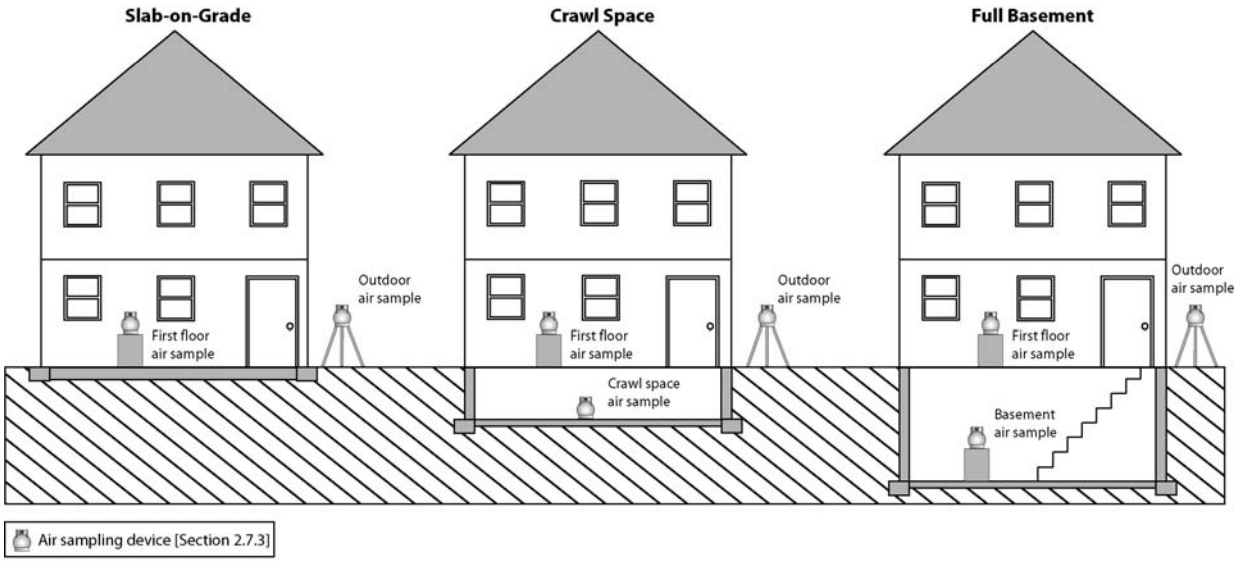


**Figure 2.1**
Schematic of indoor and outdoor air sampling locations

2.6.4 Outdoor air

Typically, an outdoor air sample is collected outside of each building where an indoor air sample is collected. However, if several buildings are being sampled within a localized area, representative outdoor air samples may be appropriate. For example, one outdoor air sample may be sufficient for three houses being sampled in a cul-de-sac. Outdoor air samples should be collected from a representative upwind location, away from wind obstructions (e.g., trees or bushes), and at a height above the ground to represent breathing zones (3 to 5 feet) [Figure 2.1]. A representative sample is one that is not biased toward obvious sources of volatile chemicals (e.g., automobiles, lawn mowers, oil storage tanks, gasoline stations, industrial facilities, etc.). For buildings with HVAC systems that draw outdoor air into the building, an outdoor air sample collected near the outdoor air intake may be appropriate.

## 2.7 Sampling protocols

The procedures recommended here may be modified depending on site-specific conditions, the sampling objectives, or emerging technologies and methodologies. Alternative sampling procedures should be described thoroughly and proposed in a work plan submitted for review by the State. The State will review and comment on the proposed procedure and consider the efficacy of the alternative sampling procedure based on the objectives of investigation. In all cases, work plans should thoroughly describe the proposed sampling procedure. Similarly, the procedures that were implemented in the field should be documented and included in the final report of the sampling results.

### 2.7.1 Soil vapor

Soil vapor probe installations [Figure 2.2] may be permanent, semi-permanent or temporary. In general, permanent or semi-permanent installations are preferred for data consistency reasons and to ensure outdoor air infiltration does not occur. Temporary probes should only be used if measures are taken to ensure that an adequate surface seal is created to prevent outdoor air infiltration and if tracer gas is used at every sampling location. [See Section 2.7.5 for additional information about the use of tracer gas when collecting soil vapor samples.] Soil vapor implants or probes should be constructed in the same manner at all sampling locations to minimize possible discrepancies. The following procedures should be included in any permanent construction protocol:

a. implants should be installed using an appropriate method based on site conditions (e.g., direct push, manually driven, auger — if necessary to attain the desired depth or if sidewall smearing is a concern, etc.);

b. porous, inert backfill material (e.g., glass beads, washed #1 crushed stone, etc.) should be used to create a sampling zone 1 to 2 feet in length;

c. implants should be fitted with inert tubing (e.g., polyethylene, stainless steel, nylon, Teflon®, etc.) of the appropriate size (typically 1/8 inch to 1/4 inch diameter) and of laboratory or food grade quality to the surface;

d. soil vapor probes should be sealed above the sampling zone with a bentonite slurry for a minimum distance of 3 feet to prevent outdoor air infiltration and the remainder of the borehole backfilled with clean material;

e. for multiple probe depths, the borehole should be grouted with bentonite between probes to create discrete sampling zones or separate nested probes should be installed [Figure 2.2]; and

f. steps should be taken to minimize infiltration of water or outdoor air and to prevent accidental damage (e.g., setting a protective casing around the top of the probe tubing and grouting in place to the top of bentonite, sloping the ground surface to direct water away from the borehole like a groundwater monitoring well, etc.).

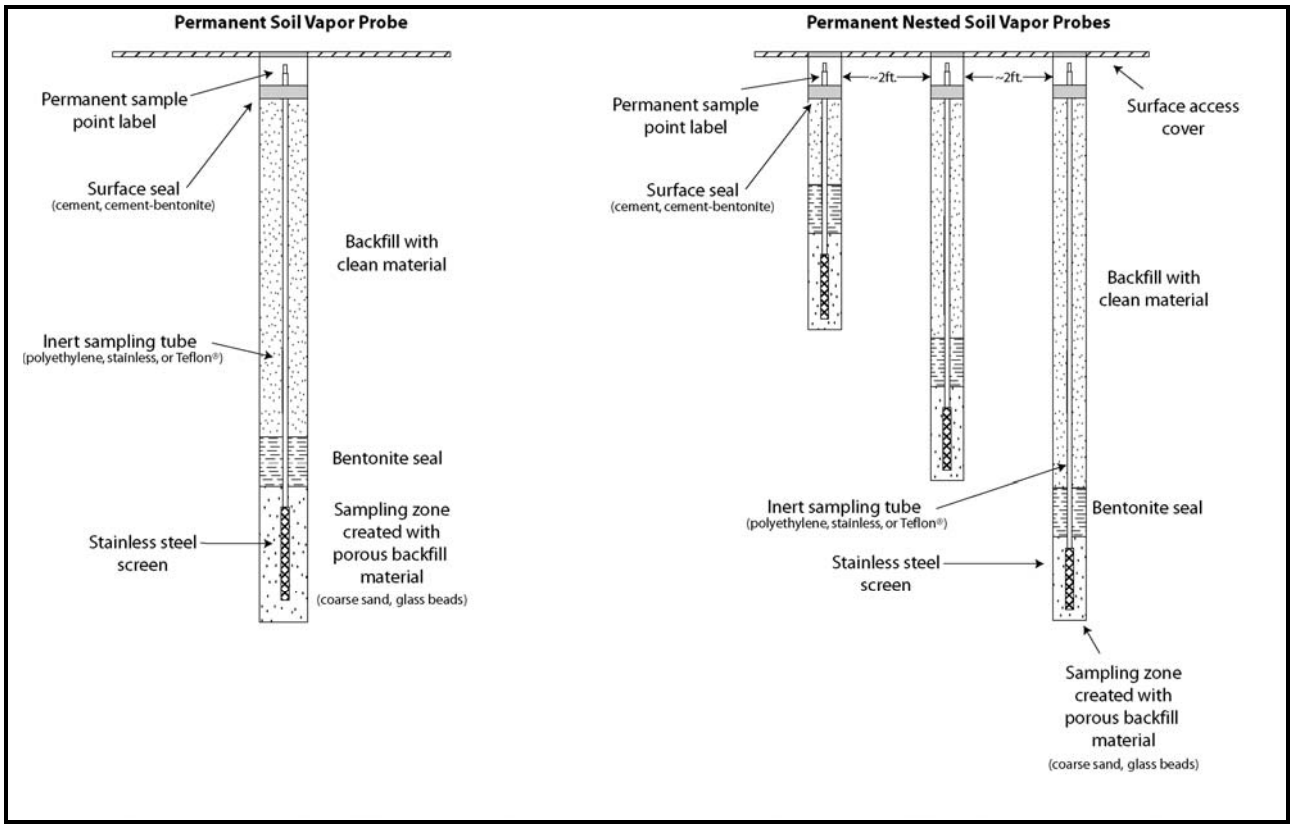


**Figure 2.2**

Schematics of a generic permanent soil vapor probe
and permanent nested soil vapor probes

[Note: Many variations exist and may be proposed in a work plan. Proposed installations should meet the sampling objectives and requirements of the analytical methods.]

To obtain representative samples and to minimize possible discrepancies, soil vapor samples should be collected in the following manner at all locations:

a. at least 24 hours after the installation of permanent probes and shortly after the installation of temporary probes, one to three implant volumes (i.e., the volume of the sample probe and tube) should be purged prior to collecting the samples;

b. flow rates for both purging and collecting should not exceed 0.2 liters per minute to minimize outdoor air infiltration during sampling;

c. samples should be collected, using conventional sampling methods, in an appropriate container — one which

   i. meets the objectives of the sampling (e.g., investigation of areas where low or high concentrations of volatile chemicals are expected; to minimize losses of volatile chemicals that are susceptible to photodegradation),

   ii. is consistent with the sampling and analytical methods (e.g., low flow rate; Summa® canisters if analyzing by using EPA Method TO-15), and

   iii. is certified clean by the laboratory;

d. sample size depends upon the volume of that will achieve minimum reporting limits [Section 2.9]; and

e. a tracer gas (e.g., helium, butane, sulfur hexafluoride, etc.) should be used when collecting soil vapor samples to verify that adequate sampling techniques are being implemented (i.e., to verify infiltration of outdoor air is not occurring) [Section 2.7.5].

In some cases, weather conditions may present certain limitations on soil vapor sampling. For example, condensation in the sample tubing may be encountered during winter sampling due to low outdoor air temperatures. Devices, such as tube warmers, may be used to address these conditions. Anticipated limitations to the sampling should be discussed prior to the sampling event so appropriate measures can be taken to address these difficulties and produce representative and reliable data.

When soil vapor samples are collected, the following actions should be taken to document local conditions during sampling that may influence interpretation of the results:

a. if sampling near a commercial or industrial building, uses of volatile chemicals during normal operations of the facility should be identified;

b. outdoor plot sketches should be drawn that include the site, area streets, neighboring commercial or industrial facilities (with estimated distance to the site), outdoor air sampling locations (if applicable), and compass orientation (north);

c. weather conditions (e.g., precipitation and outdoor temperature) should be noted for the past 24 to 48 hours; and

d. any pertinent observations should be recorded, such as odors and readings from field instrumentation.

Additional information that could be gathered to assist in the interpretation of the results includes barometric pressure, wind speed and wind direction.

The field sampling team should maintain a sample log sheet summarizing the following:

a. sample identification,

b. date and time of sample collection,

c. sampling depth,

d. identity of samplers,

e. sampling methods and devices,

f. purge volumes,

g. volume of soil vapor extracted,

h. if canisters used, the vacuum before and after samples were collected,

i. apparent moisture content (dry, moist, saturated, etc.) of the sampling zone, and

j. chain of custody protocols and records used to track samples from sampling point to analysis.

2.7.2 Sub-slab vapor

During colder months, heating systems should be operating to maintain normal indoor air temperatures (i.e., 65 – 75 °F) for at least 24 hours prior to and during the scheduled sampling time. Prior to installation of the sub-slab vapor probe, the building floor should be inspected and any penetrations (cracks, floor drains, utility perforations, sumps, etc.) should be noted and recorded. Probes should be installed at locations where the potential for ambient air infiltration via floor penetrations is minimal.

Sub-slab vapor probe installations [Figure 2.3] may be permanent, semi-permanent or temporary. A vacuum should not be used to remove drilling debris from the sampling port. Sub-slab implants or probes should be constructed in the same manner at all sampling locations to minimize possible discrepancies. The following procedures should be included in any construction protocol:

a. permanent recessed probes should be constructed with brass or stainless steel tubing and fittings;

b. temporary probes should be constructed with inert tubing (e.g., polyethylene, stainless steel, nylon, Teflon®, etc.) of the appropriate size (typically 1/8 inch to 1/4 inch diameter), and of laboratory or food grade quality;

c. tubing should not extend further than 2 inches into the sub-slab material;

d. porous, inert backfill material (e.g., glass beads, washed #1 crushed stone, etc.) should be added to cover about 1 inch of the probe tip for permanent installations; and

e. the implant should be sealed to the surface with non-VOC-containing and non-shrinking products for temporary installations (e.g., permagum grout, melted beeswax, putty, etc.) or cement for permanent installations.

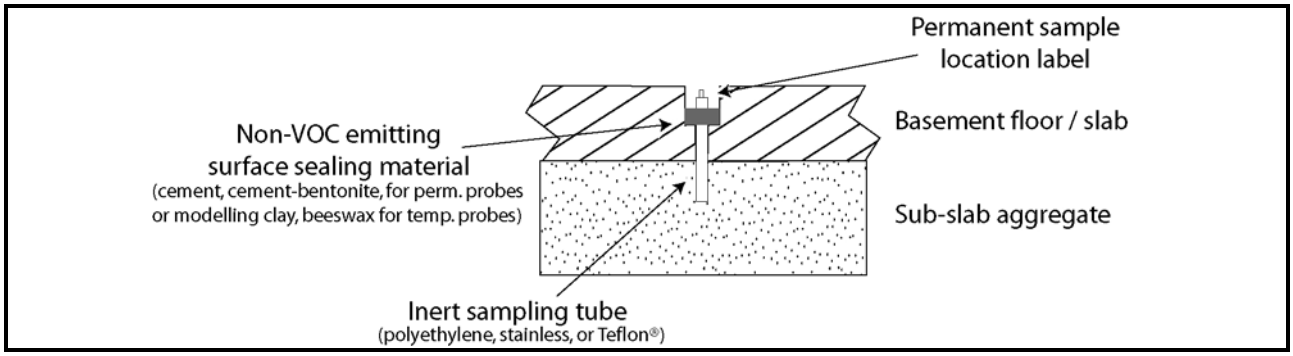


**Figure 2.3**

Schematic of a generic sub-slab vapor probe

[Note: Many variations exist and may be proposed in a work plan. Proposed installations should meet the sampling objectives and requirements of the analytical methods.]

To obtain representative samples that meet the data quality objectives, sub-slab vapor samples should be collected in the following manner:

a. after installation of the probes, one to three volumes (i.e., the volume of the sample probe and tube) must be purged prior to collecting the samples to ensure samples collected are representative;

b. flow rates for both purging and collecting must not exceed 0.2 liters per minute to minimize ambient air infiltration during sampling; and

c. samples should be collected, using conventional sampling methods, in an appropriate container — one which

    i. meets the objectives of the sampling (e.g., investigation of areas where low or high concentrations of volatile chemicals are expected; to minimize losses of volatile chemicals that are susceptible to photodegradation),

    ii. is consistent with the sampling and analytical methods (e.g., low flow rate; Summa® canisters if analyzing by using EPA Method TO-15), and

    iii. is certified clean by the laboratory;

d. sample size depends upon the volume of that will achieve minimum reporting limits [Section 2.9], the flow rate, and the sampling duration; and

e. ideally, samples should be collected over the same period of time as concurrent indoor and outdoor air samples.

When sub-slab vapor samples are collected, the following actions should be taken to document conditions during sampling and ultimately to aid in the interpretation of the sampling results [Section 3]:

a. historic and current storage and uses of volatile chemicals should be identified, especially if sampling within a commercial or industrial building (e.g., use of volatile chemicals in commercial or industrial processes and/or during building maintenance);

b. the use of heating or air conditioning systems during sampling should be noted;

c. floor plan sketches should be drawn that include the floor layout with sampling locations, chemical storage areas, garages, doorways, stairways, location of basement sumps or subsurface drains and utility perforations through building foundations, HVAC system air supply and return registers, compass orientation (north), footings that create separate foundation sections, and any other pertinent information should be completed;

d. outdoor plot sketches should be drawn that include the building site, area streets, outdoor air sampling locations (if applicable), compass orientation (north), and paved areas;

e. weather conditions (e.g., precipitation and indoor and outdoor temperature) and ventilation conditions (e.g., heating system active and windows closed) should be reported; and

f. any pertinent observations, such as spills, floor stains, smoke tube results, odors and readings from field instrumentation (e.g., vapors via PID, ppbRAE, Jerome Mercury Vapor Analyzer, etc.), should be recorded.

Additional documentation that could be gathered to assist in the interpretation of the results includes information about air flow patterns and pressure relationships obtained by using smoke tubes or other devices (especially between floor levels and between suspected

contaminant sources and other areas), the barometric pressure and photographs to accompany floor plan sketches.

The field sampling team should maintain a sample log sheet summarizing the following:

a. sample identification,
b. date and time of sample collection,
c. sampling depth,
d. identity of samplers,
e. sampling methods and devices,
f. soil vapor purge volumes,
g. volume of soil vapor extracted,
h. if canisters used, vacuum of canisters before and after samples collected,
i. apparent moisture content (dry, moist, saturated, etc.) of the sampling zone, and
j. chain of custody protocols and records used to track samples from sampling point to analysis.

### 2.7.3 Indoor air

[Reference: NYSDOH's *Indoor Air Sampling & Analysis Guidance (February 1, 2005)*]

During colder months, heating systems should be operating to maintain normal indoor air temperatures (i.e., 65 – 75 °F) for at least 24 hours prior to and during the scheduled sampling time. If possible, prior to collecting indoor samples, a pre-sampling inspection [Section 2.11.1] should be performed to evaluate the physical layout and conditions of the building being investigated, to identify conditions that may affect or interfere with the proposed sampling, and to prepare the building for sampling. This process is described in Section 2.11.1.

In general, indoor air samples should be collected in the following manner:

a. sampling duration should reflect the exposure scenario being evaluated without compromising the detection limit or sample collection flow rate (e.g., an 8 hour sample from a workplace with a single shift versus a 24 hour sample from a workplace with multiple shifts). To ensure that air is representative of the locations sampled and to avoid undue influence from sampling personnel, samples should be collected for at least 1 hour. If the goal of the sampling is to represent average concentrations over longer periods, then longer duration sampling periods may be appropriate. Typically, 24 hour samples are collected from residential settings;
b. personnel should avoid lingering in the immediate area of the sampling device while samples are being collected;
c. sample flow rates must conform to the specifications in the sample collection method and, if possible, should be consistent with the flow rates for concurrent outdoor air and sub-slab samples; and
d. samples must be collected, using conventional sampling methods, in an appropriate container — one which

i. meets the objectives of the sampling (e.g., investigation of areas where low or high concentrations of volatile chemicals are expected; to minimize losses of volatile chemicals that are susceptible to photodegradation),

ii. is consistent with the sampling and analytical methods (e.g., low flow rate; Summa® canisters if analyzing by using EPA Method TO-15), and

iii. is certified clean by the laboratory.

At sites with tetrachloroethene contamination, passive air monitors that are specifically analyzed for tetrachloroethene (i.e., "perc badges") are commonly used to collect indoor and outdoor air samples. If site characterization activities indicate that degradation products of tetrachloroethene also represent a vapor intrusion concern, perc badges may be used to indicate the likelihood of vapor intrusion (i.e., by using tetrachloroethene as a surrogate) followed, as appropriate, by more comprehensive sampling and laboratory analyses to quantify both tetrachloroethene and its degradation products. Perc badge samples ideally should be collected over a twenty-four hour period, but for no less than eight hours.

The following actions should be taken to document conditions during indoor air sampling and ultimately to aid in the interpretation of the sampling results [Section 3]:

a. historic and current uses and storage of volatile chemicals should be identified, especially if sampling within a commercial or industrial building (e.g., use of volatile chemicals in commercial or industrial processes and/or during building maintenance);

b. a product inventory survey documenting sources of volatile chemicals present in the building during the indoor air sampling that could potentially influence the sample results should be completed [Section 2.11.2];

c. the use of heating or air conditioning systems during sampling should be noted;

d. floor plan sketches should be drawn that include the floor layout with sampling locations, chemical storage areas, garages, doorways, stairways, location of basement sumps or subsurface drains and utility perforations through building foundations, HVAC system supply and return registers, compass orientation (north), footings that create separate foundation sections, and any other pertinent information should be completed;

e. outdoor plot sketches should be drawn that include the building site, area streets, outdoor air sampling locations (if applicable), compass orientation (north), and paved areas;

f. weather conditions (e.g., precipitation and indoor and outdoor temperature) and ventilation conditions (e.g., heating system active and windows closed) should be reported; and

g. any pertinent observations, such as spills, floor stains, smoke tube results, odors and readings from field instrumentation (e.g., vapors via PID, ppbRAE, Jerome Mercury Vapor Analyzer, etc.), should be recorded.

Additional documentation that could be gathered to assist in the interpretation of the results includes information about air flow patterns and pressure relationships obtained by using smoke tubes or other devices (especially between floor levels and between suspected contaminant sources and other areas), the barometric pressure and photographs to accompany floor plan sketches.

The field sampling team should maintain a sample log sheet summarizing the following:

a. sample identification,
b. date and time of sample collection,
c. sampling height,
d. identity of samplers,
e. sampling methods and devices,
f. depending upon the method, volume of air sampled,
g. if canisters are used, vacuum of canisters before and after samples collected, and
h. chain of custody protocols and records used to track samples from sampling point to analysis.

### 2.7.4 Outdoor air

Outdoor air samples should be collected simultaneously with indoor air samples to evaluate the potential influence, if any, of outdoor air on indoor air quality. They may also be collected simultaneously with soil vapor samples to identify potential outdoor air interferences associated with infiltration of outdoor air into the sampling apparatus while the soil vapor was collected. To obtain representative samples that meet the data quality objectives, outdoor air samples should be collected in a manner consistent with that for indoor air samples (described in Section 2.7.3).

The following actions should be taken to document conditions during outdoor air sampling and ultimately to aid in the interpretation of the sampling results [Section 3]:

a. outdoor plot sketches should be drawn that include the building site, area streets, outdoor air sampling locations, the location of potential interferences (e.g., gasoline stations, factories, lawn movers, etc.), compass orientation (north), and paved areas;
b. weather conditions (e.g., precipitation and outdoor temperature) should be reported; and
c. any pertinent observations, such as odors, readings from field instrumentation, and significant activities in the vicinity (e.g., operation of heavy equipment or dry cleaners) should be recorded.

### 2.7.5 Tracer gas

When collecting soil vapor samples as part of a vapor intrusion evaluation, a tracer gas serves as a quality assurance/quality control measure to verify the integrity of the soil vapor probe seal. Without the use of a tracer, there is no way to verify that a soil vapor sample has not been diluted by outdoor air.

Depending on the nature of the contaminants of concern, a number of different compounds can be used as a tracer. Typically, sulfur hexafluoride ($SF_6$) or helium are used as tracers because they are readily available, have low toxicity, and can be monitored with portable measurement devices. Butane and propane (or other gases) could also be used as a tracer in some situations. Compounds other than those mentioned here may be appropriate, provided they meet project-specific data quality objectives. Where applicable, steps should

be taken to ensure that the gas used by the laboratory to clean the air sampling container is different from the gas used as a tracer during sampling (e.g., helium).

The protocol for using a tracer gas is straightforward: simply enrich the atmosphere in the immediate vicinity of the area where the probe intersects the ground surface with the tracer gas, and measure a vapor sample from the probe for the presence of high concentrations (> 10%) of the tracer. A cardboard box, a plastic pail, or even a garbage bag can serve to keep the tracer gas in contact with the probe during the testing. If there are concerns about infiltration of ambient air through other parts of the sampling train (such as around the fittings, not just at the probe/ground interface), then consideration should be given to ensuring that the tracer gas is in contact with the entire sampling apparatus. In these cases, field personnel may prefer to use a liquid tracer — soaking paper towels with a liquid tracer and placing the towels around the probe/ground interface, around fittings, and/or in the corner of a shroud.

There are two basic approaches to testing for the tracer gas:

1. include the tracer gas in the list of target analytes reported by the laboratory; or
2. use a portable monitoring device to analyze a sample of soil vapor for the tracer prior to and after sampling for the compounds of concern. (Note that the tracer gas samples can be collected via syringe, Tedlar® bag etc. They need not be collected in Summa® canisters or minicans.)

The advantage of the second approach is that the real time tracer sampling results can be used to confirm the integrity of the probe seals prior to formal sample collection.

Figure 2.4 depicts common methods for using tracer gas. In examples a, b and c, the tracer gas is released in the enclosure prior to initially purging the sample point. Care should be taken to avoid excessive purging prior to sample collection. Care should also be taken to prevent pressure build-up in the enclosure during introduction of the tracer gas. Inspection of the installed sample probe, specifically noting the integrity of the surface seal and the porosity of the soil in which the probe is installed, will help to determine the tracer gas setup. Figure 2.4a may be most effective at preventing tracer gas infiltration, however, it may not be appropriate in some situations depending on site-specific conditions. Figures 2.4b and 2.4c may be sufficient for probes installed in tight soils with well-constructed surface seals. Figure 2d provides an example of using a liquid tracer. In all cases, the same tracer gas application should be used for all probes at any given site.

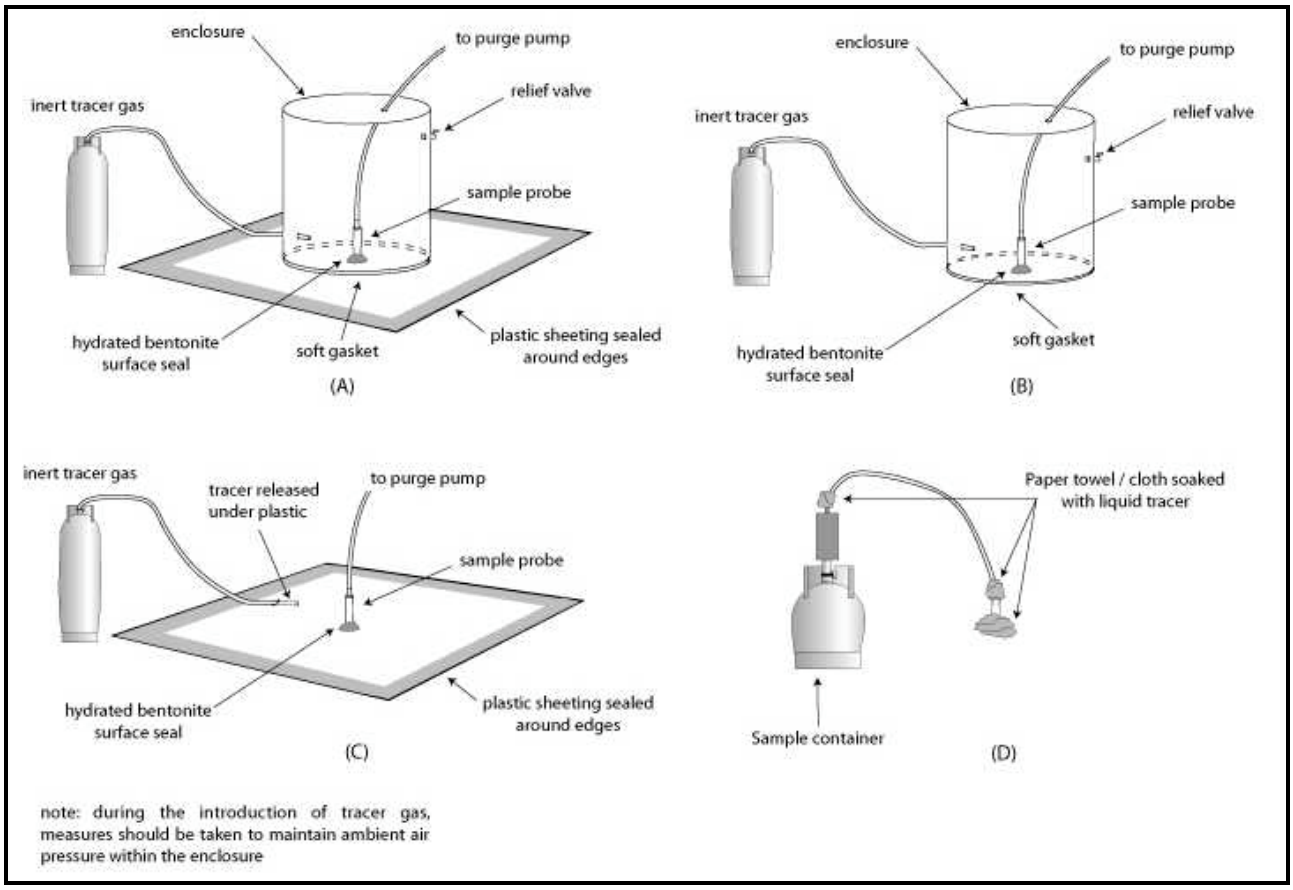


**Figure 2.4**
Schematics of generic tracer gas applications when collecting soil vapor samples

Because minor leakage around the probe seal should not materially affect the usability of the soil vapor sampling results, the mere presence of the tracer gas in the sample should not be a cause for alarm. Consequently, portable field monitoring devices with detection limits in the low ppm range are more than adequate for screening samples for the tracer. If high concentrations (> 10%) of tracer gas are observed in a sample, the probe seal should be enhanced to reduce the infiltration of outdoor air.

Where permanent or semi-permanent sampling probes are used, tracer gas samples should be collected at each of the sampling probes during the initial stages of a soil vapor sampling program. If the results of the initial samples indicate that the probe seals are adequate, reducing the number of locations at which tracer gas samples are employed may be considered. At a minimum, tracer gas samples should be collected with at least 10% of the soil vapor samples collected in subsequent sampling rounds. When using permanent soil vapor probes as part of a long-term monitoring program, annual testing of the probe integrity is recommended. Where temporary probes are used, tracer gas should be used at every sampling location, every time.

## 2.8 Quality assurance / quality control (QA / QC)

[Reference: NYSDOH's *Indoor Air Sampling & Analysis Guidance (February 1, 2005)*]

In general, appropriate QA/QC procedures should be followed during all aspects of sample collection and analysis to ensure that sampling error is minimized and high quality data are obtained. Sampling team members should avoid actions (e.g., fueling vehicles, using permanent marking pens, wearing freshly dry-cleaned clothing or personal fragrances, etc.) which can cause sample interference in the field. Portable air monitoring equipment or field instrumentation should be properly maintained, calibrated and tested to ensure validity of measurements. Air sampling equipment should be stored, transported and between samples decontaminated in a manner consistent with the best environmental consulting practices to minimize problems such as field contamination and cross-contamination. Samples should be collected using certified clean sample devices. Where applicable, steps should be taken to ensure that the gas used by the laboratory to clean the sample device is different from the gas used as a tracer during sampling (e.g., helium). Samples should meet sample holding times and temperatures, and should be delivered to the analytical laboratory as soon as possible after collection. In addition, laboratory accession procedures should be followed, including field documentation (sample collection information and locations), chain of custody, field blanks, field sample duplicates and laboratory duplicates, as appropriate.

Some methods call for collecting samples in duplicate (e.g., indoor air sampling using passive sampling devices for tetrachloroethene) to assess errors. Duplicate and/or split samples should be collected in accordance with the sampling and analytical methods being implemented.

For certain regulatory programs, a Data Usability Summary Report (DUSR) or equivalent report may be required to determine whether or not the data, as presented, meets the site or project specific criteria for data quality and data use. This requirement may dictate the level of QC and the category of data deliverable to request from the laboratory. Guidance on preparing these reports is available by contacting the NYSDEC's Division of Environmental Remediation.

New York State Public Health Law requires laboratories analyzing environmental samples collected from within New York State to have current Environmental Laboratory Approval Program (ELAP) certification for the appropriate analyte and environmental matrix combinations. If ELAP certification is not currently required for an analyte (e.g., trichloroethene), the analysis should be performed by a laboratory that has ELAP certification for similar compounds in air and uses analytical methods with minimum reporting limits similar to background (e.g., tetrachloroethene via EPA Method TO-15). Questions about a laboratory's current certification status should be directed to an ELAP representative at 518-485-5570 or by email at elap@health.state.ny.us.

The work plan should state that all samples that will be used to make decisions on appropriate actions to address exposures and environmental contamination will be analyzed by an ELAP-certified laboratory. The name of the laboratory should also be provided. Similarly, the name of the laboratory that was used should be included in the report of the sampling results. For samples collected and tested in the field for screening purposes by using field testing technology, the qualifications of the field technician should be documented in the work plan.

**APPENDIX D**

Soil Vapor Probe Specification Sheet

## Stainless Steel Vapor Implant

- Universal Barbed Fitting Accepts Tubing ID Sizes: .17-in, .25-in and .50-in
- Swagelock Fitting Available Accepts .25-in OD Tubing
- Solid End Allows for Anchor Point and Open-Hole Placement
- Constructed of Double Woven Stainless Steel Wire Screen
- All End Fittings are Stainless Steel
- Custom Lengths Available

***Applications:***

•Permanent Soil Gas Monitoring
•UST Monitoring
•Groundwater Sampling
•Air Sparging
•Pressure Measurement Points in Vacuum Testing
•Vapor Extraction Monitoring

***Specifications:***

Implant Length:
3-inches (76mm)
6-inches (152mm)
12-inches (305mm)
21-inches (533mm)
Implant OD:
0.50-inch (12.7mm)
Screen Pore Size:
0.0057-inches (.15mm)
Max Depth:
100+ feet (30m)

**APPENDIX E**

Spectrum Analytical Inc. Quality Assurance Plan

*SPECTRUM ANALYTICAL, INC.*
*Featuring*
*HANIBAL TECHNOLOGY*
*Rhode Island Division*

# QUALITY ASSURANCE PLAN
# 2012

Approved By:

Digitally signed by Hanibal C. Tayeh
Date: 2012.10.09 14:40:39 -04'00'

Hanibal C. Tayeh, Ph. D.
President, and CEO — 10/09/2012 Date

Yihai Ding
Laboratory Director — 10/09/2012 Date

Sharyn B. Lawler
Quality Assurance Director — 10/09/2012 Date

EFFECTIVE DATE: 10/26/2012

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# 3.0 INTRODUCTION

Spectrum Analytical, Inc. Featuring Hanibal Technology Rhode Island Division (formerly MITKEM and referenced as Spectrum Analytical, Inc. RI Division throughout this document going forward) is an environmental testing laboratory dedicated to providing high quality analytical data and exceptional customer service.

Opened in 1994, as Mitkem Corporation, and purchased by Spectrum Analytical, Inc. in 2007, Spectrum Analytical, Inc. RI Division's laboratory facility is designed for high throughput and efficient laboratory operations. Separate secure areas are dedicated to sample receipt and storage. Spectrum Analytical, Inc. RI Division has individual sample preparation laboratories for organic and inorganic analyses and individual instrumentation rooms for extractable organics, volatiles, metals and wet-chemistry analyses.

Spectrum Analytical, Inc. RI Division recognizes the importance of controlling in-house cross contamination. The organic preparation area and the volatile organic instrument room are in separate areas, at opposite ends of the building to minimize solvent contamination of the volatile analysis. The air handling system in the volatiles laboratory is completely isolated from the remainder of the facility. A floor plan of the facility is included (Figure 3-1).

Spectrum Analytical, Inc. RI Division has placed a priority on obtaining and operating a large fleet of the latest, most sophisticated Hewlett-Packard, Thermo Scientific and Perkin-Elmer instruments. This emphasis on instrumentation enables the lab to operate at a high level of analytical efficiency.

Spectrum Analytical, Inc. RI Division specializes in performing laboratory analyses using the newest US EPA Contract Laboratory Program (CLP) *SOM* Organic and *ISM* Inorganic methods, as well as providing CLP-format data reports for virtually any test we perform. Spectrum Analytical, Inc. RI Division provides CLP-format reporting for EPA CLP, SW-846, MCAWW and Standard Methods analyses. Much of this work is performed by the laboratory under Department of Defense Quality Systems Manual (QSM) and ISO-17025 guidelines. Spectrum Analytical, Inc. RI Division has the flexibility to provide project-specific custom method modifications to meet the needs of a unique client or analytical requirement.

Spectrum Analytical, Inc. RI Division has participated in numerous environmental laboratory programs for both state and federal agencies including: the United States Navy, the United States Army Corps of Engineers, and the Air Force Center for Environmental Excellence. In addition Spectrum Analytical, Inc. RI Division is currently providing laboratory services under the United States Environmental Protection Agency Contract Laboratory Program. Spectrum Analytical, Inc. RI Division has been a contractor to the EPA under the CLP program continuously for over 15 years.

Spectrum Analytical, Inc. RI Division is a division of Spectrum Analytical, Inc. of Agawam, Massachusetts. Spectrum Analytical, Inc is an environmental laboratory company with laboratory locations in Agawam, MA, North Kingstown, Rhode Island and Tampa, Florida, providing analyses of soil, water and air samples for a wide variety of private and government clients.

This Quality Assurance Plan (QAP) describes the policies, organization, objectives, and quality control activities. It also specifies quality assurance functions employed at Spectrum Analytical, Inc. RI Division and demonstrates our dedication to the production of accurate, consistent data of known quality. This QAP is developed by following the guidelines discussed in the EPA Requirements for Quality Assurance Project Plans for Environmental Data Operations, EPA QA/R-5, Reissued May 2006: EPA Requirements for Quality Management Plans, EPA QA/R-2, Reissued May 2006: Department of Defense (DOD QSM) Quality Systems Manual for Environmental Laboratories Version 4.2: and the National Environmental Laboratory Accreditation Conference (NELAC) standards, June 5, 2003 (Effective July 1, 2003)/ The NELAC Institute (TNI) Standards.

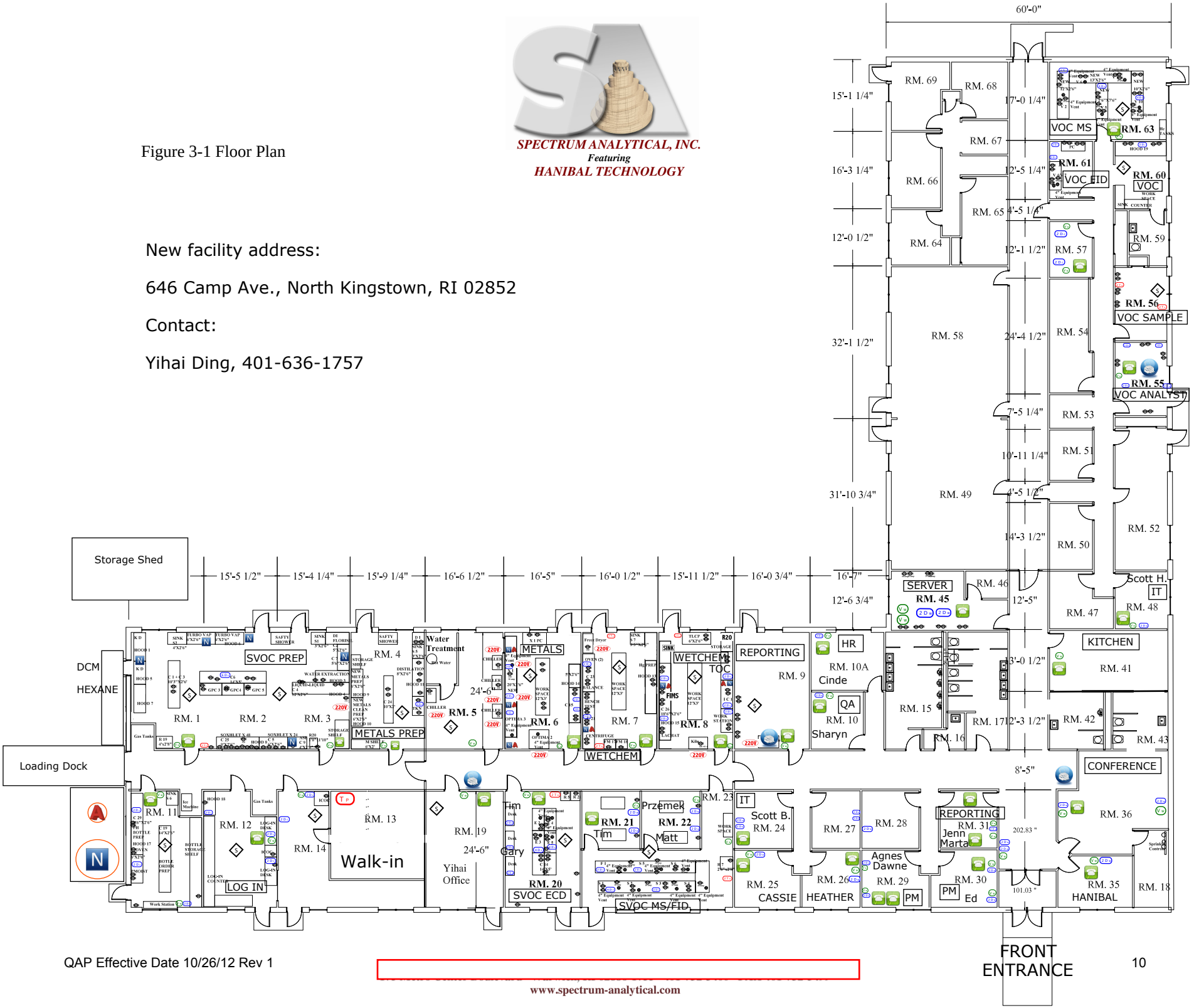
Figure 3-1 Floor Plan

New facility address:

646 Camp Ave., North Kingstown, RI 02852

Contact:

Yihai Ding, 401-636-1757



# 4.0 QUALITY ASSURANCE POLICY STATEMENT

Spectrum Analytical, Inc. RI Division is firmly committed to the production of valid data of known quality through the use of analytical measurements that are accurate, reproducible and complete. To ensure the production of such data, Spectrum Analytical, Inc. RI Division has developed a comprehensive Quality Assurance/Quality Control Program that operates throughout the entire organization.

Spectrum Analytical, Inc. RI Division Management considers Quality Assurance/Quality Control to be of the highest importance in the success of its Analytical Testing Laboratory and therefore fully supports the staff in the implementation and maintenance of a sound and thorough Quality Assurance Program.

Spectrum Analytical, Inc. RI Division's corporate success is based on its participation in the most rigorous and quality-focused environmental testing programs, such as the EPA Contract Laboratory Program, US Department of Defense programs, NELAC, and other nationwide and state-specific certification and approval programs. These programs require consistent application of the QA/QC procedures described in this document. Spectrum Analytical, Inc. RI Division's ability to demonstrate and document that analyses were performed in this manner is one of the foundations of its business. The other foundation of its business is to provide superior levels of customer service, above and beyond the norm for laboratories performing at this level of quality.

Spectrum Analytical, Inc. RI Division's approach to customer service is to aggressively meet or exceed customer expectations, particularly in terms of turnaround time for results. While the production of rapid turnaround time data may require lab employees to "go the extra mile" for the customer, without quality, the data are useless. Spectrum Analytical, Inc. RI Division constantly strives to manage its business to rapidly provide data to meet all the requirements of its quality program.

- Spectrum Analytical, Inc. RI Division management works to insure: that employees understand the primary importance of quality in its day to day operations,
- that employees will not be subjected to pressure to sacrifice quality for turnaround, financial or other considerations,
- that employees understand the importance of their ethical responsibilities in terms of data manipulation, falsification or other illegal or improper actions,
- that the company avoids involvement in activities that diminish its competence, impartiality, judgment or operational integrity.
- that employees maintain all client information in a confidential manner, and
- that employees understand that any short-term gain realized by disregarding the QA/QC program will be more than wasted by the serious penalties for these actions.
- That the laboratory has the technical personnel to identify occurrences of departure from the quality system and to initiate actions to prevent or minimize such departures.

All employees receive training in these issues as part of the initial orientation process, and are required to acknowledge that they understand their responsibilities in these areas. These issues are also discussed among all laboratory staff at company meetings and re-training sessions. The QA Officer, Technical Director and other senior management are readily available to all staff through their daily presence, "open door" policy and approachable manner. This allows any employee to readily discuss any questions, concerns or issues that may occur.

Quality Control is defined as an organized system of activities whose purpose is to demonstrate that quality data are being produced through documentation. Quality Assurance is more broadly defined as a system of activities designed to ensure that the quality control program is actually effective in producing data of the desired quality.

Quality Control is included as part of Quality Assurance. In supporting government regulatory and enforcement proceedings, a high degree of attention to quality is essential. Thorough application of quality control principles and routine quality assurance audits is required.

The basic components of the Spectrum Analytical, Inc. RI Division's QA/QC Program are control, evaluation and correction.

Control ensures the proper functioning of analytical systems through the implementation of an orderly and well-planned series of positive measures taken prior to and during the course of analysis including quality control practices, routine maintenance and calibration of instruments, and frequent validation of standards.

Evaluation involves the assessment of data generated during the control process. For example, precision and accuracy are determined from the results of duplicates and spikes, and other check samples. Long-term evaluation measures include performance and systems audit conducted by regulatory agencies, as well as the lab's quality assurance department.

Correction includes the investigation, diagnosis and resolution of any problems detected in an analytical system. Proper functioning of the system may be restored through method re-evaluation, analysis of additional check samples, trouble-shooting and repair of instrumentation or examination and comparison with historical data. Corrective actions are documented and reviewed to make sure they are implemented.

Certain situations may occur when there are occasional departures or exceptions from documented policies and procedures or standard specifications due to client or project specific protocols, unusual sample matrix, or special non-target analyte or non-routine analyses. Spectrum Analytical, Inc. RI Division's policy is to fully document all such procedures and their associated QC, and notify the client or regulatory agency. If the situation is to continue, a Standard Operating Procedure will be written and implemented.

# 5.0 QUALITY ASSURANCE MANAGEMENT, ORGANIZATION AND RESPONSIBILITY

Quality Assurance at Spectrum Analytical, Inc. RI Division is a company-wide function that depends on:

(1) cooperative working relationships at all levels within the laboratory and

(2) Multi-level review through all working levels of responsibility.

Responsibilities for QA/QC functions begin with the bench scientist and extend to the chief executive officer.

The primary level of quality assurance resides with the bench scientist. After completion of the documented training program, his/her responsibilities include:

- complying with all aspects of formally approved analytical methods and SOPs,
- carefully documenting each step of the analytical process,
- conscientiously obtaining peer review as required,
- promptly alerting laboratory supervisors and/or QA staff members to problems or anomalies that may adversely impact data quality, and
- participation in corrective actions as directed by the laboratory supervisor or QA Director.

The Manager of each laboratory department is responsible for ensuring thorough oversight of the quality of the data generated by the department supervisors, technicians and/or analysts. The Department Manager implements and monitors the specific QC protocols and QA programs with the laboratory to ensure a continuous flow of data meeting all control protocols and Spectrum Analytical, Inc. RI Division QA requirements. The Department Manager's responsibilities include providing the technicians and/or analysts with adequate resources to achieve the desired quality of performance.

The Spectrum Analytical, Inc. RI Division organizational structure is shown in the Organization Chart (Figure 5-1).

Spectrum Analytical, Inc. RI Division's lines of communication flow upward on the Organizational Chart. The open door policy allows all employees' access to anyone on the organization chart. If an employee has an issue with his/her immediate supervisor, he or she may, at any time, speak with someone in management higher up in the Organizational Chart.

Implementation of the entire Quality Assurance Program is the responsibility of the QA Director. While interacting on a daily basis with laboratory staff members, the QA Director remains independent of the laboratories and reports directly to the Laboratory

Director. The QA Director evaluates laboratory compliance with respect to the QA program through informal and formal systems and performance audits as described in Section 13. Remedial action, to alleviate any detected problems, is suggested and/or discussed with the appropriate parties and implemented when necessary.

With input from the appropriate staff members, the QA Director writes, edits and archives QA Plans, QC protocols, and Standard Operating Procedures (SOPs) in accordance with US EPA approved methodologies, and GLP procedures. If site-specific or project-specific QA Plans and/or QC protocols are required, these will be generated as needed.

An essential element of the QA program is record keeping and archiving all information pertaining to quality assurance including QA/QC data, pre-award check sample results, performance test sample results, scores, and follow-up; state certifications of the laboratory; external and internal audits with resolution of EPA and other audit team comments, recommendations and reports. The QA Director also plays an important role in the corrective action mechanism described in Section 16.

In addition, the QA Director works with laboratory staff and management to continuously upgrade procedures and systems to improve the laboratory's efficiency and data quality.

Ultimately, the success of the QA program depends on the cooperation and support of the entire organization. Spectrum Analytical, Inc. RI Division's most valuable resource is its staff of dedicated professionals who take personal pride in the quality of their performance.

Laboratory management works to ensure the competence of all who operate equipment, perform tests and calibrations, evaluate data and sign reports. When employees are in training, appropriate supervision will be provided until the employee has demonstrated the appropriate level of understanding, training, and skill.

Spectrum Analytical, Inc. RI Division's personnel job descriptions:

Responsibilities of each staff area in the laboratory include:

Technician / Preparation Laboratory Areas:

- Analysis of samples through compliance with all aspects of formally approved analytical methods and laboratory SOPs.
- Carefully documenting each step of the analytical process.
- Noting in the appropriate logbook area any unusual occurrences or sample matrix problems.
- Conscientiously obtaining peer review as required.

- Promptly alerting laboratory supervisor, Department Managers and/or QA staff members to problems or anomalies that may adversely impact data quality.
- Routine housekeeping duties for their laboratory area.

Analyst / Instrument Laboratory Areas:

- Analysis of samples through compliance with all aspects of formally approved analytical methods and laboratory SOPs.
- Routine maintenance of instrumentation.
- Preparation of analytical standards and spiking solutions which are documented and traceable to their original source.
- Carefully documenting each step of the analytical process.
- Noting in the appropriate logbook area any unusual occurrences or sample matrix problems.
- Conscientiously obtaining peer and Department Manager review as required.
- Promptly alerting the appropriate Department Manager and/or QA staff members to problems or anomalies that may adversely impact data quality.
- Documenting the initial review of analysis data to determine compliance with established company QA/QC protocols and any project-specific QA criteria, and noting any unusual occurrences or discrepancies on the data review checklist.
- Routine housekeeping duties for their laboratory area.

Data Reporting Specialists:

- Assemble CLP-format data reports by organizing data report forms and raw data in proper order to allow for technical data review.
- Enter data into LIMS or other data reporting computer programs, and print report forms as appropriate.
- Provide non-technical typographical review of data entered into computer systems by other individuals.
- Deliver data reports to customers by FAX or electronic mail.
- Paginate, photocopy, scan, save to CD (bookmark if required) and archive copies of customer reports or other documentation to be retained by the laboratory, or prepare paperless reports.
- Ship, or organize for courier delivery, final data reports to customers.
- Assist the QA Director in management of the document control system.
- Assist Project Managers with bottle order requests and shipment of coolers.
- Assist Project Managers in other tasks as required.

Laboratory Department Manager/Supervisors:

- Oversight of supervisors (where applicable), technicians and/or analysts in their laboratory areas.

- Monitors the status of all work in their laboratory area to insure compliance with holding time and turnaround time requirements.
- Training new scientists in the appropriate procedures and methods in the laboratory.
- Works with Laboratory Director and the QA staff to review, revise and implement SOPs.
- Insures adequate resources to perform the needed tasks by working with administrative personnel to order needed supplies.
- Insures all supplies and reagents meet the QC requirements of their intended task prior to their use in the laboratory.
- Insures all staff are using proper safety protocols.
- Works with Laboratory Director on the annual review of personnel performance.
- Interviews prospective new employees to insure they have the minimal level of qualifications, experience, education and skills necessary to perform their tasks, as well as the appropriate work ethic and social skills necessary for proper teamwork and productivity.
- Review of analytical data to insure compliance with method/SOP requirements prior to release to the client.
- Documents any non-compliance or other unusual occurrences noted during sample analysis and data review such that these can be included in the report narrative and explained to the client.

Data Reviewer:

- Review of analytical data to insure compliance with method/SOP requirements prior to release to the client.
- Generates paperless CLP and CLP-like data packages.
- Documents any non-compliance or other unusual occurrences noted during sample analysis and data review such that these can be included in the report narrative and explained to the client.
- Compiles narrative.
- Assist Laboratory Director, Supervisors and Department Managers in other tasks as required.

Laboratory Director:

- Works with Department Managers to coordinate laboratory areas in the completion of analytical projects.
- Review of analytical data to insure compliance with method/SOP requirements prior to release to the client.
- Works with QA Director to implement new SOPs and to annually review and revise existing SOPs.
- Works with the QA Director, Department Managers and Supervisors to develop and implement corrective action when needed.

- Works with management and supervisory staff to continuously improve the quality and efficiency of all company procedures.
- Assists Department Managers in the annual review of personnel performance.
- Supervises all Management, QA and Supervisory staff to insure compliance with company QA policies and other company procedures.
- Provides technical assistance to all areas of the laboratory staff.
- Acts as technical consultant for chemistry related issues that arise in the lab.
- Provides assistance with instrument optimization or performance issues as needed.
- Offers input on the purchase and operation of new instrumentation.
- Trains other analysts in procedures and methodologies.

Director of Business Development

- Pursues new contracts/projects as well as clients.
- Works with Spectrum Marketing to prepare Bids.
- Ensures laboratory is aware of specific requirements of new projects/contracts.
- Works with clients to insure all questions and concerns are addressed and answered.
- Works with clients to insure their understanding of complex technical issues.
- Works with Quality Services Department staff to continuously improve the quality and efficiency of all company procedures.

Data Reporting Supervisor:

- Works with Laboratory Director, Department Managers and Supervisors to prioritize and coordinate laboratory areas in the timely completion of analytical projects.
- Review of analytical data to insure compliance with method/SOP requirements prior to release to the client.
- Writes project report narratives to document any unusual occurrences noted during sample analysis.
- Works with management and supervisory staff to continuously improve the quality and efficiency of all company procedures.
- Works with Laboratory Director on the annual review of personnel performance.

Project Manager:

- Works with the client to completely understand the requirements of all incoming work.
- Evaluates the client’s requirements as compared to the abilities of the laboratory as stated in Standard Operating Procedure (SOP) #110.0023 Project Management.
- Works with the Data Reporting staff to continuously improve the quality and efficiency of all company procedures.

- Communicates the customer's requirements to all laboratory staff working on the project.
- Works with the customer to determine the number and type of sample containers required for the project.
- Works with the Sample Custodian to resolve and communicate to the client any problem or discrepancies with incoming samples.
- Maintains open, responsive and continuous communication with the customer.
- Follows up with the client to assess level of satisfaction, and insure all project goals have been accomplished.
- Assist Business Development and Marketing Staff in other tasks as required.

Information Technology Director:

- Oversees the operations of the three divisions of Spectrum Analytical, Inc. (MA, FL and RI). The IT Director's role is technical guidance, IT long term planning, coordination/communication between the divisions, oversees and makes the necessary decision to support the overall IT function.

Information Technology Manager:

Primary function is to oversee the operations of the Spectrum Analytical, Inc. RI Division's IT department.

- Oversee the operations of the network, including servers and workstations.
- Plan for hardware and software updates
  1) Support users IT needs.
  2) Support client IT needs.
  3) Oversee security of network
- Development and expansion of LIMS.
  1) Program new functionality into LIMS including program based protocols requirements
  2) hard copy reports
  3) electronic reports
  4) processing of data to web site
  5) tracking of data
  6) maintenance of LIMS
  7) security of LIMS
- Generate and troubleshoot more complex EDDs
- Provide backup for the Information Technology Specialist when out and support when it is needed.

Secondary function is to work with the other divisions to try and make transfer of information as seamless as possible. Lend technical support to other divisions and help to bring technical help from other divisions to Spectrum Analytical, Inc. RI Division IT department.

Information Technology Specialist:

- Primary duty is to generate and review EDDs using EDD module.
  a) Generate and validate EDDs using EDD specific tools (CRA, Tetra Tech, CH2M Hill, etc…).
  b) Generate all SEDD files for the EPA SOM contract, and work with the chemists to resolve any defects, if possible.
- Perform server room duties.
  a) Monitor the servers and troubleshoot (if needed)
  b) Perform backup/archive of data on servers
  c) File grooming at the end of the month
  d) Monitor event logs of the servers for issues.
  e) Monitor status of centralized anti-viral program (AVG). Includes monitoring AVG status of workstations
  f) Monitor centralized Windows System Update Server (WSUS). Includes monitoring WSUS status of workstations.
- Handles user issues with printer/scanner/copier systems from Ikon. Based on evaluation, schedule service calls or replaces consumable parts.

Quality Assurance Director:

- Implements the entire QA program.
- Interacts on a daily basis with laboratory staff.
- Evaluates compliance with the QA program through formal and informal reviews of data and processes.
- Implements the corrective action system.
- Maintains a master list of all SOPs and monitors review schedules.
- Works with Department Managers and Supervisors to implement new SOPs and to annually review and revise existing SOPs.
- Controls all master and controlled-copies of SOPs and QAP as per SOP #80.0012; Production of Standard Operating Procedures.
- Posts to intranet, and archives all old and edited revisions of SOPs and QA manual as per SOP# 80.0012; Production of Standard Operating Procedures.
- Interfaces with certification authorities and agencies to maintain existing certifications and programs, and obtain new certifications.
- Maintains records of employee training and certification as per SOP# 80.0016; Training Procedures and Tracking.
- Instructs laboratory personnel on ethics in the workplace.
- Oversees analytical trends that need to be evaluated and corrected.
- Oversees the implementation of MDLs and control limit studies.
- Directs the internal audit program as per SOP# 80.0006; Internal Audits.
- Coordinates all external audit corrective action reports and investigations.
- Maintains certification of NIST thermometers and weights.

- Schedules annual hood inspections and balance calibrations.

In Spectrum Analytical, Inc. RI Division's organizational structure, the Laboratory Director has the ultimate authority for all chemistry-related aspects of the company.

The QA Director reports directly to the Laboratory Director. She has the authority within the management system to bring any issue to the highest levels of the company management and ownership, as well as to halt the release of data she believes to be questionable or suspend the performance of an analysis she believes to be unreliable.

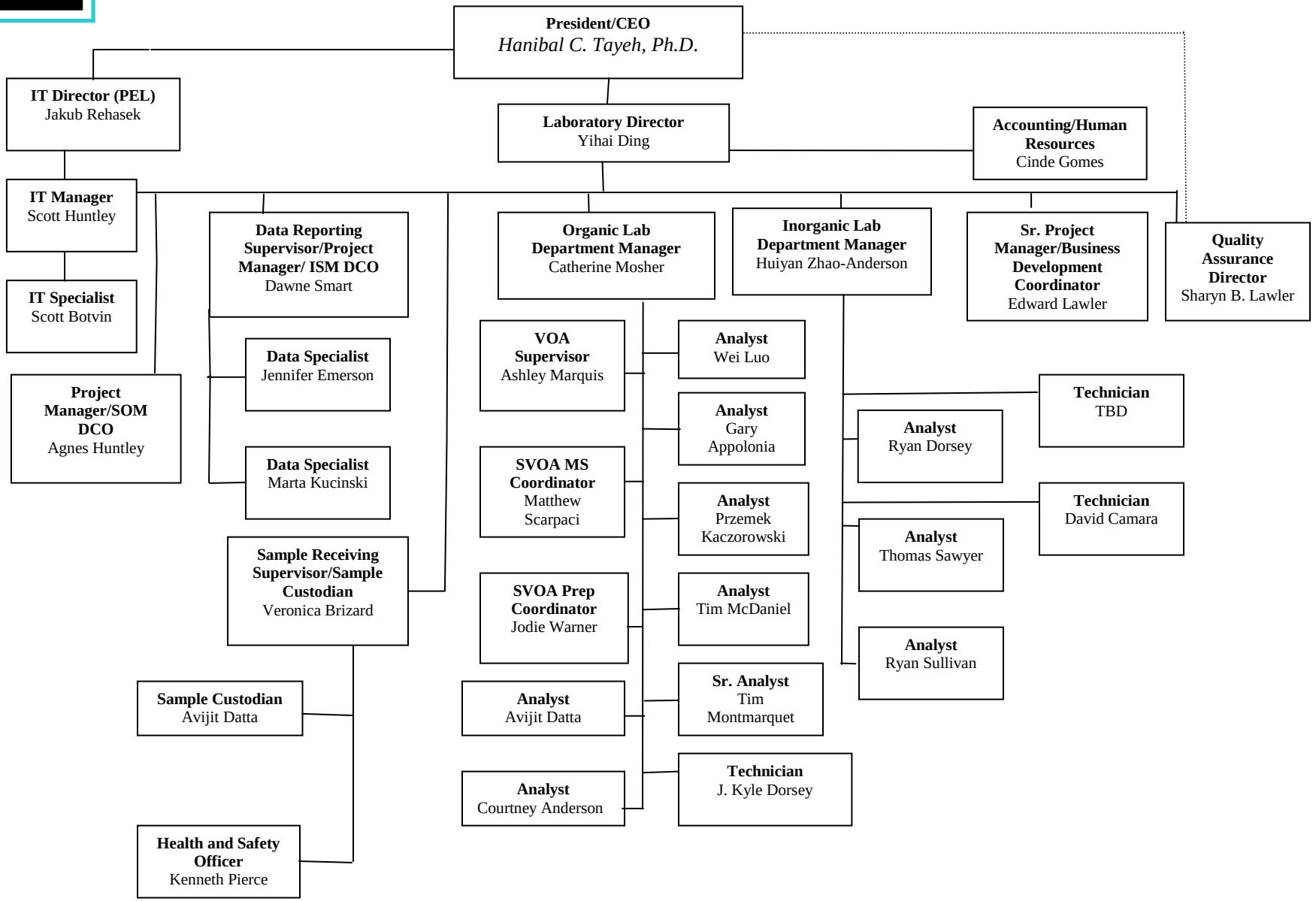
The Director of Business Development works with the project managers and marketing staff and with the Department Managers and Supervisors to prioritize and coordinate work within the laboratories.

The personnel training records are located in the QA department on-site as well as additional training documents being saved in pdf form on the Spectrum network. All individual training is documented including new employee training, individual training, annual retraining procedures, and Health and Safety training.

Figure 5-1
SPECTRUM ANALYTICAL, INC. RI Division's Organizational Chart

**Spectrum**
**Analytical RI**

# Organizational Chart



# 6.0 QUALITY ASSURANCE OBJECTIVES FOR MEASUREMENT DATA IN TERMS OF PRECISION, ACCURACY, REPRESENTATION, COMPLETENESS AND COMPARABILITY AND QA REPORTING

As part of the evaluation component of the overall QA Program, laboratory results are compared with the data quality indicators defined as follows:

- Precision: the agreement of reproducibility among individual measurements of the same property usually made under identical conditions.

- Accuracy: the degree of agreement of a measurement with the true or accepted value.

- Representation: the degree to which data accurately and precisely represent a characteristic of a population, parameter variations of a sample of a finite process condition, or of a finite environmental condition.

- Completeness: a measure of the amount of valid data obtained from a measurement system compared with the amount that was expected to be obtained under normal conditions.

- Comparability: an expression of the confidence with which one laboratory data set can be compared with another laboratory data set in regard to the same property and laboratory sample population.

Quality Assurance objectives may vary by project and requested parameters. The accuracy, precision, and representation of data will be functions of the origins of the sample material, the procedures used to analyze sample and generate data, and the specific sample matrices involved in each project. Quality control practices utilized in the evaluation of these data quality indicators include blanks, replicates, spikes, standards, check samples, calibrations and surrogates. The process for quantifying or assessing the above indicators for data quality is addressed in Section 15.

## 6.1 Precision and Accuracy:

For each parameter analyzed, the QA objectives for precision and accuracy will be determined from:

- Published historical data;
- Method validation studies;
- Spectrum Analytical, Inc. RI Division’s experience with similar samples and/or;
- Project-specific requirements, such as those stipulated by the USEPA in the CLP protocols and control documents.

6.2 Representation:

Analytical data should represent the sample analyzed regardless of the heterogeneity of the original sample matrix. In most cases, representation is achieved by mixing the laboratory sample well before removing a portion for analysis. On occasion, multi-phase laboratory samples may require that each phase be analyzed individually and reported in relation to its proportion in the whole sample.

6.3 Completeness:

The completeness goal is 100% in all cases and includes:

- Analysis of all samples;
- Generation and analysis of all required QC samples;
- Sufficient documentation of associated calibration, tuning and standardization;
- Records of data reduction processes, including manual calculations.

While the laboratory staff is responsible for achieving the completeness objective stated above, assigning each project a specific project manager whose functions include sample management and tracking ensures completeness.

6.4 Comparability:

To assure comparability, Spectrum Analytical, Inc. RI Division employs established and approved analytical methods (e.g. USEPA protocols), consistent analytical bases (dry weight, volume, etc.) and consistent reporting units (mg/Kg, µg/L, etc.). Where data from different samples must be comparable, the same sample preparation and analysis protocols are used for all of the samples of interest.

6.5 QA Reporting

General QA procedures require that an MS/MSD or DUPLICATE/MS be reported with each sample batch up to 20 samples. In addition, each batch requires a method blank (MB) and laboratory control sample (LCS).

An acceptance criterion for the MB depends upon the method criteria. In-house control limits dictate the acceptability of the LCS in many methods. Several methods have set LCS control limits. A high bias LCS is considered acceptable if the analyte is not present in the samples above the reporting limit. A low bias LCS will require re-extraction (if sample volume allows) and re-analysis.

DUP, MS, and MSD recoveries and calculated RPDs are specified in the analytical methods. Recoveries outside the limits require some form of corrective action, whether that includes a post-digestion/distillation spike, re-extraction, re-analysis and/or notification to the client in the project narrative.

LIMS will flag any QA samples outside method criteria on the reporting forms. Formal written corrective action reports are required for any incident that does not meet method criteria and cannot be remedied or explained by the laboratory. The QA Officer signs off on any corrective actions and can also track QA trends in this manner.

# 7.0 SAMPLING PROCEDURES

For most projects, outside sampling teams deliver or send samples to Spectrum Analytical, Inc. RI Division's. When sampling by Spectrum Analytical, Inc. RI Division's personnel is required, the sampling team follows the sampling procedures outlined in the EPA *Test Methods for Evaluating Solid Wastes*, SW-846, 3rd Edition, or procedures found in the EPA "Handbook for Sampling and Sample Preservation of Water and Wastewater".

Appropriately prepared sample containers are supplied by Spectrum Analytical Inc., RI Division at clients' request. When required, preservatives are added to the sample containers. Tables 7-1 through 7-3 provide the Spectrum Analytical, Inc. RI Division Recommended Container, Preservation Techniques and Holding Times. Additional sample volumes may be required if additional QC functions are to be performed.

Holding times for SW846, CLP Methods, Standard Methods and certain USEPA methods are different and are presented in Tables 7-1 to 7-3. Holding times for most methods are calculated from the date of sample collection. Holding times for CLP methods are calculated from the Validated Time of Sample Receipt (VTSR). It should be noted that the CLP analysis program combines chemical analyses and contract compliance procedures in one document. For laboratory analysis and contract compliance purposes, holding times are calculated from VTSR, while post-analysis data usability and validation (generally performed by the client or a third party) compares holding times to the SW-846 method holding times calculated from date of sample collection.

Representative portions of samples are taken for analysis by following Spectrum Analytical, Inc. RI Division's SOP 110.0039 Standard Operating Procedure for Sub-Sampling.

# Table 7-1

## Recommended Container, Preservation Techniques and Holding Times For SW-846 Analyses

| Analytes | Method | Containers | Required* Volume | Preservation | Holding Times |
|---|---|---|---|---|---|
| Volatile Organics | | | | | |
| Solid | 8260, 5030 | Amber glass jar with Teflon lining | Minimal head-space in jar | 4°C | 14 days |
| Solid [a] | 8260, 5035 | 40mL vial or Encore with Teflon lining | 5.0gram ± 0.5 | 4°C, unpreserved | 48 hours |
| | | | | DI Water -10 to –20$^0$C | 14 days |
| | | | | Sodium bisulfate -10 to –20$^0$C, 4$^0$C | 14 days |
| | | | | Methanol 4$^0$C | 14 days |
| Aqueous | 8260, 5030 | 40mL VOA Vials with Teflon septum | 40mL | 4°C HCl, pH<2 | 14 days |
| Semivolatile Organics | | | | | |
| Solid | 3540, 3550 8270 | Amber glass jar with Teflon lining | 30gram | 4°C | Extraction within 14 days Analysis within 40 days |
| Aqueous | 3510, 3520 8270 | Amber glass bottles with Teflon lining | 1L | 4°C | Extraction within 7 days Analysis within 40 days |
| Polychlorinated Biphenyls | | | | | |
| Solid | 3540, 3550 8082 | Amber glass jar with Teflon lining | 30gram | 4°C | Extraction within 14 days Analysis within 40 days |
| Aqueous | 3510, 3520 8082 | Amber glass bottle with Teflon lining | 1L | 4°C | Extraction within 7 days Analysis within 40 days |
| Organochlorine Pesticides | | | | | |
| Solid | 3540, 3550 8081 | Amber glass jar with Teflon lining | 30gram | 4°C | Extraction within 14 days Analysis within 40 days |
| Aqueous | 3510, 3520 8081 | Amber glass bottle with Teflon lining | 1L | 4°C | Extraction within 7 days Analysis within 40 days |
| Chlorinated Herbicides | | | | | |
| Solid | 8151 | Amber glass jar with Teflon lining | 30gram | 4°C | Extraction within 14 days Analysis within 40 days |
| Aqueous | 8151 | Amber glass bottle with Teflon lining | 1L | 4°C | Extraction within 7 days Analysis within 40 days |

Table 7-1 (cont'd)

Recommended Containers, Preservation Techniques and Holding Times
For
SW846 Analyses

| Analytes | Method | Containers | Required* Volume | Preservation | Holding Times |
|---|---|---|---|---|---|
| Total Petroleum Hydrocarbons | | | | | |
| Gasoline Range Organics, including Maine-GRO** | | | | | |
| Solid | 8015, 5030<br>ME 4.1.17 | Amber glass jar<br>With Teflon lining | Minimal head-space in jar | 4°C | 14 days |
| Solid [a] | 8015, 5035 | 40mL vial or Encore with Teflon lining | 5.0gram ± 0.5 | 4°C, unpreserved | 48 hours |
| | | | | 4°C, Methanol | 14days |
| Aqueous | 8015, 5030<br>ME 4.1.17 | 40mL VOA vials<br>With Teflon septum | 40mL | 4°C<br>HCl, pH<2 | 14 days |
| Diesel Range Organics, including Maine-DRO | | | | | |
| Solid | 3540, 3550<br>8015<br>ME 4.1.25 | Amber glass jar<br>with Teflon lining | 30gram | 4°C | Extraction within 14 days<br>Analysis within 40 days |
| Aqueous | 3510, 3520<br>8015<br>ME 4.1.25 | Amber glass bottle<br>with Teflon lining | 1L | 4°C<br>$H_2SO_4$, pH<2 | Extraction within 7 days<br>Analysis within 40 days |
| Total Metals except Mercury and Chromium (VI) | | | | | |
| Solid | 3050<br>6010 | Amber glass jar<br>with Teflon lining | 10g | 4°C | 180 days |
| Aqueous | 3005, 3010 | Polyethylene bottle | 100mL | $HNO_3$, pH<2 | 180 days |
| Chromium (VI) | | | | | |
| Solid | 3060, 7196 | Amber glass jar<br>with Teflon lining | 10g | 4°C | Digestion within 30 days<br>Analysis within 96 hours |
| Aqueous | 7196 | Polyethylene bottle | 25mL | 4°C | 24 hours |
| Mercury | | | | | |
| Solid | 7471 | Amber glass jar | 10g | 4°C | 28 days |
| Aqueous | 7470 | Polyethylene bottle | 100mL | 4°C<br>$HNO_3$, pH<2 | 28 days |
| Cyanide | | | | | |
| Solid | 9012 | Amber glass jar<br>with Teflon lining | 10g | 4°C | 14 days |
| Aqueous | 9012 | Polyethylene bottle | 50mL | 4°C<br>NaOH, pH≥12 | 14 days |
| Flashpoint | | | | | |
| Aqueous | 1010 | Amber glass bottle | 30mL | 4°C | 28 days |

Table 7-1 (cont'd)

Recommended Containers, Preservation Techniques and Holding Times
For
SW846 Analyses

| Analytes | Method | Containers | Required* Volume | Preservation | Holding Times |
|---|---|---|---|---|---|
| Chloride | | | | | |
| Aqueous | 9056 | Polyethylene bottle | 50mL | 4°C | 28 days |
| Nitrate | | | | | |
| Aqueous | 9056 | Polyethylene bottle | 50mL | 4°C | 48 hours |
| Nitrite | | | | | |
| Aqueous | 9056 | Polyethylene bottle | 50mL | 4°C | 48 hours |
| Orthophosphate | | | | | |
| Aqueous | 9056 | Polyethylene bottle | 50mL | 4°C | 48 hours |
| Sulfates | | | | | |
| Aqueous | 9056 | Polyethylene bottle | 50mL | 4°C | 28 days |

# Table 7-2

## Recommended Container, Preservation Techniques and Holding Times For CLP/ASP Analyses

| Analytes | Method | Containers | Required* Volume | Preservation | Holding Times |
|---|---|---|---|---|---|
| Volatile Organics | | | | | |
| Solid | CLP/ASP | Amber glass jar with Teflon lining | Minimal head-space in jar | 4°C | 10 days from VTSR |
| Aqueous | CLP/ASP | 40mL VOA vials with Teflon septum | 40mL | 4°C HCl, pH<2 | 10 days from VTSR |
| | CLP Low | 40mL VOA vials with Teflon septum | 40mL | 4°C HCl, pH<2 | 10 days from VTSR |
| Semivolatile Organics | | | | | |
| Solid | CLP/ASP | Amber glass jar with Teflon lining | 30gram | 4°C | 10 days from VTSR Analysis within 40 days |
| Aqueous | CLP/ASP | Amber glass bottle with Teflon lining | 1L | 4°C | 5 days from VTSR Analysis within 40 days |
| | CLP Low | Amber glass bottle with Teflon lining | 1L | 4°C | 5 days from VTSR Analysis within 40 days |
| Organochlorine Pesticide/PCB | | | | | |
| Solid | CLP/ASP | Amber glass jar with Teflon lining | 30gram | 4°C | 10 days from VTSR Analysis with 40 days |
| Aqueous | CLP/ASP | Amber glass bottle with Teflon lining | 1L | 4°C | 5 days from VTSR Analysis within 40 days |
| | CLP Low | Amber glass bottle with Teflon lining | 1L | 4°C | 5 days from VTSR Analysis within 40 days |
| Cyanide | | | | | |
| Solid | CLP/ASP | Amber glass jar | 10gram | 4°C | 12 days from VTSR |
| Aqueous | CLP/ASP | Polyethylene bottle | 50mL | 4°C NaOH, pH>12 | 12 days from VTSR |
| Total Metals except Mercury | | | | | |
| Solid | CLP/ASP | Amber glass jar | 10gram | 4°C | 180 days from VTSR |
| Aqueous | CLP/ASP | Polyethylene bottle | 100mL $HNO_3$, pH<2 | 4°C | 180 days from VTSR |

Table 7-2 (cont'd)

Recommended Container, Preservation Techniques and Holding Times
For
CLP/ASP Analyses

| Analytes | Method | Containers | Required* Volume | Preservation | Holding Times |
|---|---|---|---|---|---|
| Mercury | | | | | |
| Solid | CLP/ASP | Amber glass jar | 10gram | 4°C | 26 days from VTSR |
| Aqueous | CLP/ASP | Polyethylene bottle | 100mL | 4°C $HNO_3$, pH<2 | 26 days from VTSR |

# Table 7-3

## Recommended Containers, Preservation Techniques and Holding Times for Other Analyses

| Analytes | Method | Containers | Required* Volume | Preservation | Holding Times |
|---|---|---|---|---|---|
| Volatile Organics | | | | | |
| Aqueous | 624 | 40mL VOA vials with Teflon septum | 40mL | 4°C HCl, pH<2 | 14 days |
| Semivolatile Organics | | | | | |
| Aqueous | 3510, 3520 625 | Amber glass bottle with Teflon lining | 1L | 4°C | Extraction within 7 days Analysis within 40 days |
| Organochlorine Pesticide/PCB | | | | | |
| Aqueous | 3510, 3520 608 | Amber glass bottle with Teflon lining | 1L | 4°C | Extraction within 7 days Analysis within 40 days |
| EDB/DBCP | | | | | |
| Aqueous | 8011 | 40mL VOA vials with Teflon septum | 35mL | 4°C HCl, pH<2 | 28 days |
| MA Extractable Petroleum Hydrocarbons (EPH) | | | | | |
| Solid | 3540, 3550 MADEP | Amber glass jar with Teflon lining | 10gram | 4°C | Extraction within 7 days Analysis within 40 days |
| Aqueous | 3510, 3520 MADEP | Amber glass bottle with Teflon lining | 1L | 4°C HCl, pH<2 | Extraction within 14 days Analysis within 40 days |
| MA Volatile Petroleum Hydrocarbons (VPH) | | | | | |
| Solid | MADEP | Amber glass jar with Teflon lining | 10gram | 4°C 10mL Methanol | 14 days |
| Aqueous | MADEP | 40mL VOA vial with Teflon lining | 40mL | 4°C HCl, pH<2 | 14 days |
| Total Metals excluding Mercury | | | | | |
| Aqueous | 200.7, 200.8 | Polyethylene bottle | 100mL | $HNO_3$, pH<2 | 180 days |
| Mercury | | | | | |
| Aqueous | 245.1 | Polyethylene bottle | 100mL | $HNO_3$, pH<2 | 28 days |
| Cyanide | | | | | |
| Aqueous | 335.4 | Polyethylene bottle | 50mL | NaOH, pH>12 | 14 days |

Table 7-3 (cont'd)

Recommended Containers, Preservation Techniques and Holding Times
for
Other Analyses

| Analytes | Method | Containers | Required Volume* | Preservation | Holding Times |
|---|---|---|---|---|---|
| Chloride | E300.0 | Polyethylene bottle | 50mL | 4°C | 28 days |
| COD | | | | | |
| Aqueous | SM5220D | Amber VOA vial | 40mL | 4°C $H_2SO_4$, pH<2 | 28 days |
| Color | | | | | |
| Aqueous | SM2120B | Polyethylene bottle | 50mL | 4°C | Immediate |
| Nitrate | | | | | |
| Aqueous | E300.0 | Polyethylene bottle | 50mL | 4°C | 48 hours |
| Nitrite | | | | | |
| Aqueous | E300.0 | Polyethylene bottle | 50mL | 4°C | 48 hours |
| Orthophosphate | | | | | |
| Aqueous | SM4500-P, E E300.0 | Polyethylene bottle | 50mL | 4°C | 48 hours |
| Total phosphate | | | | | |
| Aqueous | SM4500-P B5,E | Polyethylene bottle | 50mL 50mL | 4°C $H_2SO_4$, pH<2 | 28 days |
| Phenols | | | | | |
| Aqueous | SM5530B E420.1 | glass | 250mL | 4°C $H_2SO_4$, pH<2 | 28 days |
| Sulfates | | | | | |
| Aqueous | SM426 15th Ed. SM4500-SO4 E, E300.0 | Polyethylene bottle | 50mL | 4°C | 28 days |
| Sulfide Total | | | | | |
| Aqueous | SM4500-S-D | Polyethylene bottle | 50mL | 4°C NaOH, pH>12 ZnAc | 28 days |
| Reactivity | | | | | |
| Solid | Chapter 7 SW846 | Amber glass jar | 10gram | 4°C | 28 days |
| Aqueous | Chapter 7 | Polyethylene bottle | 250mL | 4°C | 28 days |
| Total Organic Carbon (TOC) | | | | | |
| Solid | Lloyd Kahn Walkley-Black | Amber glass jar | 10g | 4°C | 14 days |

Table 7-3 (cont'd)

Recommended Containers, Preservation Techniques and Holding Times
For
Other Analyses

| Analytes | Method | Containers | Required* Volume | Preservation | Holding Times |
|---|---|---|---|---|---|
| Total Organic Carbon | | | | | |
| Aqueous | SM5310B | 40mL VOA vials | 40mL | 4°C $H_3PO_4$, pH<2 | 28 days |
| TKN | | | | | |
| Aqueous | SM4500Norg C | Polyethylene bottle or Amber glass bottle | 50mL | 4°C $H_2SO_4$, pH<2 | 28 days |
| Total Solids (TS) | | | | | |
| Aqueous | SM2540B | Polyethylene bottle | 200mL | 4°C | 7 days |
| Total Dissolved Solids (TDS) | | | | | |
| Aqueous | SM2540C | Polyethylene bottle | 200mL | 4°C | 7 days |
| Total Suspended Solids (TSS) | | | | | |
| Aqueous | SM2540D | Polyethylene bottle | 200mL | 4°C | 7 days |
| Settleable Solids | | | | | |
| Aqueous | SM2540F | Polyethylene bottle | 200mL | 4°C | 48 hours |
| Chromium (VI) | | | | | |
| Aqueous | SM3500 Cr+ | Polyethylene bottle | 25mL | 4°C | 24 hours |
| Alkalinity | | | | | |
| Aqueous | SM2320B | Polyethylene bottle | 100mL | 4°C | 14 days |
| Ammonia | | | | | |
| Aqueous | SM4500NH3B | Polyethylene bottle | 100mL | 4°C $H_2SO_4$, pH<2 | 28 days |
| Oil & Grease | | | | | |
| Aqueous | 1664 | Amber glass bottle with Teflon lining | 1L | 4°C HCl, pH<2 | 28 days |

* These represent minimum required volume. Additional sample volumes should be collected to minimize headspace loss for volatile analysis. Additional sample aliquots are also required to perform QA/QC functions (e.g. spikes, duplicates), % moisture for solid samples and sample re-analysis (if needed).

[a] For Massachusetts analyses, the Volatile Organics soil samples are preserved in Methanol in the field.

EPA SW-846 Method 5035 provides several options for preservation of soil samples for volatile organics. Certain projects have not adopted these options to-date, and continue to recommend the collection of unpreserved soil sample aliquots for volatiles analysis. Spectrum Analytical Inc., RI Division's preference for low-level analysis is to collect approximately 5 grams of soil into 5mL of organic-free DI water and to preserve by freezing within 48hours of collection. A separate container with approximately 5 grams of

soil into 5mL of methanol is also collected for potential medium-level analysis. A separate container of unpreserved soil also must be collected to perform percent moisture analysis.

** Maine GRO soil analysis requires a medium level methanol extraction. A 10 gram sample and 10mL methanol volume is used.

# 8.0 SAMPLE CUSTODY

## 8.1 Chain of Custody:

Samples are physical evidence collected from a facility or the environment. In hazardous waste investigations, sample data may be used as evidence in (EPA) enforcement proceedings. In support of potential litigation, laboratory chain-of-custody procedures have been established to ensure sample traceability from time of receipt through the disposal of the sample.

A sample is considered to be in the custody under the following conditions:

- It is in an authorized person's actual possession, or
- It is in an authorized person's view, after being in that person's physical possession, or
- It was in an authorized person's possession and then was locked or sealed to prevent tampering, or
- It is in a secure area.

Chain-of-custody originates as samples are collected. Chain-of-custody documentation accompanies the samples as they are moved from the field to the laboratory with shipping information and appropriate signatures indicating custody changes along the way.

Laboratory chain-of-custody is initiated as samples are received and signed for by the Sample Custodian or his/her designated representative at Spectrum Analytical, Inc. RI Division. Documentation of sample location continues as samples are signed in and out of the designated storage facility for analysis in the several laboratory departments, using the Internal Chain of Custody (IntCOC) barcode system. After analysis, any remaining sample is held in the designated storage area to await disposal. Spectrum Analytical Inc., RI Division's policy is to hold spent samples for a period of at least thirty days from submittal of final report, unless other arrangements are agreed upon with the client. USEPA samples and empty containers are held for 60 days.

## 8.2 Laboratory Security:

Samples and all data generated from the analyses of samples at Spectrum Analytical, Inc. RI Division are kept within secure areas during all stages of residence, including the periods of time spent in preparation for analysis, while undergoing analysis, and while in storage.

The entire laboratory is designated as a secure area. The doors to the laboratory are under continuous surveillance, are kept locked after regular business hours

and may only be accessed by key or keypad entry. Only authorized personnel are allowed to enter the secure areas. The laboratory facility and IT office are only accessed through keypad entry. A Spectrum Analytical, Inc. RI Division staff member must accompany visitors to the laboratory.

8.3 Duties and Responsibilities of Sample Custodian:

Duties and responsibilities of the Sample Custodian include:

8.3.1 Receiving samples.

8.3.2 Inspecting and documenting sample shipping containers for presence/absence and condition of:

8.3.2.1 Custody seals, locks, “evidence tape”, etc.;

8.3.2.2 Container breakage and/or container integrity, including air space in aqueous samples, or proper preservation for soil samples for Volatiles analysis.

8.3.3 Recording condition of both shipping containers and sample containers (cooler temperature, bottles, jars, cans, etc.).

8.3.4 Signing documents shipped with samples (i.e. air bills, chain-of-custody record(s), Sample Management Office (SMO) Traffic Reports, etc.)

8.3.5 Verifying and recording agreement or non-agreement of information on sample documents (i.e. sample tags, chain-of-custody records, traffic reports, air bills, etc.). If there is non-agreement, recording the problems, contacting the project manager for direction, and notifying appropriate laboratory personnel. (Client’s corrective action directions shall be documented in the case file.)

8.3.6 Initiating the paper work for sample analyses on laboratory documents (including establishing sample workorder files) as required for analysis or according to laboratory standard operating procedures.

8.3.7 Label samples with laboratory sample identification numbers and cross-referencing laboratory numbers to client numbers and sample tag numbers.

8.3.8 Scanning samples into the ICOC system.

8.3.9 Placing samples and spent samples into appropriate storage and/or secure areas.

8.3.10 Where applicable, making sure that sample tags are removed from the sample containers and included in the workorder file.

8.3.11 Where applicable, accounting for missing tags in a memo to the file or documenting that the sample tags are actually labels attached to sample containers or were disposed of, due to suspected contamination.

8.3.12 Monitoring storage conditions for proper sample preservation and prevention of cross-contamination.

8.3.13 Sending shipping containers with prepared sample bottles and sample instructions to clients who request them.

8.3.14 Calibrating the non-contact infrared temperature gun quarterly.

8.3.15 Disposal of samples after a specified time period determined by contract or client request.

8.4 Sample Receipt:

The Sample Custodian or his/her designated representative receives sample shipments at Spectrum Analytical, Inc. RI Division. Unless the shipment is a continuation of a previous workorder, a new workorder file is started for the sample.

The cooler is inspected for the following (if applicable) and findings are documented on the Sample Login Form (Figure 8.4-1) for USEPA CLP samples, and on the Sample Condition Form (Figure 8.4-2) for all other samples:

- Custody seal (conditions and custody number)
- Air bill (courier and air bill #)

The cooler is then opened and the following items are checked (in order). Make sure the hood is turned on when the cooler is opened.

- Chain of custody (COC) records (or traffic report). These are usually taped to the inside of the cooler cover.
- Radioactivity using the Geiger counter, which continuously monitors the receiving area for radiation
- Cooler temperature using the non-contact infrared temperature gun. Record the temperature of a temperature blank if available, using a calibrated thermometer. Record each temperature on the COC.

The Sample Custodian will perform the following:

- Remove the sample containers and arrange them in the same order as documented in the chain of custody report.
- Inspect condition of the sample containers.
- Assign laboratory sample ID and cross-reference the laboratory ID to the client ID.
- Remove tags and place in the workorder file.
- Check preservative and document in the Sample Condition Form (Figure 8.4-2) if needed. If additional preservative is needed, it is added at this time.
- Check for air bubbles in aqueous samples and for proper preservation and immersion of soil samples designated for volatile organic analysis.
- Ensure peer review occurs for proper cross-referencing and labeling of sample containers.

Any discrepancies or problems are noted in the Sample Condition Notification Form (Figure 8.4-3).

The sample custodian conveys the information to the project manager who will in turn inform the client, or may directly inform the client of the discrepancies.

Samples can be rejected at Spectrum Analytical, Inc. RI Division for any of the following reasons:

1. Complete and proper documentation was not sent with the samples.
2. Sample labels cannot be identified because indelible ink was not used during the sampling procedure.
3. Hold times had already been exceeded when samples arrived at the laboratory.
4. Inadequate sample volume.
5. Potential cross-contamination has occurred among samples.
6. Samples are inadequately preserved.
7. The samples or shipping container is badly destroyed during shipping.
8. The samples are potentially radioactive.
9. The samples represent untreated fecal waste for which Spectrum Analytical Inc., RI Division employees are currently not inoculated against.

In all instances, the client is contacted initially before any action is taken at Spectrum Analytical, Inc. RI Division.

The Sample Custodian signs the Sample Receipt Form and originates a file folder for the set of samples. The following forms are included in the file: the Sample Receipt Form, chain of custody records, shipping information, and an orange

Sample Condition Notification Form if any problems or discrepancies need to be addressed.

When the Sample Custodian is not available to receive samples, another lab staff member will sign for the sample container. The time, date and name of the person receiving the container are recorded on the custody records. In addition, the cooler temperature is measured and recorded on the Sample Condition Form. The samples are then stored in the centralized walk-in refrigerator in the sample receipt area. The sample receipt area is located in the secure central storage facility of the laboratory. VOA samples are stored in the VOA analysis laboratory. The samples are officially received and documented by the Sample Custodian or designee before the next business day.

At times, samples will be sent to another lab for analysis not performed at Spectrum Analytical, Inc. RI Division. These subcontracted analyses are performed by laboratories certified to perform the analyses. The use of a subcontractor laboratory is discussed with the client prior to sending samples, per Spectrum Analytical, Inc. RI Division's Project Management Standard Operating Procedure.

These samples are packed to prevent breakage and stored in a cooler in the walk-in or stored in the small refrigerator in the central storage facility. The samples are either hand delivered to a local sub-contract lab, or shipped with sufficient coolant to maintain a 4 degree temperature by air courier under Spectrum Analytical, Inc. RI Division's chain-of-custody (Figure 8.4-4).

8.5 Sample Log-in Identification:

8.5.1 Sample Identification:

To maintain sample identity, each sample received at Spectrum Analytical, Inc. RI Division is assigned unique sample identification (Sample ID) numbers. Samples are logged into the laboratory via the Laboratory Information Management System (LIMS).

After inspecting the samples, the Sample Custodian logs each sample into the LIMS, which assigns a lab Sample ID Number. These Numbers are assigned sequentially in chronological order. Spectrum Analytical Inc., RI Division Sample Identification Numbers appear in the following format: **YXXXX-NNF**

In which: Y – represents the current year with A for 2002, B for 2003, C for 2004, etc.
XXXX – represents a four-digit work order number that is assigned sequentially to each submittal of samples
NN – represents the sample number within the group or workorder.

F – represents the fraction. All sample portions that are received in identical bottles with identical preservatives are grouped into one fraction.

For example, the first fraction of the fifth sample of the 20th workorder of 2003 would have the number: B0020-05A

The Sample ID Number is recorded on the Sample Login Form (Figure 8.4-1) for USEPA CLP samples, and on the Sample Condition Form (Figure 8.4-2) for all other samples. Information on these forms cross-reference the Sample ID Numbers with SDG numbers, sample tag numbers and/or other client identifiers. Each sample is clearly labeled with its lab Sample ID Number by the Sample Custodian. The same sample ID Number appears on the LIMS status report, on each sample preparation container and extract vial associated with the sample.

8.5.1.1 Sample Extract Identification:

As described in Section 8.5.1, a sample extract is identified with the same unique sample identification number as the sample from which it derives

8.5.2 Sample Login:

The sample login system at Spectrum Analytical, Inc. RI Division consists of computerized entry using LIMS (Figure 8.5-1). The information recorded onto the Workorder Report includes:

- Workorder number
- Client name
- Project name and location
- Final data report format
- Date of receipt
- Date sample collected
- Due date, fax and/or hardcopy
- EDD requirements
- Comments or notes on the workorder
- Lab Sample Identification numbers
- Client Sample Identification numbers
- Sample matrix
- Analyses required
- Case number, where used by the client
- SDG number, where used by the client

8.5.3 Sample Information:

After sample information is properly recorded and the samples have been properly logged into the LIMS, bottle labels are generated and applied to the sample containers. The Sample Custodian notifies the Project Manager or peer or supervisor to review the sample bottle labeling. This person reviews all the information associated with the samples. He/she verifies (by initialing) the correctness of the information on the Sample Condition Form or Sample Log-In Form. Sample login information is available through the LIMS to all appropriate laboratory staff. The Sample Custodian then scans the samples into the IntCOC system and posts the samples.

The Sample Custodian initiates a red workorder file. This file contains the original Sample Log-In Form or Sample Condition Form, air bills, SMO traffic reports, sample tags, workorder reports and all correspondence with the Client or SMO or others. The red workorder file is forwarded to the Project Manager for review of the login paperwork, and for updating status of the workorder in the LIMS. Once the login information is thoroughly reviewed for correctness, the red workorder file is stored in the data reporting area. Analytical data are placed in this as analyses are completed and data are reviewed.

8.6 Sample Storage and Disposal:

Samples at Spectrum Analytical, Inc. RI Division are stored in a central storage facility or in satellite designated areas, (see SOP 30.0003 Sample Receipt Storage Tracking and Disposal). After sample receipt and login procedures are completed, the Sample Custodian places the samples in the centralized walk-in refrigerator. Volatile Organic sample aliquots are released to the volatile organic lab with documentation (Figure 8.6-1).

The central storage facility is for samples only; no standards or reagents are to be stored there. Access to the centralized sample storage facility is limited by keypad entry at all times. All sample storage areas are within the secure laboratory facility.

All sample/extract refrigerators are maintained at 4°C ± 2°C. Standards are kept in freezers maintained at -10 to -20°C. The temperature is recorded electronically using temperature probes that are affixed inside all refrigerator and freezer units (see SOP #80.0020 Temperature Monitoring Systems).

When analysis is complete, any remaining sample is retained in the designated storage facility until it may be removed for disposal (see SOP 30.0024 Sample Disposal). Broken and damaged samples are promptly disposed in a safe manner. Unless there is a specific request by the client, excess, unused sample aliquots are stored for at least 30 days after the submission of compliant data (USEPA is 60 days for samples and empty containers). The samples are then disposed after such

period. USEPA and NYS ASP extracts are stored under refrigeration for at least one year. Other extracts are stored under refrigeration for up to three months, unless there is a specific agreement with the client. After such time, the extracts are disposed. All disposals are performed in a manner compliant with federal and state regulations. International samples require special disposal procedures associated with the USDA Soil Permit (see SOP #30.0024 Sample Disposal).

8.6.1 Extract Transfer:

The extracts generated during the preparation for the organic analyses are transferred from the Organic Prep Lab to the Analysis Labs. The transfer of extracts for Semivolatiles, TPH, Pesticides and PCBs, are documented electronically in the Prep Batch Log with the storage location (refrigerator ID).

Metals analysis samples that are transferred from the prep area to the analysis room are also documented in the Prep Batch Log with the storage location (ICP or Hg lab).

There is no extract transfer that occurs with either Wet Chemistry or VOA samples.

8.6.2 Extract Storage:

Semivolatile, Pesticide/PCB, and TPH extracts, which are contained in crimp top vials or screw cap vials with Teflon lined septa, are stored at 4°C ± 2°C. Semivolatile and Pesticide/PCB extracts are stored in refrigerators in the Semivolatiles Analysis room. They are catalogued numerically by workorder number that approximates chronological order, according to date of receipt. USEPA CLP extracts are stored separately within the refrigerator from sample extracts of other clients.

Excess Pesticide extracts, not analyzed, are stored in screw cap vials with Teflon lined septa in the Organic Prep Lab. In most instances, they consist of the remaining 8-9 mL aqueous and soil sample extracts and are stored chronologically by workorder.

8.7 Sample Tracking:

When a sample is removed from storage, the analyst must scan each jar or bottle taken, using the IntCOC program and their user ID. When the sample(s) are returned to the central storage facility, the analyst must scan the samples back into the system using the IntCOC program and their user ID, and return the physical samples to their original storage location. In addition to the individual's initials, the date and time is recorded. This system maintains the location of the sample at any point in time.

Chain-of-custody of a sample ensures that the sample is traceable from the field, where it was taken, through laboratory receipt, preparation, analysis and finally disposal. The primary chain-of-custody documents are used to locate a sample at any point in time.

1. The chain-of-custody form from the field describes the origin and transportation of a sample;

2. The ICOC document acceptance of a sample by Spectrum Analytical Inc., RI Division; and

3. The ICOC documents which analyst has custody of the sample after removal from storage.

4. The sample preparation logs and/or extract transfer logs document when the extracts or digestates were received by the analytical labs and where they are stored in the refrigerator.

Figure 8.4-1
USEPA CLP Sample Login Form

SAMPLE LOG-IN SHEET
FORM DC-1

| Lab Name | | | | | Page _____ of _____ |
|---|---|---|---|---|---|
| Received By (Print Name) | | | | | Log-in Date |
| Received By (Signature) | | | | | |
| Case Number | | Sample Delivery Group No. | | | Mod. Ref. No. |
| Remarks: | | | Corresponding | | |
| | | EPA Sample # | Sample Tag # | Assigned Lab # | Remarks: Condition of Sample Shipment, etc. |
| 1. Custody Seal(s) | Present/Absent* Intact/Broken | | | | |
| 2. Custody Seal Nos. | _____ | | | | |
| 3. Traffic Reports/ Chain of Custody Records (TR/COCs) or Packing Lists | Present/Absent* | | | | |
| 4. Airbill | Airbill/Sticker Present/Absent* | | | | |
| 5. Airbill No. | _____ | | | | |
| 6. Sample Tags | Present/Absent* | | | | |
| Sample Tag Numbers | Listed/Not Listed on Chain-of-Custody | | | | |
| 7. Sample Condition | Intact/Broken*/ Leaking | | | | |
| 8. Cooler Temperature Indicator Bottle | Present/Absent* | | | | |
| 9. Cooler Temperature | _____ | | | | |
| 10. Does information on TR/COCs and sample tags agree? | Yes/No* | | | | |
| 11. Date Received at Laboratory | _____ | | | | |
| 12. Time Received | _____ | | | | |
| Sample Transfer | | | | | |
| Fraction | Fraction | | | | |
| Area # | Area # | | | | |
| By | By | | | | |
| On | On | | | | |

* Contact SMO and attach record of resolution

| Reviewed By | Logbook No. |
|---|---|
| Date | Logbook Page No. |

SAMPLE LOG-IN SHEET

| Lab Name: Spectrum Analytical Inc., Rhode Island Division | | Page __ of ___ |
|---|---|---|
| Received By (Print Name) | | Log-in Date |
| Received By (Signature) | | |
| Case Number | Sample Delivery Group No. | Mod. Ref. No. |

| Remarks: | |
|---|---|
| 1. Custody Seal(s) | Present/Absent* Intact/Broken |
| 2. Custody Seal NOs. | _____ _____ |
| 3. Traffic Reports/Chain of Custody Records or Packing Lists | Present/Absent* |
| 4. Airbill | Airbill/Sticker Present/Absent* |
| 5. Airbill No. | _____ _____ |
| 6. Sample Tags<br>Sample Tag Numbers | Present/Absent*<br>Listed/Not Listed on Traffic Report/Chain of Custody Record |
| 7. Sample Condition | Intact/Broken*/ Leaking |
| 8. Cooler Temperature Indicator Bottle | Present/Absent* |
| 9. Cooler Temperature | _____ |
| 10. Does information on Traffic Reports/Chain of Custody Records and sample tags agree? | Yes/No* |
| 11. Date Received at Lab | _____ |
| 12. Time Received | _____ |

| Sample Transfer | |
|---|---|
| Fraction | Fraction |
| Area# | Area# |
| By | By |
| On | On |

| | EPA Sample # | Aqueous/ Water Sample pH | Corresponding | | Remarks: Condition of Sample Shipment, etc. |
|---|---|---|---|---|---|
| | | | Sample Tag # | Assigned Lab # | |
| 1 | | | | | |
| 2 | | | | | |
| 3 | | | | | |
| 4 | | | | | |
| 5 | | | | | |
| 6 | | | | | |
| 7 | | | | | |
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| 18 | | | | | |
| 19 | | | | | |
| 20 | | | | | |
| 21 | | | | | |
| 22 | | | | | |

* Contact SMO and attach record of resolution

| Reviewed By | Logbook No. |
|---|---|
| Date | Logbook Page No. |

Figure 8.4-2
Sample Condition Form

# SPECTRUM ANALYTICAL, INC. RI DIVISION

**Sample Condition Form**



| Received By: | Reviewed By: | Date: | Spectrum RI Work Order #: |
|---|---|---|---|
| Client Project: | | Client: | |

| | | Lab Sample ID | | Preservation (pH) | | | | | VOA Matrix | Soil Headspace or Air Bubble ≥ 1/4" |
|---|---|---|---|---|---|---|---|---|---|---|
| | | | | $HNO_3$ | $H_2SO_4$ | HCl | NaOH | $H_3PO_4$ | | |
| 1) Cooler Sealed | Yes / No | | | | | | | | | |
| | | | | | | | | | | |
| 2) Custody Seal(s) | Present / Absent | | | | | | | | | |
| | Coolers / Bottles | | | | | | | | | |
| | Intact / Broken | | | | | | | | | |
| | | | | | | | | | | |
| 3) Custody Seal Number(s) | | | | | | | | | | |
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| | | | | | | | | | | |
| 4) Chain-of-Custody | Present / Absent | | | | | | | | | |
| | | | | | | | | | | |
| 5) Cooler Temperature | | | | | | | | | | |
| IR Temp Gun ID | | | | | | | | | | |
| Coolant Condition | | | | | | | | | | |
| | | | | | | | | | | |
| 6) Airbill(s) | Present / Absent | | | | | | | | | |
| Airbill Number(s) | | | | | | | | | | |
| | | | | | | | | | | |
| | | | | | | | | | | |
| | | | | | | | | | | |
| | | | | | | | | | | |
| 7) Samples Bottles | Intact / Broken / Leaking | | | | | | | | | |
| | | | | | | | | | | |
| 8) Date Received | | | | | | | | | | |
| | | | | | | | | | | |
| 9) Time Received | | | | | | | | | | |
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| | | | | | | | | | | |

Preservative Name/Lot No.:

VOA Matrix Key:

| | |
|---|---|
| US = Unpreserved Soil | A = Air |
| UA = Unpreserved Aqueous | H = HCl |
| M = MeOH | E = Encore |
| N = NaHSO4 | F = Freeze |

See Sample Condition Notification/Corrective Action Form yes / no

Form ID: QAF.0006

Rad OK yes / no

Figure 8.4-3
Sample Condition Notification Form

# *Spectrum Analytical, Inc. RI Division* Sample Condition Notification

**Project#:**_____ **Date of Receipt:**_____
**Client:**_____ **Received By:**_____
**Client project #/name:**_____

**Unusual Occurance Description:**

**Client Contacted:**

**Contacted via: Phone/Fax/E-mail**
**Date:**_____ **Time:**_____
**Contacted By:**_____
**Name of person contacted:**_____

**Client Response:**

**Responded via: Phone/Fax/E-mail**
**Date:**_____
**Name of person responding:**_____
**Responding to:**_____

**Action Taken:** _____

Form ID: QAF.0005

Figure 8.4-4
Spectrum Analytical, Inc. RI Division Chain-of-custody Form

SPECTRUM ANALYTICAL, INC.
*Featuring*
HANIBAL TECHNOLOGY

Page _____ of _____

# CHAIN OF CUSTODY RECORD

☐ 11 Almgren Drive
Agawam, MA 01001
(413) 789-9018

☐ 8405 Benjamin Road, Ste A
Tampa, FL 33634
(813) 888-9507

☐ 175 Metro Center Blvd
Warwick, RI 02886
(401) 732-3400

**Special Handling:**

TAT- Indicate Date Needed: _____
· All TATs subject to laboratory approval. Min. 24-hour notification needed for rushes.
· Samples disposed of after 60 days unless otherwise instructed.

Report To: _____
_____
_____
_____
Telephone #: _____
Project Mgr. _____

Invoice To: _____
_____
_____
_____
P.O. No.: _____ RQN: _____

Project No.: _____
Site Name: _____
Location: _____ State: _____
Sampler(s): _____

1=$Na_2S2O_3$ 2=HCl 3=$H_2SO_4$ 4=$HNO_3$ 5=NaOH 6=Ascorbic Acid 7=$CH_3OH$
8= $NaHSO_4$ 9= Deionized Water 10=$H_3PO_4$ 11= _____ 12= _____

List preservative code below:

QA/QC Reporting Notes:

DW=Drinking Water GW=Groundwater WW=Wastewater
O=Oil SW= Surface Water SO=Soil SL=Sludge A=Air
X1= _____ X2= _____ X3= _____

G=Grab C=Composite

| Lab Id: | Sample Id: | Date: | Time: | Type | Matrix | Containers: # of VOA Vials | # of Amber Glass | # of Clear Glass | # of Plastic | | Analyses: | | | | | | | | |
|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|
| | | | | | | | | | | | | | | | | | | | |
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QA/QC Reporting Level
☐ Level I ☐ Level II
☐ Level III ☐ Level IV
☐ Other _____

State-specific reporting standards:
_____

| Relinquished by: | Received by: | Date: | Time: | Temp°C |
|---|---|---|---|---|
| | | | | |
| | | | | |
| | | | | |
| | | | | |

☐ EDD Format _____
☐ E-mail to _____

Condition upon receipt:
☐ Ambient ☐ Iced ☐ Refrigerated ☐ DI VOA Frozen ☐ Soil Jar Frozen

Figure 8.5-1
Workorder Information Form

# Spectrum Analytical, Inc. Featuring Hanibal Technology -- Rhode Island Division

**WorkOrder: L1458**

**Client ID:** MITKEM_WARWICK
**Project:** INTERNAL TESTING
**WO Name:** INTERNAL TESTING
**Location:** WATER_TESTING, WW, 6/2012
**Comments:** Internal test

**Case:**
**SDG:**
**PO:** INTERNAL TESTING

**HC Due:** 07/12/12
**Fax Due:**
**Fax Report:** ☐

**Report Level:** LEVEL 2
**Special Program:**
**EDD:**

| Lab Samp ID | Client Sample ID | Collection Date | Date Recv'd | Matrix | Test Code | Samp / Lab Test Comments | HF | HT | MS | SEL | Storage |
|---|---|---|---|---|---|---|---|---|---|---|---|
| L1458-01A | WW-6/28-G | 06/28/2012 08:05 | 06/28/2012 | Aqueous | E624 | / | | | | Y | VOA |
| L1458-01B | WW-6/28-G | 06/28/2012 08:05 | 06/28/2012 | Aqueous | E625 | / Needs benzidine, 1,2-diphenyhydr, n-nitrosodimethl | | | | Y | Disposed |
| L1458-01C | WW-6/28-G | 06/28/2012 08:05 | 06/28/2012 | Aqueous | E335.4 | / | | | | | Disposed |
| L1458-02A | WW-6/28-C | 06/28/2012 15:00 | 06/28/2012 | Aqueous | E200.7 | / Cd, Cr, Cu, Pb, Ni, Ag, Zn | | | | Y | Disposed |
| L1458-02B | WW-6/28-C | 06/28/2012 15:00 | 06/28/2012 | Aqueous | SM5220 | / | | | | | Disposed |

HF = Fraction logged in but all tests have been placed on hold

HT = Test logged in but has been placed on hold

Figure 8.6-1
Volatiles Receiving Logbook Form

# Spectrum Analytical, Inc. RI Division : VOLATILE SAMPLES RECEIVING LOGBOOK

| VOA Log-In Date | Workorder | Client ID | Sample Numbers | Relinquished by: | Received by: | Pres. Used | F/R | Returned to R1 |
|---|---|---|---|---|---|---|---|---|
| | | | | | | | | |
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**Logbook ID 90.0191-04/12**

**Reviewed By:**

**"Preservative Used" Key**

**UA** = Unpreserved Aqueous **H** = HCL **A** = Air **M** = MeOH **E** = Encore

**US** = Unpreserved Soil **N** = $NaHSO_4$ **F** = Freeze **T** = Trace, HCL

# 9.0 CALIBRATION PROCEDURES AND FREQUENCIES

All purchased equipment, materials, and services must meet specific method requirements, standard requirements, or project specific requirements. These requirements are documented in the individual analytical or project SOPs.

## 9.1 Instruments:

Specific calibration and check procedures are given in the analytical methods referenced in Section 10. The frequencies of calibration and the concentrations of calibration standards are determined by the cited methods and any special project or contract-specific requirements. Standard calibration curves of signal response versus concentration are generated on each analytical instrument used for a project, prior to analysis of samples. A calibration curve of the appropriate linear range is established for each parameter that is included in the analytical procedure employed and is verified on a regular basis with check standards as specified in the appropriate CLP Protocols. For non-CLP work, Spectrum Analytical, Inc. RI Division adheres to the calibration criteria specified by SW-846 and/or Standard Methods for both organic and inorganic analyses. Where requested, other method specific calibration criteria are used. Refer to the individual Standard Operating Procedures listed in Figure 11.7-1 of this QAP for the specific calibration and check procedures as well as concentration and frequency requirements.

For organic analyses whenever possible, unless otherwise specified in the individual methods, the initial calibration standards (ICAL), continuing calibration verification standards (CCV), laboratory control sample spike (LCS) and matrix spike (MS) will all be from the same source. The initial calibration verification (ICV) standards are prepared from a separate source. Refer to the Standard Operating Procedures listed in Figure 11.7-1 of this QAP for the specific calibration source and procedural requirements of each method. The following are examples of calibration procedures for various instrumental systems:

**GC/ECD and GC/FID** – An initial calibration is performed using five different concentration levels for each parameter of interest for SW-846 analyses. The initial calibration is done on each column and each instrument, and is repeated each time a new column is installed or whenever a major change is made to the chromatographic system.

Initial calibration verification (ICV), near mid level concentration for all analytes, is performed immediately after the calibration. If the ICV does not meet method specific criteria, a new calibration curve is generated and an ICV is analyzed. If repeated ICV failures are encountered, the system is checked to find the cause of these failures, and the problem is corrected. For certain GC/FID analyses (i.e. GRO /DRO), the instrument is calibrated using individual compounds while the laboratory control sample or ICV uses a product (diesel or gasoline).

Continuing calibration verification (CCV), near a mid-level concentration for all analytes, is run at intervals determined by sample number or time allowed, as required by the individual methods. If CCV values are determined outside the upper limit of the method specified range and if no analytes were detected in the samples, the run will be accepted as valid and 'Non Detects' reported for the sample. If an analyte is detected and the CCV is out at the high end, the problem will be identified and corrected and the affected samples will be re-analyzed with a compliant CCV.

If a CCV value is out of the method specified limits at the lower limit, the cause of the problem will be identified and corrected, and all samples affected by the out of control CCV will be rerun with a compliant CCV.

For CLP-type analyses, the continuing calibration takes place at the beginning of the analytical sequence and once every twelve (12) hours throughout the analytical sequence, and again at the end of the sequence. The percent difference in calibration factors for each standard must not exceed the criteria specified by the method.

If a CCV fails to meet criteria limits, a new calibration curve will be generated and all samples affected will be re-analyzed.

**GC/MS** – For CLP methods, a minimum of five-level calibration (four-level for select semivolatile compounds) is carried out for each analyte per system before analysis of samples take place.

Continuing calibrations, near midpoint levels, are analyzed every twelve hours of instrument analysis time for CLP analyses.

Re-calibration takes place whenever a major change occurs in the system, such as a column change in the GC or a source cleaning of the mass spectrometer or when the continuing calibration fails to meet method specific requirements.

Tunes are performed once every twelve (12) hours of instrument run time for all CLP-type and SW846 analyses. The GC/MS system is tuned to USEPA specifications for bromofluorobenzene (BFB) or decafluorotriphenylphosphine (DFTPP) for volatile and semivolatile analyses, respectively. Extended tune time is allowed in CLP SOM protocols where an ending CCV is acceptable as an opening CCV.

More detailed instrument and method-specific calibration procedures and criteria are described in the individual analysis SOPs.

**ICP/AES and ICP/MS** – Instrument calibration, for each wavelength used, occurs at the start of each analysis. The calibration curve is constructed per method specification.

An initial calibration verification and initial calibration blank (ICB) are analyzed before analysis of samples. If the ICV and ICB do not meet method specific criteria for an analyte, the analyte is re-analyzed with a new calibration.

During the analysis, a continuing calibration verification (CCV) and continuing calibration blank (CCB) is analyzed at least every ten (10) samples or two hours depending on method. If either the CCV or CCB fails to meet method specific criteria for an analyte, the source of the problem is investigated. If it can be determined that the failed CCV and/or CCB is not representative (such as for instrument carryover from previous sample or from an empty autosampler tube), the CCV and/or CCB are re-analyzed and the reason for the failure documented. If a failure still occurs, further corrective action is performed, and the analyte is re-analyzed with a new calibration.

The CCV is obtained from a source independent from that of the standards. The CCV concentration for the different analytes are at method specified levels.

**The Flow Injection Mercury System (FIMS)** - Instrument calibration occurs at the start of each analysis. The calibration curve is constructed per method specification.

An initial calibration verification (ICV) and initial calibration blank (ICB) are analyzed before analysis of samples. If the ICV and ICB do not meet method specific criteria for Mercury, re-calibration and reanalysis are required.

During the analysis, a continuing calibration verification (CCV) and continuing calibration blank (CCB) is analyzed at least every ten (10) samples. If either the CCV or CCB fails to meet method specific criteria for Mercury, the source of the problem is investigated. If it can be determined that the failed CCV and/or CCB is not representative (such as for instrument carryover from previous sample or from an empty autosampler tube), the CCV and/or CCB are re-analyzed and the reason for the failure documented. If a failure still occurs, further corrective action is performed, and the analyte is re-analyzed with a new calibration.

The CCV is obtained from a source independent from that of the standards. The CCV concentration for Mercury is at method specified levels.

Other instrumentation:

**IC-** The Ion Chromatograph is calibrated each day of use. Calibration verification is analyzed at the beginning, end, and at least every 10 samples. The verification standard is from an independent source. If the calibration verification does not

meet method specific criteria for an analyte, it is re-analyzed once. If failure still occurs, a new calibration curve is established and any affected samples are reanalyzed.

**pH**- the meter is calibrated at two pH levels (4.0 and 10.0) before analyses of samples. The pH 7.0 buffer is analyzed as an LCS and recovery is calculated.

**Lachat 8000**- automated flow-through spectrophotometer is calibrated per method specification before the analyses of samples.

An initial calibration verification and initial calibration blank (if required) are analyzed before analysis of samples. If the ICV and/or ICB do not meet method specific criteria for an analyte, re-calibration must occur.

During the analyses, continuing calibration verification and continuing calibration blanks are analyzed at least every ten (10) samples. If either the CCV or CCB fails to meet specified criteria for an analyte, the source of the problem is investigated. If it can be determined that the failed CCV and/or CCB is not representative (such as for instrument carryover from previous sample or from an empty autosampler tube), the CCV and/or CCB are re-analyzed and the reason for the failure documented. If a failure still occurs, further corrective action is performed, and the analyte is re-analyzed with a new calibration.

The CCV is obtained from a source independent from that of the standards. The CCV concentration for the different analytes are at method specified levels.

**SpecGenesys**- manual spectrophotometer is calibrated per method specification.

Calibration curve calibration verification is analyzed at the beginning, end, and at least every 10 samples. The verification standard is from an independent source. If the calibration verification does not meet method specific criteria for an analyte, it is re-analyzed once. If failure still occurs, a new calibration curve is established and any affected samples are reanalyzed. Calibration curves are established at least quarterly.

Annual calibration and preventative maintenance is required by an outside vendor unless calibration can be performed in-house using a calibration kit.

**Balances**: are calibrated by an outside source on an annual basis.

The balances are calibrated externally each day of use by a lab technician with NIST traceable Class “1” or “2” weights. The weights are certified by an outside service on a regular basis, not to exceed five years.

**Thermometers** are calibrated once a year against a NIST-verified thermometer or as they are replaced. Digital thermometers are verified quarterly. The NIST-verified thermometers are certified by an outside certified service annually.

**Gel Permeation Chromatography** is used to clean samples according to CLP and client requirements. GPCs are calibrated using a calibration standard provided by Ultra Scientific, Cat. # CLP-340. Once a successful calibration is achieved it is valid for a period of seven days.

9.2 Standards and Reagents:

**Standard** reference materials used for routine calibration, calibration checks, and accuracy are obtained from commercial manufacturers. These reference materials are traceable to the source and readily compared to EPA references. All standards come with a Certificate of Analysis which is kept on record in the appropriate laboratories. When a chemical standard can not be purchased in solution form, a neat source may be bought. The lab must attempt to obtain the highest purity available. If the lab can not find a neat source with at least 97% purity, the laboratory must document why. In addition, the impurity correction factor must be used when calculating the standard concentration. See SOP #80.0001, Standard Preparation, Equivalency and Traceability, for more details. While most standards are traceable to NIST; however, certain projects, especially those involving pesticide registration, may necessitate the use of reference standards supplied by the client. New standards are also routinely validated against known standards that are traceable to EPA or NIST reference materials.

Organic Preparatory Lab Surrogate and Matrix spikes are prepared in the appropriate instrument labs and then QA'd by diluting the standard and analyzing it on the GC or GC/MS. Criteria for the diluted spike analysis must meet the method or in-house criteria. If acceptable, the spike is able to be used. If unacceptable, another standard is prepared and the same steps repeated. Data from the QC analysis is retained in the laboratory for reference and traceability.

Primary, intermediate and working standards are all named using specific nomenclature as designated in the QA Department SOP# 80.0001, Standard Preparation, Equivalency and Traceability.

Standards are dated and labeled upon arrival. Any material exceeding its shelf life as described by the methods in QAP Section 10 is discarded and replaced. Standards are periodically analyzed for concentration changes/degradation and inspected for signs of deterioration such as color change and precipitate formation. Standards Logbooks, which contain all pertinent information regarding the source and preparation of each analytical standard, are maintained by each of the laboratory departments in the LIMS.

See individual analytical SOPs (listed in Figure 11.7-1), sections 7 and 8 for standards preparation procedures.

**Solvents** are tested for purity prior to use to ensure there is no external source of contamination. For organic solvents, each lot number of solvent is QC'd prior to use. This is accomplished by concentrating an aliquot of solvent or extracting with reagent media (such as sodium sulfate) in the same manner as the samples and analyzing it for contamination by GC/MS. Any detectable analyte could render the solvent or reagent unsuitable for use. Supervisors make the final decision as to the suitability of the solvent or reagent, and whether the lot may be used for standard or sample preparation.

**Chemicals and Reagents** are stored in the respective laboratories during use. Backup supplies are stored in the stockroom. Reagent grade chemicals are used in all tests. All dry chemicals and reagents are given a 5-year expiration period unless designated otherwise by the manufacturer. Sometimes the viability of the reagent does not remain throughout the entire 5-year period (as determined through investigation following poor results in a preparation method blank or bench analysis, for example). In this case, the chemical or reagent is readily discarded. Acids/caustics are given a 3-year expiration period unless designated otherwise by the manufacturer. Solvents are given a 1-year expiration period unless designated otherwise by the manufacturer.

Chemicals and reagents are logged into the laboratory and each bottle is given a unique ID. The ID is based upon the date of its arrival at the laboratory. The only exceptions include cases/cycletainers of solvents and cases of acids. For solvents and acids, the boxes/cases are labeled with received date to insure first in/first out usage. All other chemicals and reagents are named using specific nomenclature as designated in the QA Department SOP # 80.0013, Reagent Purchasing and Tracking.

When a bottle is opened in the laboratory, it is inspected to ensure it meets the requirements of the method. The analyst records his or her initials on the bottle along with the date opened and the ID. Any applicable certificates of analysis (COA) are scanned and archived. They may also be stored in the individual laboratories or in the QA Department.

# 10.0 ANALYTICAL PROCEDURES

Spectrum Analytical, Inc. RI Division uses the methods specified in Tables 10-1 through 10-6 unless otherwise specified by the client. Spectrum Analytical, Inc. RI Division performs analyses on non-potable waters, groundwater and soil/solids. The RI Division does not perform regulatory potable (drinking) water analyses with the exception of trace metals by EPA 200.8, or environmental lead (paint chips, wipes, etc. for RIDOH compliance) testing. Associated Standard Operating Procedures related to these analytical procedures can be found in Figure 11.7-1 of this QAP.

Table 10-1
Potable Water Analytical Methods

| Parameter | Method Description | Method Reference |
|---|---|---|
| Metals | ICP-MS | 200.8 |

Table 10-2
Non-potable Water Analytical Methods

| Parameter | Method Description | Method Reference |
|---|---|---|
| Metals | ICP-AES | 200.7 |
| Mercury | Cold Vapor | 245.1 |
| Cyanide | Midi-distillation<br>Automated | EPA 335.4 |
| Alkalinity | Titration | SM2320B |
| Anions<br>Chloride<br>Sulfate<br>Nitrate<br>Nitrite<br>Orthophosphate<br>Bromide<br>Fluoride | Ion Chromatography | EPA 300.0 |
| Volatile Fatty Acids<br>Acetic<br>Butyric<br>Lactic<br>Propionic<br>Pyruvic | Ion Chromatography | EPA 300.0 Mod |
| pH | Electrode | SM4500 H+ B |
| Sulfate | Turbidimetric | SM4500-SO4 E. |
| Ammonia | Distillation/Titration | SM4500-NH3 B, C |
| Total Kjeldahl Nitrogen | Digestion<br>Distillation/Titration | SM4500- Norg C<br>SM4500- NH3 B, C |
| Orthophosphate | Ascorbic, Manual | SM4500-P E |
| Total phosphate | Persulfate, Manual | SM4500-P B5 & E |

Table 10-2
Non-potable WaterAnalytical Methods (cont.)

| Parameter | Method description | Method Reference |
|---|---|---|
| Chemical Oxygen Demand | Spectrophotometric(Closed Reflux) | SM5220-D |
| Total Organic Carbon | Combustion | SM5310 B |
| Phenols | Distillation, 4-AAP,<br>Direct Photometric | SM5530 B<br>E420.1 |
| Total Dissolved Solids | Gravimetric | SM2540 C |
| Total Solids | Gravimetric | SM2540 B |
| Total Suspended Solids | Gravimetric | SM2540 D |
| Total Settleable Solids | Imhoff cones | SM2540 F |
| Hexavalent Chromium | Diphenyl Carbazide Colorimetric | SM 3500Cr B |
| Volatile Organics | | |
| Halocarbons | Purge & Trap, GC/MS | 624 |
| Aromatics | Purge & Trap, GC/MS | 624 |
| Semivolatile Organics | Extraction, GC/MS | 625 |
| Organochlorine Pesticides/ PCBs | Extraction, GC/ECD | 608 |
| Oil & Grease (HEM, SGT) | Extraction, Gravimetric | 1664A |

Table 10-3
SW-846 Inorganic Analytical Methods

| Parameter | Method Description | Method Reference |
|---|---|---|
| Metals | | |
| Aqueous | Acid digestion | Method 3005A/3010A |
| | ICP/AES | Method 6010C |
| | ICP/MS | Method 6020A |
| Solid | Acid digestion | Method 3050B |
| | ICP/AES | Method 6010C |
| | ICP/MS | Method 6020A |
| Mercury | | |
| Aqueous | Permanganate digestion<br>Cold Vapor analysis | Method 7470A |
| Solid | Permanganate digestion<br>Cold Vapor analysis | Method 7471B |
| Hexavalent Chromium | | |
| Aqueous | Colorimetric | Method 7196A |
| Solid | Acid Digestion<br>Colorimetric | Method 3060A/7196A |
| Cyanide | | |
| Aqueous | Midi-distillation<br>Automated | Method 9012B |
| Solid | Midi-distillation<br>Automated | Method 9012B |
| pH | | |
| Solid | Electrode | Method 9045D |
| Ignitability (Flashpoint) | | |
| Aqueous | Pensky-Martens closed cup | Method 1010A |
| Solid | Pensky-Martens closed cup | Method 1010A Mod. |
| Reactive Cyanide | | |
| Solid & Aqueous | Distillation<br>Automated | SW 846 7.3.3.2 |

Table 10-3
SW-846 Inorganic Analytical Methods (cont.)

| Parameter | Method Description | Method Reference |
|---|---|---|
| Reactive Sulfide<br>Solid & Aqueous | Distillation<br>Colorimetric | SW 846 7.3.4.2 |
| Anions<br>Chloride<br>Sulfate<br>Nitrate<br>Nitrite<br>Orthophosphate<br>Bromide<br>Fluoride | Ion Chromatography | SW 846 9056A |
| Total Organic Carbon | Combustion | SW 846 9060A |
| Toxicity Characteristic Leaching Procedure (TCLP) | | |
| Aqueous | Leachate by Filtration | Method 1311 |
| Solid | Leachate Generation | Method 1311 |
| Synthetic Precipitation Leaching Procedure (SPLP) | | |
| Aqueous | Leachate by Filtration | Method 1312 |
| Solid | Leachate Generation | Method 1312 |

Table 10-4
SW-846 Organic Analytical Methods

| Parameter | Sample Preparation | Sample Analysis |
|---|---|---|
| Volatile Organic Compounds | | |
| Aqueous | Method 5030B | Method 8260C |
| Solid | Method 5035A | Method 8260C |
| 1,2-Dibromo-3-chloropropane<br>1,2-Dibromomethane | Micro extraction<br>GC\ECD Analysis | Method 8011 |
| Semivolatile Organic Compounds | | |
| Aqueous | Method 3510C<br>Method 3520C | Method 8270D |
| Solid | Method 3540C<br>Method 3550B<br>Method 3545<br>Method 3570 | Method 8270D |
| Organochlorine Pesticides | | |
| Aqueous | Method 3510C<br>Method 3520C | Method 8081B |
| Solid | Method 3540C<br>Method 3550B<br>Method 3545<br>Method 3570 | Method 8081B |
| Polychlorinated Biphenyls (Aroclors and Congeners) | | |
| Aqueous | Method 3510C<br>Method 3520C | Method 8082A |
| Solid | Method 3540C<br>Method 3550B<br>Method 3545<br>Method 3570 | Method 8082A |
| Total Petroleum Hydrocarbons | | |
| Aqueous | Method 3510C<br>Method 3520C | Method 8015B,D |
| Solid | Method 3540C<br>Method 3550B | Method 8015B,D |

Table 10-4
SW-846 Organic Analytical Methods (cont.)

| Parameter | Sample Preparation | Sample Analysis |
|---|---|---|
| Herbicides | | |
| Aqueous | Method 8151A | Method 8151A |
| Solid | Method 8151A | Method 8151A |
| Toxicity Characteristic Leaching Procedure (TCLP) | | |
| Aqueous | Method 1311 | |
| Solid | Method 1311 | |
| Synthetic Precipitation Leaching Procedure (SPLP) | | |
| Aqueous | Method 1312 | |
| Solid | Method 1312 | |
| Gel Permeation Chromatography (GPC) | | |
| Aqueous | Method 3640A | |
| Solid | Method 3640A | |
| Florisil Cleanup | | |
| Aqueous | Method 3620B | |
| Solid | Method 3620B | |
| Silica Gel Cleanup | | |
| Aqueous | Method 3630C | |
| Solid | Method 3630C | |
| Sulfur Cleanup | | |
| Aqueous | Method 3660B | |
| Solid | Method 3660B | |
| Sulfuric Acid Cleanup | | |
| Aqueous | Method 3665A | |
| Solid | Method 3665A | |

Table 10-5
CLP-Type Analytical Methods

| Parameter | Method Reference |
|---|---|
| USEPA CLP Organics | OLM04.3, SOM01.2 |
| USEPA CLP Inorganics | ILM05.4, ISM01.3 |
| USEPA Low Level Organics | OLC03.2 |
| NYS-ASP CLP Organics | ASP 2000/2005 SOW |
| NYS-ASP CLP Organics | ASP 2000/2005 SOW |

Table 10-6
Other Analytical Methods

| Parameter | Method Reference |
|---|---|
| Volatile Petroleum Hydrocarbons | |
| Aqueous | MADEP VPH 1.1 |
| Solid | MADEP VPH 1.1 |
| Extractable Petroleum Hydrocarbons | |
| Aqueous | MADEP EPH 1.1 |
| Solid | MADEP EPH 1.1 |
| Extractable Total Petroleum Hydrocarbons | |
| Aqueous | CT ETPH 99-3 |
| Solid | CT ETPH 99-3 |
| Diesel Range Organics | |
| Aqueous | ME 4.1.25 |
| Solid | ME 4.1.25 |
| Gasoline Range Organics | |
| Aqueous | ME 4.2.17 |
| Solid | ME 4.2.17 |

## 10.1 Analytical References

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9. The EPA 600 Series. Methods for Organic Chemical Analysis of Municipal and Industrial Wastewater, Appendix A, 40 CFR Part 136, Federal Register, Vol. 49, No. 209, 1984.

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12. Standard Methods for the Examination of Water and Wastewater, 20th Edition, APHA, Washington, D. C., 1998.

13. Test Methods for Evaluating Solid Waste-Physical/Chemical Methods, SW-846, 3rd Edition Final Updates I through IV. Office of Solid Waste and Emergency Response, USEPA, Washington, D. C., 1998. Status table found at http://www.epa.gov/epawaste/hazard/testmethods/sw846/pdfs/methstat.pdf

14. USEPA Contract Laboratory Program. Statement of Work for Organic Analysis, USEPA, OLM04.3, OLC03.2, and SOM01.2.

15. USEPA Contract Laboratory Program. Statement of Work for Inorganic Analysis, USEPA ILM05.4, and ISM01.2.

16. Maine Health and Environmental Testing Laboratory. Modified GRO and DRO Methods, Method 4.2.17 and 4.1.25, September 6th 1995.

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# 11.0 DATA COLLECTION, REDUCTION, VALIDATION AND REPORTING

## 11.1 Data Collection:

Most of the lab's data is uploaded into the LIMS systems directly from the instruments. The exception is the GC's and GC/MS's in which data is first processed in Target and then uploaded into the LIMS.

Either the instrument analyst or data reporting group will upload the data into the LIMS. The person who performs the upload does a technical review to ensure recoveries of CCVs, MS, MSD, and LCS all seem to be correct. A completeness review is done at this time to ensure all applicable samples have been uploaded for all the necessary analytes.

Next, an employee with a technical background will perform the QA process of the uploaded data. This person is either a supervisor or someone with extensive experience in environmental chemistry. Corrections to the run are made at this step if necessary. When the review is complete, this technical person authorizes the data to be reported by "QA-ing" the run in the LIMS. For a more detailed view of the LIMS uploading/review procedure, see SOP # 110.0028, Data Validation/Self Inspection Procedures.

## 11.2 Data Reduction:

Instrument printouts, computer terminal displays, chromatograms, strip chart recordings and physical measurements provide raw data that are reduced to concentrations of analytes through the application of the appropriate calculations.

Equations are generally given within the analytical methods referenced in Section 10. Data reduction may be performed automatically by computerized data systems on the instrument, manually by the analyst, or by PCs using verified spreadsheets and/or data base software.

## 11.3 Data Verification:

The verification process requires the following checks to be made on data before they are submitted to the client:

- A completeness inspection is required which ensures that all required data are included in the data packages submitted to the client and that the appropriate signatures are present on the data packages.

- A contract compliance screening to ensure that contractual requirements have been satisfied.

- A consistency check to ensure that nominally identical or similar data appearing in different places within a data package are consistent with respect to value and units.

- All manual integrations are properly performed and documented.

- A correctness check to ensure that reported data have been calculated correctly or transcribed correctly.

11.4 Data Validation:

Data validation is an essential element of the QA evaluation system. Validation is the process of data review and subsequent acceptance or rejection based on established criteria.

The following analytical criteria are employed by Spectrum Analytical, Inc. RI Division in the technical evaluation of data:

- Accuracy requirements.
- Precision requirements.
- Detection limits requirements.
- Documentation requirements.

As in the case of EPA/CLP procedures, data acceptance limits may be defined within the method. As one means of tracking data acceptability, quality control charts are plotted for specific parameters determined in similar, homogeneous matrices. Control limits for non-CLP methods are statistically determined as analytical results are accumulated unless provided by method or program.

Upon completion of the evaluation, the evaluator dates and initials the data review checklist as described in Section 11.5 below.

11.5 Data Interpretation and Reporting:

Interpretation of raw data and calculation of results are performed by a scientist experienced in the analytical methodology. Upon completion of data reduction, the scientist signs for the reported results on the data review checklist. For GC/ECD, GC/FID and GC/MS, a technical peer review is performed using the data processing software prior to form generation.

The laboratory supervisor is responsible for the data generated in that department. The supervisor or other senior technical staff performs an independent review of data and completed report forms. Members of the QA staff also check the results on selected sets of data (usually 10%).

11.5.1 Report Formats:

Spectrum Analytical, Inc. RI Division uses a flexible data reporting system where final report format is based on the requirements of the client. The two most common types of data reports generated by the Spectrum Analytical Inc., RI Division are Level 2 or "commercial-format" and Level 4 or "CLP-format". The lab adapts its data report format, wherever possible, to meet customer requirements. Occasionally reports are generated that are a compromise between "commercial" and CLP-format deliverables or are designed to meet the needs of a particular regulatory format or sampling program.

Drinking water Metals samples have special reporting requirements and client notification criteria for results exceeding the MCL. Clients are notified via facsimile or e-mail of all samples that exceed any EPA maximum contaminant level (MCL), maximum residual disinfectant level or reportable concentration within 24 hours of obtaining valid data. Drinking water Metals analyses are reported using a custom reporting format that will list the associated MCL and certification status for each element. Additionally, the requirement for the 24 hour MCL exceedence report will be highlighted in the comment section of the Subcontract Work Order for any subcontracted potable water samples.

Commercial data reports are generated using the LIMS. All instrumental analysis data are uploaded from instruments to the LIMS by electronic data transfer. Non-instrumental analysis data or sample preparation data are manually entered into the LIMS. All manual data entry steps are double-checked to insure they are correct, and instrumental data are spot-checked to insure the proper functioning of the data upload system. All data receive a 100% review before they are released to the client as final.

CLP data reports are generated using specialized CLP report modules in the LIMS for all inorganic and most organic analyses. These reports also undergo a 100% review before they are released to the client in their final form.

Records are maintained for all data, even those results that are rejected as invalid.

11.6 Levels of Data Review:

Spectrum Analytical, Inc. RI Division employs five (5) levels of data review. These are based on requirements outlined in several government and other environmental analysis programs including the U. S. Army Corps of Engineers, Air Force Center for Environmental Excellence (AFCEE), Naval Facilities Engineering Service Center (NFESC), HAZWRAP, Department of Defense

ELAP (QSM), EPA Contract Laboratory Program (SOM/ISM), as well as commercial engineering firm programs.

The data review and evaluation process is structured to insure that all data reported to customers has been thoroughly reviewed and approved using a multi-step process designed to identify and correct any error. At any step in the data evaluation and review process, the reviewer has the responsibility and authority to return any data not meeting requirements back to the previous step for re-analysis or correction. No reports are released to the client as final data without successfully passing through each step in the data evaluation and review process. The steps of the data review process are documented, generally using a checklist. Several checklists are used, depending on the type and format of analysis data being reviewed. Any data released prior to the completion of the full review process are released with the statement that the data is preliminary pending final review. The word "Preliminary" is automatically printed on the bottom of all data sheets that are generated prior to completion of data review.

The five levels of data review are detailed in SOP # 110.0028 Data Validation/Self Inspection Procedures. A Flow chart of the data review process follows in Figure 11.6-1.

11.7 Document Control:

All login sheets, Chains-of-Custody (COC) and Sample Condition Forms (SCF) and other sample transmittal documentation are generated in Sample Receiving. A red Workorder File is initiated to contain all workorder-specific hard copy documents. Samples are signed in/out of the sample receiving area by analysts. In the Prep lab, samples and all pertinent information is recorded into logbooks. Once samples are moved to the instrument lab, the transfer of extracts is documented in the electronic transfer logbook (ICOC). In the instrument lab, the analysis of extracts is recorded in the instrument run log. All analysis data, including ICAL, CAL and raw data are acquired using computer-controlled instruments, and stored on the hard drive of the computer performing data acquisition. Data are automatically copied to the company file server after acquisition. Organics analysis data are processed using Thru-Put Systems' Target software. This system creates a folder on the file server for each analysis fraction for each work order or SDG. This folder contains raw data, processed analysis results, instrument tune, initial calibration and continuing calibration results as well as a copy of the data processing method used. This allows for long-term archiving and complete reconstruction of the data at any time in the future. Organic data files are also uploaded into LIMS so reporting forms can be printed. The raw data are printed electronically and arranged with all appropriate sample-preparation and instrument run logbook page copies for technical review.

Inorganic data files are uploaded into LIMS and reporting forms are printed electronically. The original instrument data files and the processed SDG are

stored on the file server where they can later be archived by the LIMS Administrator. PDF printouts for reporting forms, instrument data output and all associated preparation logbook page copies are assembled for technical data review through a custom reporting system, Package Maker.

Spectrum RI is primarily utilizing a paperless reporting system with the exception of our EPA CLP reports which require a hard copy report.

See SOP # 110.0029, Electronic Data Management for a detailed description of data management activities used to support laboratory activities.

Following technical review and generation of the report narrative, results go into the workorder file in data reporting. The original copy or electronic pdf version (dependent on client requirements) of the report is sent to the client. Spectrum offers our clients secure access to their pdf reports and EDDs via our website eServices portal. All other information associated with the report, including data review checklists are kept in the red workorder file. The non-reported data (NRD) is scanned into the optical file database for long-term archiving. As documents are scanned into the database they are recorded for permanent storage on hard drives within the fileserver. The archived electronic data is kept for a minimum of ten (10) years or according to contract/program requirements. Prior to the use of the optical file database, hardcopy reports and NRD were shipped to an offsite storage area where they will remain for a minimum of ten (10) years. After this time, these older files will be destroyed.

11.7.1 Logbooks:

All logbooks are issued and controlled by the QA Department. Logbooks are given a unique ID that includes the mm/yy the logbook was printed. Laboratory personnel must sign for the logbook when it has been released by the QA Department. When logbooks are complete, the analyst returns them to the QA Department for archiving unless still needed for reference in the lab. A new logbook is released. The archived logbooks are stored in an on-site storage box for approximately 4-6 months and then are stored in an off-site storage facility or may remain on-site depending on storage space. Refer to SOP # 80.0040, Logbook Use, Review, and Control for more detail. In addition, refer to SOP # 110.0027, Documentation Policy and Procedures for details on Spectrum Analytical, Inc. RI Division's Logbook policies. Logbooks are archived for a minimum of ten (10) years or according to contract/program requirements.

11.7.2 Workorder/Data Files:

Spectrum Analytical, Inc. RI Division is a secured, limited access building. The doors are secured with a keypad entry system. All hard copy information pertaining to the analysis of samples is maintained and stored in a workorder file folder. This information includes all login

sheets, COC, SCF, bench sheets and printed analytical data. Electronic data are also stored by laboratory workorder number on the company file server, and in the optical file database of completed reports and NRD as mentioned in section 11.7. File folders containing any remaining workorder information are stored in an off-site storage facility or may remain on-site for a total of 10 years.

The off-site storage facility referred to in the above sections is a locked storage area. Access is limited to the Laboratory Director or his designee and request to retrieve a file will be made to this person.

In the event Spectrum Analytical, Inc. RI Division changes ownership, the maintenance, control, storage and eventual disposal at the end of the appropriate time period, of all records, including client data and QA/QC files, will transfer to the new owners.

In the event Spectrum Analytical, Inc. RI Division decides to cease operations, clients will be notified prior to the cessation of operations and their files/records will be made available to them. Within a designated time period after notification, the client will be responsible for taking custody and the future maintenance of their records. If the client determines they do not want to maintain the records, these will be disposed of properly.

11.7.3 Standard Operating Procedures (SOPs):

SOPs are prepared by the Lab Supervisor and laboratory personnel in conjunction with the QA Director. The QA Director/Staff downloads a copy of the current SOP to the network at Public on 'Bernoulli'. The SOPs can be found in Q:\QA_SOPs. In addition a .pdf file of the SOP is located in Q:\QA_PUBLIC\PDF-MITKEM SOPs. A list of the current SOPs in use at Spectrum Analytical, Inc. RI Division is given in Figure 11.7-1.

The laboratory staff revises the SOPs by making changes to the document that is then reviewed by the department supervisor only if the supervisor is not the party responsible for the revisions. Any additional changes are made at this point.

The QA Department is notified that revisions are completed. The QA Director/Staff moves the revised copy of the SOP to the QA directory, QA Safety/SOPs Needing QA Revision. The QA Director makes changes to the document to include revision number and date and title clarification, if necessary. Changes from the last revision are clearly marked using 'Track Changes' in Microsoft Word.

The QA Director prints a searchable pdf copy of the SOP. At this time, hard copies of several pages are printed for original signatures of the Laboratory or Technical Director, and the QA Director. The effective date is then added to the SOP and the signed pages are scanned and inserted into the pdf document. If an older version of the SOP exists, it is moved to its archive location. The new version will be moved into the Spectrum Analytical, Inc. RI Division Intranet SOP Database as the only version accessible by laboratory personnel. Each analyst who performs any duties related to the SOP must review the new version and enter electronically that he or she has read and understands the material there.

SOP review/revisions occur on an annual basis. The procedure for preparing, reviewing, approving, revising and distributing SOPs as well as the SOP Revision Schedule are described in SOP No. 80.0012.

Minor changes to the SOP between revision dates can be done as needed. Minor changes are recorded in the Revision Record that is a part of the master copy. Edits are clearly marked. This allows readers quick access to the changes.

11.7.4 Quality Assurance Manual:

The lab will review the QA Manual annually at a minimum. Past versions of the QA Manual are maintained and archived by the QA Director in the same manner as SOPs. Edits to the QA Manual are made by the QA Director in conjunction with the laboratory management. Spectrum Analytical, Inc. RI Division will amend the QAP and any affected SOPs within 14 days when technical changes (or any of the circumstances outlined in the USEPA SOW for SOM or ISM, Exhibit E, section 5.3.2) occur. The revised QAP with visible markups will be sent to the USEPA as per section 5.3.2.1.

11.7.5 Method Updates:

In most cases it is the laboratory's policy to implement new revisions of frequently used methods within six months of the date the method revision is promulgated or published as a final method (non-CLP methods, for CLP methods see below). The QA Director, Deputy Director for Quality Services, Technical Director and Laboratory Director make the final decision on when a method revision will be adopted by the laboratory. Additionally, if a client specifically requests or mandates that an "older" method, Spectrum Analytical, Inc. RI Division will advise the client that it is not the most recent method. If the client still insists upon the older method, the lab will comply and make a note in the narrative.

When the laboratory is in the middle of a client's project, the lab will continue using the same revision for the entire sampling event unless advised otherwise by the client. Consequently, once the laboratory has formally adopted a new method revision, both the old and new revision may be in use at the same time, depending on the project.

If a client should not specify which methods to be used, the methods employed by the laboratory shall be fully documented and validated. Additionally, the methods shall be published in a reputable technical journal or text or by a reputable technical organization or instrument manufacturer.

Revisions to USEPA CLP methods are required to be implemented within 14 days of notification when the EPA modifies the technical requirements of the statement of work, or the contract. At this same time, the QAP will be amended as necessary as noted in section 11.7.4.

Laboratory-developed methods can be used as long as they have been documented and validated by qualified personnel. In all cases the client should be notified.

Figure 11.6-1
Data Review Flow Diagram

Spectrum Analytical, Inc. RI Division
Review Process Flow Diagram

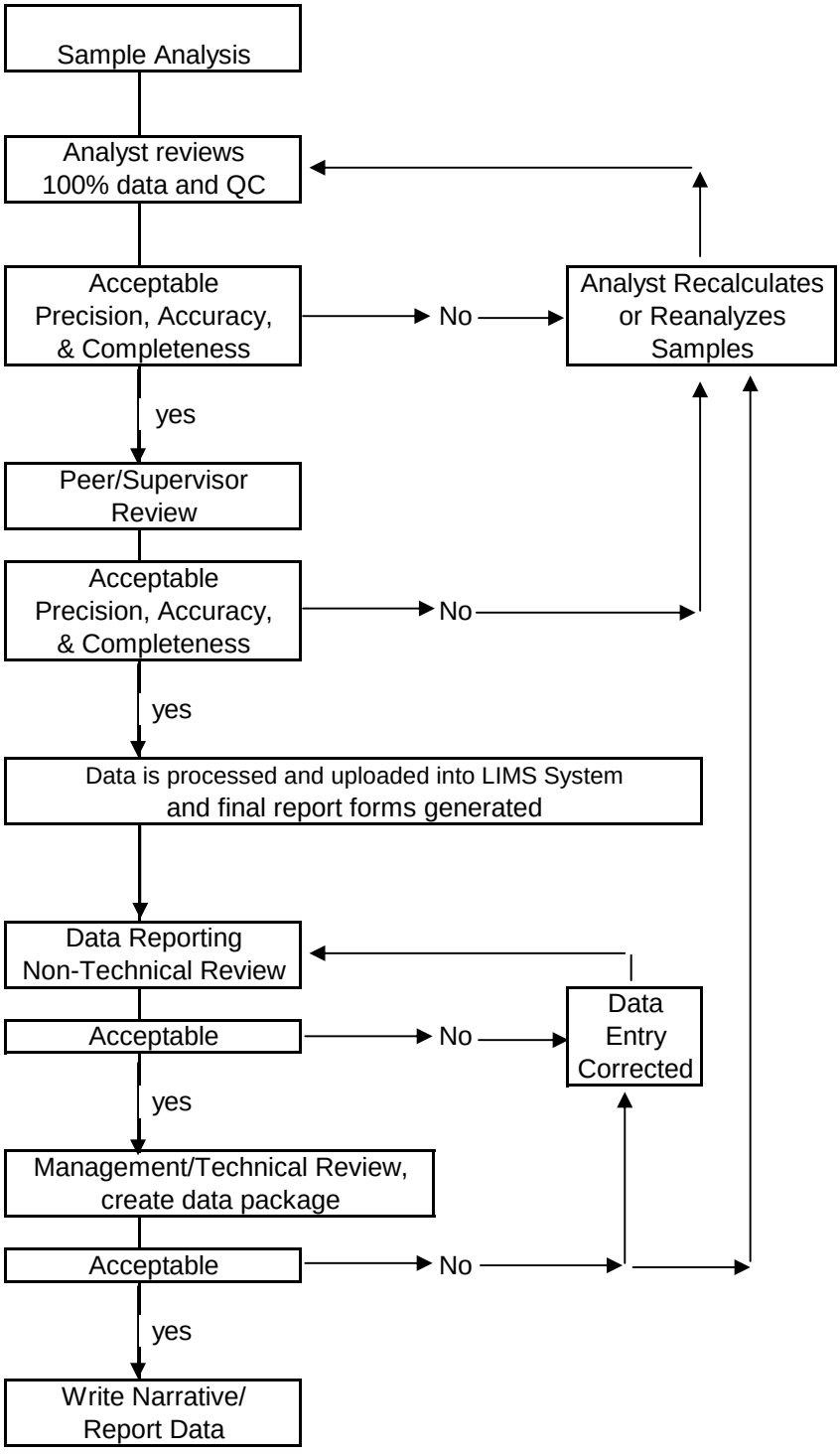


Figure 11.7-1
Standard Operating Procedures (SOPs)

**Spectrum Analytical, Inc. Rhode Island Division**

Standard Operating Procedures (SOPs)

Master List

| SOP # | Title |
|---|---|
| 10.0016 | Assembly of Inorganic CLP and CLP-type Reports |
| 10.0017 | Assembly of Organic CLP and CLP-type Reports |
| 10.0018 | Assembly of Commercial Data Reports |
| 10.0021 | Data Report Options |
| 10.0036 | EPA/SOM Organic Data PDF Bookmarking |
| 10.0037 | EPA/ISM Inorganic Data PDF Bookmarking |
| | |
| 20.0003 | Logging Workorders into Omega |
| 20.0005 | Level 2 LIMS report preparation |
| | |
| 30.0002 | Bottle order preparation |
| 30.0003 | Sample Receipt, Storage, Tracking and Disposal |
| 30.0024 | Sample and Waste Disposal |
| 30.0030 | ICOC Procedures using IntCOC program |
| | |
| 50.0004 | Glassware Cleaning - Organics |
| 50.0027 | Organic Preparation of Aqueous/Soil Samples for Chlorinated Herbicides by SW-846 Method 8151A |
| 50.0030 | SOM01.2 Sulfur Cleanup |
| 50.0031 | SW-846 Method 3665A Acid Cleanup |
| 50.0032 | Gel Permeation Chromatography by SW-846 Method 3640A |
| 50.0033 | SW-846 Method 3620B Florisil Cleanup |
| 50.0034 | SW-846 Method 3630C Silica Gel Cleanup |
| 50.0035 | Oil&Grease (HEM&SGT) by Method 1664 Revision A |
| 50.0036 | SW-846 Method 3660B Sulfur Cleanup |

**Spectrum Analytical, Inc. Rhode Island Division**

Standard Operating Procedures (SOPs)

Master List

| SOP # | Title |
|---|---|
| 50.0050 | Organic Preparation of Aqueous Samples by Continuous Liquid-Liquid (Method 3520) |
| 50.0051 | Organic Preparation of Aqueous Samples by Separatory Funnel (Method 3510) |
| 50.0052 | Organic Preparation of Soil Samples by Sonication (Method 3550) |
| 50.0053 | Organic Preparation of Soil Samples by Soxhlet (Method 3540) |
| 50.0054 | Organic Extract Filtration and Concentration Techniques |
| 50.0060 | Organic Preparation of Aqueous Samples by Continuous Liquid-Liquid for Pesticides/Aroclors for SOM01.2 |
| 50.0061 | Organic Preparation of Aqueous Samples by Separatory Funnel for Pesticides/Aroclors for SOM01.2 |
| 50.0062 | Organic Preparation of Solid Samples by Sonication for Pesticides/Aroclors for SOM01.2 by Method 3550B |
| 50.0063 | Organic Preparation of Aqueous Samples by Continuous Liquid-Liquid for Semivolatiles for SOM01.2 |
| 50.0064 | Organic Preparation of Solid Samples by Sonication for Semivolatiles for SOM01.2 |
| 50.0100 | Preparation of Soil Samples by MSE by Method 3570 |
| 50.0101 | Preparation of Soil Samples by PFE by Method 3545 |
| 50.0102 | Percent Lipid Determination in Tissue Samples |
| | |
| 60.0002 | Pesticide/PCB Analysis by EPA Method 608 |
| 60.0003 | Determination of Polychlorinated Biphenyls by Gas Chromatography/Electron Capture Detector Analysis by SW846 Method 8082A |
| 60.0006 | Determination of Pesticides by Gas Chromatography/Electron Capture Detector Analysis by SW846 Method 8081B |
| 60.0007 | EDB/DBCP by EPA Method 504.1 and SW-846 8011 |

**Spectrum Analytical, Inc. Rhode Island Division**

Standard Operating Procedures (SOPs)

Master List

| SOP # | Title |
|---|---|
| 60.0034 | Determination of Chlorinated Herbicides by Gas Chromatography/Electron Capture Detector Analysis by |
| 60.0048 | Aroclor Analysis GC/ECD by USEPA SOW SOM01.2 |
| 60.0049 | Pesticide Analysis GC/ECD by USEPA SOW SOM01.2 |
| 60.0050 | Total Petroleum Hydrocarbons by GC-FID using EPA SW-846 Methods 8015/State Methods |
| 60.0053 | PCB Congeners by SW-846 Method 8082 (MOD) |
| 60.0054 | PCB Homologs by E680 GC/MS SIMS (MOD) |
| | |
| 70.0011 | Determination of Semivolatile Organic Compounds by Gas Chromatography/Mass Spectrometry (GC/MS) Analysis by SW846 Method 8270D |
| 70.0030 | Screeening for Semivolatile Organic Analysis by Gas Chromatography/Mass Spectrometry for SOM01.2 |
| 70.0033 | SIM Analysis by GC/MS (Modified EPA Method 8270D) |
| 70.0035 | Semivolatile Organic Analysis by SIM Gas Chromatography/Mass Spectrometry for SOM01.2 |
| 70.0048 | Semivolatile Organic Analysis by Gas Chromatography/Mass Spectrometry for SOM01.2 |
| 70.0051 | Semivolatile Organics by GC/MS for Aqueous Samples by EPA Method 625 |
| | |
| 80.0001 | Standard Equivalency/Traceability |
| 80.0002 | Client Complaint Policies |
| 80.0004 | QA Data Pkg Review |
| 80.0005 | Method Detection Limit Determination |
| 80.0006 | Internal Audit Procedures |
| 80.0007 | Corrective Action Procedures |

**Spectrum Analytical, Inc. Rhode Island Division**

Standard Operating Procedures (SOPs)

Master List

| SOP # | Title |
|---|---|
| 80.0009 | Newly Implemented Methods (Demonstration of Acceptable Performance) |
| 80.0010 | Control Chart Generation and Use |
| 80.0012 | The Production of Standard Operating Procedure |
| 80.0013 | Reagent Purchasing & tracking |
| 80.0016 | Training Procedures and Tracking |
| 80.0020 | Temperature Monitoring Systems |
| 80.0030 | Labware Volume Verification |
| 80.0040 | Logbook Use, Review, and Control |
| 80.0050 | Performance Testing Procedures |
| | |
| 90.0012 | Determination of Volatile Organic Compounds by Gas Chromatography/Mass Spectrometry (GC/MS) Analysis by SW846 Method 8260C |
| 90.0035 | Low/Med Volatile OrganicsAnalysis GC/MS by USEPA SOM01.2 |
| 90.0036 | Trace Volatile OrganicsAnalysis GC/MS for USEPA SOM01.2 |
| 90.0038 | Gasoline Range Organics by GC/FID using Methods SW-846 8015 and Maine 4.2.17 |
| 90.0040 | Trace Volatile OrganicsAnalysis GC/MS using SIM for USEPA SOM01.2 |
| 90.0052 | Volatile Organics by GC/MS for Aqueous Samples by EPA Method 624 |
| 90.0060 | Methane, Ethane, and Ethene by GC/FID Method RSKSOP-175 |
| | |
| 100.0001 | Glassware Cleaning - Inorganics |
| 100.0002 | Alkalinity (by Standard Method 2320) |
| 100.0003 | Sample Preparation of Aqueous Samples by Acid Digestion ICP (3005/3010) |
| 100.0004 | Total Cyanide by Automated Colorimetric with Midi-distillation by SW846 9012B |

**Spectrum Analytical, Inc. Rhode Island Division**

Standard Operating Procedures (SOPs)

Master List

| SOP # | Title |
|---|---|
| 100.0005 | Determination of Metals and Trace Elements in Water and Waste by ICP - Atomic Emission Spectrometry by EPA Method 200.7 |
| 100.0006 | ICAP 3000XL/4300DV Operation |
| 100.0007 | Aqueous sample Prep E200.8 |
| 100.0010 | Nitrite Analysis by Standard Method 4500-NO2 B |
| 100.0011 | pH Value by Standard Methods 4500-H+ B |
| 100.0012 | Mercury Analysis in Aqueous Samples by Flow Injection Analysis System for Atomic Analysis by Method 7470A/7471B |
| 100.0013 | Total and Ortho Phosphate using Ascorbic Acid Method by Standard Method 4500-P E |
| 100.0014 | Mercury (Manual Cold Vapor Technique) by EPA Method 245.1 |
| 100.0015 | The Preparation of Waste Samples for reactive Cyanide and Sulfide; Determination of Reactive Cyanide by Automated Colorimetric Method and Reactive Sulfide by Spectrophotometric Method SW-846 Methods 7.3.3.2 and 7.3.4.2 |
| 100.0016 | Preparation of Soil Samples for Sulfide Analysis by Modified SW-846 Method 9031 |
| 100.0017 | Inorganic Analysis of Sulfates in Aqueous Samples by SM 426 C 15th Ed and SM4500 SO4 E-. |
| 100.0018 | Inorganic Analysis of Sulfides in Aqueous Samples (Methylene blue method) |
| 100.0019 | Total Dissolved Solids Dried at 180°C by Standard Method 2540 C |
| 100.0020 | Total Solids Dried at 103-105°C by Standard Method 2540 B |
| 100.0021 | Total Suspended Solids Dried at 103-105°C by Standard Method 2540 D |
| 100.0022 | TKN Distillation and Determination by Manual Spectrophotometric Analysis by Standard Method 4500-N |
| 100.0023 | Color Analysis by Visual Comparison by Modified Standard Methods 2120B |
| 100.0024 | Flashpoint Analysis by SW846 Method 1010A |
| 100.0025 | Total Organic Carbon by Methods SW-846 9060A and SM5310B |

**Spectrum Analytical, Inc. Rhode Island Division**

Standard Operating Procedures (SOPs)

Master List

| SOP # | Title |
|---|---|
| 100.0026 | Settleable Solids by Standard Method 2540 F |
| 100.0027 | Paint Filter Liquids Test by SW-846 Method 9095A |
| 100.0028 | Carbon Dioxide (CO2) and Forms of Alkalinity by Calculation by Standard Method 4500-CO2 D |
| 100.0029 | Ferrous Iron Analysis by Standard Method 3500-Fe B, Phenanthroline Method |
| 100.0030 | Phenols Analysis by EPA Method 420.1 and Standard Method 5530 B & D, Cleanup and Direct Photometric Method |
| 100.0032 | Total Volatile Solids for Solids by SM 2540 E, E160.4; Fixed and Volatile Solids Ignited at 550 C |
| 100.0033 | Total Cyanide by Auto-Colorimetric with Midi-Distillation by EPA Method 335.4 |
| 100.0053 | ISM01.3 ICP-AES Analysis |
| 100.0054 | ISM01.3 ICP-MS Analysis |
| 100.0055 | Mercury Preparation and Analysis by ISM01.3 |
| 100.0056 | Cyanide Preparation and Analysis by ISM01.3 |
| 100.0100 | Sample Preparation of Soils by Acid Digestion for ICP/MS (3050B/6020A) |
| 100.0103 | AVS and SEM |
| 100.0104 | Sample Preparation of Soils by Acid Digestion for ICP/AES (3050B/6010C) |
| 100.0106 | Chemical Oxygen Demand Determination SM5220D |
| 100.0110 | Determination of Metals in Water and Wastes by Inductively Coupled Argon Plasma Mass Spectrometry by SW846 Method 6020A |
| 100.0111 | Determination of Metals in Water and Wastes by Inductively Coupled Argon Plasma Atomic Emission Spectrometry by SW846 Method 6010C |
| 100.0112 | pH in Soil Samples by SW846 9045D/SOM1.2 |
| 100.0113 | Determination of Metals and Trace Elements in Water by ICP - MS by EPA Method 200.8 |
| 100.0121 | ICP Aqueous Preparation by ISM01.3 |

**Spectrum Analytical, Inc. Rhode Island Division**

Standard Operating Procedures (SOPs)

Master List

| SOP # | Title |
|---|---|
| 100.0122 | Prep of Soil, Wipe/Air Filter for ICP Analysis by ISM01.3 |
| 100.0201 | Ammonia Distillation & Determination SM4500-NH3 B&C |
| 100.0208 | Inorganic Analysis of Hexavalent Chromium in Soil Samples by SW846 Methods 3060A & 7196A |
| 100.0209 | Mercury Speciation…SW846 Method 3200 |
| 100.0308 | Inorganic Analysis of Hexavalent Chromium in Aqueous Samples by SM 3500 Cr +6 B |
| 100.0400 | Inorganic Anions by IC EPA 300.0 and 9056A |
| 100.0410 | TOC in Soil by Lloyd-Kahn and SW-846 9060 |
| 100.0420 | Volatile Fatty Acids by IC using EPA 300.0 (modified) |
| 100.0430 | Walkley Black TOC in Soil |
| 100.0440 | Total, Fixed and Volatile Solids in solid/semisolid samples by SM2540G |
| | |
| 110.0006 | Thermometer Calibration |
| 110.0007 | Balance Calibration |
| 110.0008 | Manual Integration of GC, IC and GC/MS Chromatograms |
| 110.0012 | Laboratory Security |
| 110.0013 | North Carolina Samples |
| 110.0021 | Bids and Proposals |
| 110.0023 | Project Management |
| 110.0025 | Toxicity Characteristic Leaching Procedure by SW846 Method 1311 |
| 110.0026 | Handling of Evidentiary Materials |
| 110.0027 | Documentation Policy and Procedures |
| 110.0028 | Data Validation-Self Inspection Procedures |
| 110.0029 | Electronic Data Management |
| 110.0031 | Synthetic Precipitation Leaching Procedure by SW-846 Method 1312 |
| 110.0032 | ASTM Leachate Procedure D3987-06 |

**Spectrum Analytical, Inc. Rhode Island Division**

Standard Operating Procedures (SOPs)

Master List

| SOP # | Title |
|---|---|
| 110.0034 | Sample Data Control for Inorganic CLP (ILM/ISM) |
| 110.0035 | Sample Data Control for Organic CLP (SOM) |
| 110.0038 | Percent Solids Determination as Required for Various SW-846 and EPA Methods |
| 110.0039 | Sub-Sampling for Soil and Solid Samples |
| 110.0040 | Instrument Maintenance |
| 110.0041 | Multiple Extraction Procedure by SW846 EPA Method 1320 |
| 110.0043 | Standard Elutriate Preparation |
| 110.0060 | Tissue Sample Preparation |

# 12.0 LABORATORY QUALITY CONTROL CHECKS

Spectrum Analytical, Inc. RI Division's analytical procedures are based on sound quality control methodology, which derives from three primary sources:

1. Specific EPA and other approved analytical methods, and

2. "Handbook for Analytical Quality Control in Water and Wastewater Laboratories" (EPA 600/4-79-019).

3. Standards for Good Laboratory Practice.

In the application of established analytical procedures Spectrum Analytical, Inc. RI Division employs, at a minimum, the QC protocols described in the references found in the Analytical Methods section of this document. Specific projects may require additional quality control measures, due to such factors as difficult sample matrices or use of innovative techniques. For those projects Spectrum Analytical, Inc. RI Division will recommend and implement, subject to client approval, QC measures to produce data of known quality.

Each of the Spectrum Analytical, Inc. RI Division laboratory departments have an individual QC program, which includes, but is not limited to, the practices described below.

## 12.1 Method Detection Limit Determination/Verification:

Method Detection Limits are developed annually for certain inorganic and many organic analyses. Per NELAC Standards, MDLs are not required where target analytes are not reported below the lowest calibration standard concentration. For these analyses, results are only reported within the calibration range, and MDLs are not appropriate or needed. The reporting limit for these compounds is the concentration of the lowest standard in the calibration. For certain inorganic analyses and most organic analyses, Spectrum Analytical, Inc. RI Division typically reports analytes below the lowest level of the calibration range, but above the MDL, as estimated and are qualified with the "J" flag. Spectrum Analytical, Inc. RI Division reports estimated values below the calibration range for those analyses where results are able to be confirmed as in dual column confirmation, or by two concurrent determinative tests such as retention time and mass spectra as in GC/MS analyses. For these analyses MDLs are determined or verified annually, depending on program requirements.

MDLs are determined for all test methods where required by specific program or state regulations. Methods analyzed for the State of Massachusetts which do not detail MDL requirements within the published method, require preparation and analysis of the MDL samples over a minimum of three days. This is believed to

better mirror real world samples and day to day variability of preparatory and analytical steps.

In addition, to address special project requirements, MDLs can be determined for those tests which are not routinely reported below calibration range. If a client requests results to be reported below the calibration range without an MDL study, this is clearly identified in the workorder narrative.

Following an MDL study, the determined limits are verified by the analysis of an MDL Verification Standard. This standard is analyzed at approximately 2 to 3 times the calculated MDL for single analyte tests or 1-4 times the calculated MDL for tests with multiple analytes. This spike concentration is also referred to as the Limit of Detection in Department of Defense Quality Systems Manual (DoD QSM). DoD QSM requires quarterly verification of the LOD. For more details refer to SOP 80.0005 Determination of Method Detection Limits.

12.2 Personnel Training:

Chemists who begin their employment at Spectrum Analytical, Inc. RI Division are to be instructed under the lab's Safety Training Program within the first month. The Safety Training Program includes laboratory basics, safety video and testing, and MSDS instruction.

Before performing any analyses, a chemist is required to read the appropriate protocols and SOPs. The chemist is required to sign off on all documents read in the electronic SOP database located on our lab Intranet.

The new analyst must become familiar with the laboratory equipment and the analytical methods, and begins a training period during which he or she works under strict supervision. Independent work is only permitted after the chemist successfully completes an accuracy and precision study.

The accuracy and precision study is also commonly referred to as a Demonstration of Capability exercise. Upon the successful completion of the Initial Demonstration of Capability exercise, the QA Department issues a Demonstration of Capability Certificate (IDOC) which is signed by both the QA Director and Laboratory Director.

Demonstration of Capability studies requires the acceptable mean recovery of 4 LCS samples for each matrix or the acceptable analysis of a blind spike sample such as a Performance evaluation sample. Acceptance limits are established by the method. It is necessary to pass the study whether for extraction and/or analysis.

Annually thereafter the employee must perform an acceptable demonstration of capability study to document continued acceptable performance in his/her

particular preparatory or analytical method specialty. This is referred to as the On-going DOC. All DOCC documentation is filed in the employee's personnel folder, which is stored in the QA Department/or in the electronic personnel folder as the system has transitioned to a paperless filing system for DOCC.

Initial and on-going personnel training include data integrity training. The 4 required elements of the data integrity system include: 1) data integrity training, 2) signed data integrity documentation, 3) in-depth, periodic monitoring of data integrity, and 4) data integrity procedure documentation.

Data integrity training topics will include the need for honesty and full disclosure in all analytical reporting, how and when to report integrity issues and what those issues could be. Employees will understand that infractions of data integrity procedures can result in an investigation that could lead to serious consequences which include immediate termination, and civil or criminal prosecution. At the start of employment all new employees read, discuss and sign a Confidentiality, Ethics and Data Integrity Agreement. Annually, an on-going integrity training session is held. An attendance sheet will be generated for every integrity session. These sheets are filed in the QA Office under "Training". Another option for the annual training session is having all staff review refresher materials online and documents their having done so. This is done within the framework of the SOP database on the lab's intranet.

Data integrity procedures are reviewed and updated annually by senior management.

Training for the EPA Statement of Work occurs according to the above requirements. In addition, analysts are required to read the CLP Statement of Work as a part of the documentation training.

12.3 Control Charts:

For organic and inorganic analyses, the recoveries of analytes in the lab control samples are plotted on control charts. These charts are used to establish control and warning limits.

12.3.1 Control limits are calculated ,compared, and/or updated at least annually from the LCS, MS/MSD, and Surrogate data points for each analyte and matrix using the following equations:

$$Average(\bar{x}) = \frac{\left[\sum_{i=1}^{n} x_i\right]}{n}$$

$$SD = \sqrt{\frac{\sum_{i=1}^{n} \left(x_i - \bar{x}\right)^2}{n-1}}$$

In which:

SD = Standard Deviation
N = number of data points

Warning Limits = Average ± 2 * SD

Control Limits = Average ± 3 * SD

12.3.2 Control limits must be approved by the QA Director and by the Laboratory Director prior to adoption by the laboratory. In the event that limits are wider than method recommended limits, the method recommended limits may be adopted and the analytical procedure will be re-evaluated and/or re-determined to identify possible causes. Additionally, in the event that control limits are tighter than 15% from the average, the lab may adopt a control limit of ±15% from the average. If in the experience of the laboratory, statistical control limits are unreasonably wide or narrow, alternative limits may be used until appropriate statistical limits are developed. Alternative limits are based on sources such as DoD QSM published guidelines, EPA limits from the specific test method or from similar methods, laboratory experience with the method or other sources.

12.3.3 Control charts are plotted in EXCEL using the LIMS system.

Data from each laboratory is uploaded into the LIMS. The compounds, recoveries, and date analyzed for each test are recorded in the system. In order for LIMS generated control limits to be valid, all data, including data not meeting existing recovery criteria, must be uploaded. A control chart is then printed for review by the QA Director and by the Lab Supervisor. Out of control situations noted on the control chart are discussed with the Supervisor or Laboratory Director by the QA Director.

An example control chart is presented as Figure 12.3-1. LCS data must be reviewed and evaluated daily against the Control Limits to establish that the system is in control.

12.3.4 The following situations constitute an out of control situation on a control chart:

- One data point above or below the Control Limit line.
- Two consecutive data points above or below the Warning Limit line.
- Six or more consecutive data points above the Average Line or six or more consecutive data points below the Average Line. This situation suggests a trend and suggests the procedure has been changed in some way (for better or worse). The cause for this trend must be investigated.

12.4 General QC Protocols:

12.4.1. Organics Laboratory:

- Trip blanks and holding blanks, when applicable, are analyzed to detect contamination during sample shipping, handling and storage.
- Method blanks, at a minimum of one in every 20 samples, are analyzed to detect contamination during analysis.
- Volatile organic method blanks are analyzed once during each analytical sequence.
- One blank spike (Laboratory Control Sample or LCS) consisting of an analytical sample of laboratory water, anhydrous sodium sulfate, or Ottawa sand with every batch of 20 or fewer samples, is analyzed to determine accuracy.
- Sample spikes and spike duplicates, as requested, are analyzed to determine accuracy and the presence of matrix effects. The Relative Percent Difference (RPD) is also determined for matrix spike/matrix spike duplicates to measure precision. The criteria followed are stated in the individual methods. For batches without a sample duplicate (for example, if insufficient sample volume is provided), a duplicate blank spike (LCSD) is performed to provide for precision measurement.

- Performance evaluation samples from EPA and state agencies are analyzed to verify continuing compliance with EPA and NELAC QA/QC standards.

- Surrogate standards are added to samples and calculations of surrogate recoveries are performed to determine matrix effect and extraction efficiency.

- Internal standards for GC/MS analysis are added to sample extracts to account for sample-to-sample variation.

- Analysis of EPA traceable standards (ICV) to verify working standard accuracy and instrument performance.

- Initial multi-level calibrations are performed to establish calibration curves.

- Instrument calibration is established or verified with every analytical sequence.

- Tuning of GC/MS systems once every 12 hours for CLP and SW-846 methods or 24 hours for methods 624/625 to method specifications is implemented for consistency in data generation.

- Quarterly analysis of LOD and/or LOQ check samples to verify low level detection and reporting limits for Department of Defense QSM programs.

- Annual Verification of MDL for NELAC/TNI.

When QC limits are not met during an analytical run, the source of the problem must be investigated. Following an evaluation of the data, those samples affected must be re-analyzed after the problem has been solved. If QC limits continue to be out of control, the instrument must be checked and/or a service call made and/or further corrective action implemented.

12.4.2. Inorganic Laboratory:

- Trip blanks are analyzed when applicable, to detect contamination during sample shipping, handling and storage.

- Method blanks are analyzed at a minimum of one every 20 samples, to detect contamination during analysis.

- One matrix spike of an analytical sample or laboratory water or soil is made and spike recoveries are calculated with every batch up to 20 samples to determine accuracy. Duplicate samples are analyzed and the RPD between the sample and duplicate is calculated for every batch up to 20 samples. If insufficient volume of sample is received, a note is made in the appropriate preparation logbook.

- Performance evaluation samples from EPA and state agencies are analyzed to verify continuing compliance with EPA and NELAC QA/QC standards.

- Metals analysis instruments are calibrated for every analytical run.

- Analysis of EPA traceable standards (ICV) to verify working standard accuracy and instrument performance.

- QC/LCS checks samples are analyzed during every analytical batch of up to20 samples in order to document accuracy.

- Quarterly analysis of LOD and LOQ check samples to verify low level detection and reporting limits for Department of Defense QSM programs.

- Annual Verification of MDL for NELAC/TNI.

When QC limits are not met during an analytical run, the source of the problem must be investigated. Following an evaluation of the data, those samples affected must be re-analyzed after the problem has been solved. If QC limits continue to be out of control, the instrument must be checked and/or a service call made and/or further corrective action implemented.

12.5. Lab Pure Water used for method blanks and dilutions:

Spectrum Analytical, Inc. RI Division uses several systems to generate analyte-free water for use in the laboratory. These systems generate high quality, analyte free water dedicated to the needs of specific analyses.

12.5.1. For inorganic analyses the wet chemistry and metals labs use a US Filter mixed-bed deionization system followed by particle and carbon filters. This is followed by a polishing system using Barnstead E-Pure cartridges optimized for removal of inorganic constituents. Purity is monitored using an in-line electrical resistivity meter with integral cell. Finished Inorganic reagent water is tested for conductance on a routine basis (at least annually), through the use of an external conductivity meter.

12.5.2. For organic analyses, the extractable organics laboratory uses a Barnstead E-Pure system optimized for removal of organic constituents. As organic contaminants are not measured by a resistivity meter, this is not a relied-upon method to monitor the quality of organic analyte-free water. Instead, laboratory method blanks are used, typically several per working day, to monitor the acceptability of the water for its intended use. Any analyte detected above (half of) the reporting limit is investigated. If this can be traced to the water purification system as its source, maintenance is performed on the water purification system. The volatile organics laboratory uses a Whirlpool Model WHER25 Reverse Osmosis Drinking water system to provide analyte free water.

Figure 12.3-1
Example Control Chart

## Test Code: SW8081_W Analyte: 4,4´-DDD

| SampType | Sample ID | Analysis Date | Batch ID | Low Limit | High Limit | % Recovery |
|---|---|---|---|---|---|---|
| LCSD | LCSD-65227 | 3/23/2012 | 65227 | 25 | 150 | 94.0 |
| LCS | LCS-65227 | 3/23/2012 | 65227 | 25 | 150 | 88.6 |
| LCS | LCS-65354 | 4/3/2012 | 65354 | 25 | 150 | 93.9 |
| LCS | LCS-65320 | 4/3/2012 | 65320 | 25 | 150 | 91.4 |
| LCSD | LCSD-65320 | 4/3/2012 | 65320 | 25 | 150 | 84.5 |
| LCS | LCS-65743 | 4/26/2012 | 65743 | 25 | 150 | 92.8 |
| LCS | LCS-65925 | 5/7/2012 | 65925 | 25 | 150 | 91.6 |
| LCS | LCS-66030 | 5/14/2012 | 66030 | 25 | 150 | 75.4 |
| LCS | LCS-66116 | 5/15/2012 | 66116 | 25 | 150 | 93.2 |
| LCSD | LCSD-66116 | 5/15/2012 | 66116 | 25 | 150 | 92.7 |
| LCS | LCS-66132 | 5/16/2012 | 66132 | 25 | 150 | 92.8 |
| LCS | LCS-66631 | 6/12/2012 | 66631 | 25 | 150 | 94.4 |
| LCSD | LCSD-66631 | 6/12/2012 | 66631 | 25 | 150 | 99.1 |
| LCS | LCS-66758 | 6/18/2012 | 66758 | 25 | 150 | 90.8 |
| LCSD | LCSD-66767 | 6/18/2012 | 66767 | 25 | 150 | 82.5 |
| LCSD | LCSD-66758 | 6/18/2012 | 66758 | 25 | 150 | 79.9 |
| LCS | LCS-66767 | 6/18/2012 | 66767 | 25 | 150 | 89.5 |
| LCS | LCS-66817 | 6/19/2012 | 66817 | 25 | 150 | 92.8 |
| LCSD | LCSD-66817 | 6/19/2012 | 66817 | 25 | 150 | 92.6 |
| LCS | LCS-66801 | 6/20/2012 | 66801 | 25 | 150 | 99.0 |
| LCSD | LCSD-66801 | 6/20/2012 | 66801 | 25 | 150 | 98.6 |
| LCS | LCS-66899 | 6/28/2012 | 66899 | 25 | 150 | 80.5 |
| LCSD | LCSD-66899 | 6/28/2012 | 66899 | 25 | 150 | 83.1 |
| LCSD | LCSD-67208 | 7/19/2012 | 67208 | 25 | 150 | 84.8 |
| LCS | LCS-67208 | 7/20/2012 | 67208 | 25 | 150 | 89.5 |
| LCS | LCS-67206 | 7/20/2012 | 67206 | 25 | 150 | 80.6 |
| LCS | LCS-68027 | 9/13/2012 | 68027 | 25 | 150 | 96.9 |
| LCS | LCS-68082 | 9/13/2012 | 68082 | 25 | 150 | 100.3 |

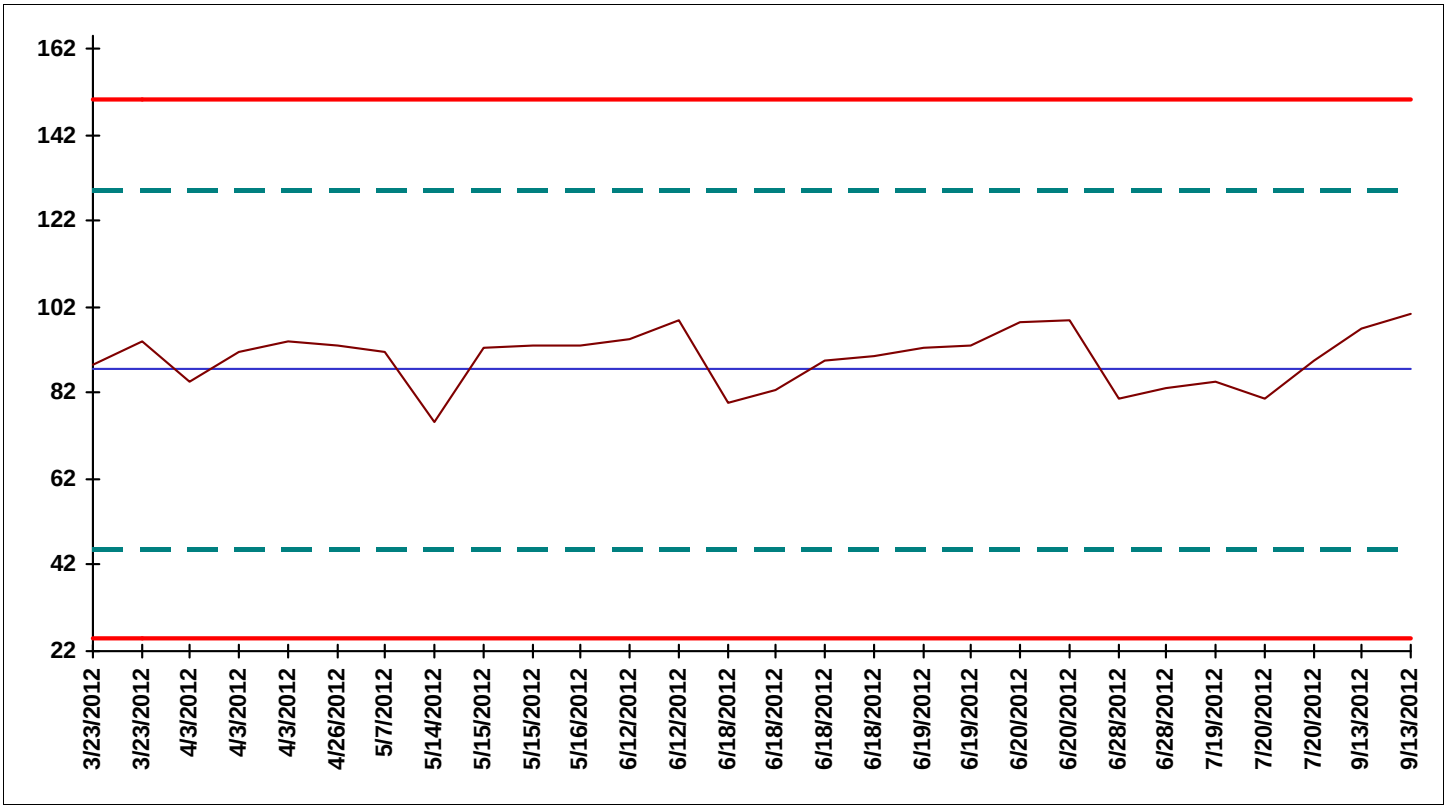
**Spectrum Analytical, Inc. Featuring Hanibal Technology**

# REC QUALITY CONTROL CHART

**Date:** ***24-Sep-12***

**Test Code: SW8081_W Analyte: 4,4´-DDD**

| SampType | Sample ID | Analysis Date | Batch ID | Low Limit | High Limit | % Recovery |
|---|---|---|---|---|---|---|



# 13.0 QUALITY ASSURANCE SYSTEMS AUDITS, PERFORMANCE AUDITS AND FREQUENCIES, PEER REVIEW

The Spectrum Analytical, Inc. RI Division Quality Assurance staff performs routine internal audits of the laboratory. The frequency of such audits depends on the workload in house but is done annually, at a minimum. These audits entail reviewing laboratory logbooks and all appropriate operations to ensure that all laboratory systems including sample control, analytical procedures, data generation and documentation meet contractual requirements and comply with good laboratory practices.

## 13.1 System Audits:

The QA Director audits each individual laboratory annually in order to detect any sample flow, analytical or documentation problems and to ensure adherence to good laboratory practices as described in Spectrum Analytical, Inc. RI Division's Standard Operating Procedures and Quality Assurance Plan. A checklist used in an internal systems audit is presented in Figure 13.1-1.

Areas covered by the internal audit include logbook documentation and review, standard traceability, standard storage and expiration dates, method criteria adherence, instrument maintenance records, SOP review, and knowledge of the analysts. Often, deficiencies that have been noted during "outside" audits will also be reviewed.

Upon the completion of the internal audit, a formal audit report is presented to the laboratory supervisor who is given a specific timeframe to respond in writing to the deficiencies. The QA Department will do a follow up audit to check that at least the major deficiencies have been corrected. The follow-up audit occurs within 30-45 days from the date of the audit response.

## 13.2 Performance Audits:

Spectrum Analytical, Inc. RI Division participates in external Performance Test (PT) studies under the National Environmental Accreditation Program (NELAP) through the New Jersey Department of Environmental Protection (Primary Accreditation Authority). The QA department administers the Performance Evaluation Samples for Wastewater/Solid Waste (WW/SHW). Additionally, performance samples are administered for test methods not certified through the New Jersey program, such as specific state methods. PT samples are handled (i.e., managed, analyzed, and reported) in the same manner as real environmental samples utilizing the same staff, methods as used for routine analysis of that analyte, procedures, equipment, facilities, and frequency of analysis. When analyzing a PT sample, a laboratory shall employ the same calibration, laboratory quality control and acceptance criteria, sequence of analytical steps, number of replicates and other procedures as used when analyzing routine samples. PT

samples are reported electronically via the vendor's website (ERA, RTC…), and results are sent directly to all applicable state or agency certification programs.

Clients also send performance evaluation samples (PES) to Spectrum Analytical, Inc. RI Division as part of their own quality control program. Spectrum Analytical, Inc. RI Division is blind to the true values of the PES. The USEPA CLP program provides quarterly blind (QB) studies for all tests and matrices. The lab is informed of their performance after the study has been graded through an Individual Laboratory Summary Report. When results in any section are less than 90.0%, the lab is required to complete a formal corrective action report to the EPA.

Spectrum Analytical, Inc. RI Division also participates in external electronic data QA monitoring audits and data package audits through the USEPA CLP program. On request, the Spectrum Analytical, Inc. RI Division CLP Project Manager submits instrument data tapes and all applicable documentation for tape audits, including a copy of the data package. All original documentation generated during sample analyses may be requested. The results of the tape audit are sent to Spectrum Analytical, Inc. RI Division in report format in the same manner as an on-site audit (see below). A formal response is required.

Several times a year outside agencies (federal, state, or private) may schedule an audit at Spectrum Analytical, Inc. RI Division in order to check the laboratory's processes. Most often these audits begin and end with a meeting between auditors and laboratory management. Each individual laboratory is examined. The QA Director and/or Senior Management Staff are most likely to remain with the auditors at all times during the audit.

Sometime after the audit, the lab receives a formal audit report to which it must respond. The audit report is initially reviewed by the QA Director who copies and distributes the report to each laboratory supervisor. The supervisors are required to respond in writing to the findings that pertain to his or her department. The QA Officer compiles the formal response that could be tweaked several times before the auditing authority accepts the results. A specific timeframe is set by the individual agency involved.

The QA Officer then sends a memo to each supervisor to detail what needs to be done in each department within a specific timeframe. The QA Department then follows up with the labs to ensure procedures have been modified and the corrective actions are in place.

Internally, performance is monitored on a daily basis at Spectrum Analytical, Inc. RI Division through the use of surrogate and internal standards, and LCS and MS/MSD samples. Check samples from independent commercial sources are employed routinely in each of the Spectrum Analytical, Inc. RI Division laboratory departments and ensure continuing high-level performance. The QA

Director may distribute internal blind PE samples to each laboratory department as needed. These blind PE samples can also be used to show on-going analyst proficiency in lieu of 4 LCS studies.

13.3. Peer Review:

Peer review is used as a vital quality control tool within all areas of the laboratory, and at all levels. Peer review allows defects in the acquisition, evaluation and reporting of sample data to be identified before moving on to the next step in the process of preparing and analyzing samples. Several steps of peer review are included at Spectrum Analytical, Inc. RI Division to prevent and catch mistakes, whether caused by human error or a system malfunction. As soon as samples enter the laboratory they are logged into the LIMS system and given unique sample identifiers that correspond to the client's IDs listed on the chain of custody. The individual jars or bottles are labeled and the technician employs a peer review of this labeling process. A project manager or peer technician visually inspects each jar or bottle for proper identification and matching lab/client IDs. Once the samples are sent into the labs for test preparation, they again undergo peer review as they are set up for extraction, digestion or distillation... This time the samples are inspected to confirm the samples at the bench match the identifications written into the lab preparation logbooks. Once the concentrated extract, digestate or distillate is ready for analysis and set up on the analytical instrument, an analyst will perform another peer review of the autosampler set up to avoid any misplacements of sample vials. In some lab areas this review may occur after instrument analysis, to verify all sample data were acquired electronically. Every analytical instrument sequence (GC/ECD, GC/FID, GC/MS, ICP/MS, ICP/AES, CVAA, FIA, IC) undergoes a technical peer review by a qualified analyst to verify positive and false positive results as well as manual integrations. Data reports are also reviewed at length according to the 5 level review processes described in Section 11 of the QAP as well as in SOP No. 110.0028 Data Validation/Self Inspection Procedures. At each point in the process, the peer review is documented.

Figure 13.1-1
QA Systems Audit Checklist

Quality Assurance Department
Spectrum Analytical, Inc. RI Division
.

Quality Review of Laboratory Department

Auditor:
Date:

**Purpose**

The Quality Review is a necessary tool to assess a department's quality and service functions. Each department will undergo a review of their process and procedures to evaluate their needs and areas of possible improvement. Each department will be tracked for quality, safety, compliance, reoccurring errors and process improvement.

**Process**

Each department will be broken down into several categories or areas of review. Each category will be reviewed and assessed for compliance. The categories will include at a minimum:

Personnel Training and Knowledge
Equipment
SOP Updates and Review
Logbook Review and Control
Chemicals/Standard Storage and Preparation
Sample Procedures and Method Compliance
QA/QC Procedures
Corrective Actions in process

Each category will be reviewed and a listing of any deficiency or findings will be documented for response and correction. The department Supervisor (s) will be required to respond to each deficiency or finding within 30 days of receipt of this report. All deficiencies or findings must have its correction(s) documented. For example, logbook deficiencies will require a photocopy of the correction(s). All other responses will require a written response or adequate explanation. Deficiencies will be tracked for reoccurrence. All documentation should be forward to the QA department for evaluation. A follow up audit may be scheduled.

**Findings:**

**Personnel Training and Knowledge**

.

**Equipment**

**SOP Updates and Review**

**Logbook Review and Control**

**Chemicals/Standard Storage and Preparation**

**Sample Procedures and Method Compliance**

**QA/QC Procedures**

**Corrective Actions in process**

Items marked with an* asterisk will require a written response by the lab supervisor or his designee to the QA Dept. This response must be submitted to the QA Department by *mm/dd/yyyy*. The response can be entered directly into this document in a different font color. Please note date that the CA was completed.

Auditor _____ Date _____

# 14.0 PREVENTIVE MAINTENANCE

Preventive maintenance is a routine practice at Spectrum Analytical, Inc. RI Division for all instrumentation. Scheduled preventive maintenance minimizes instrument downtime and subsequent interruption of analysis.

Only those equipment items meeting or exceeding applicable performance requirements are used for data collection. This includes items such as laboratory balances as well as major analytical instruments such as ICPs, ICP/MS, GCs and GC/MSs. All major instrumentation and equipment, as well as backup alternatives, are listed in Appendix A. Spectrum Analytical, Inc. RI Division SOP No. 110.0040, Instrument Maintenance, describes routine maintenance in detail. Individual analytical standard operating procedures describe maintenance as well (See Figure 11.7-1 for SOP listing).
When new software is purchased or developed, it is loaded onto one workstation with copies of data that have been previously processed using older software, and known to be correct. The data is then reprocessed using the new software and then the new results are compared to the original results for defects. If the software was purchased and found to contain a defect, the vendor is contacted and a solution and/or patch are requested. If the software was developed in-house, the problems are identified and corrected. This process is applicable to all software including enhancements made to customize the LIMS and network servers.

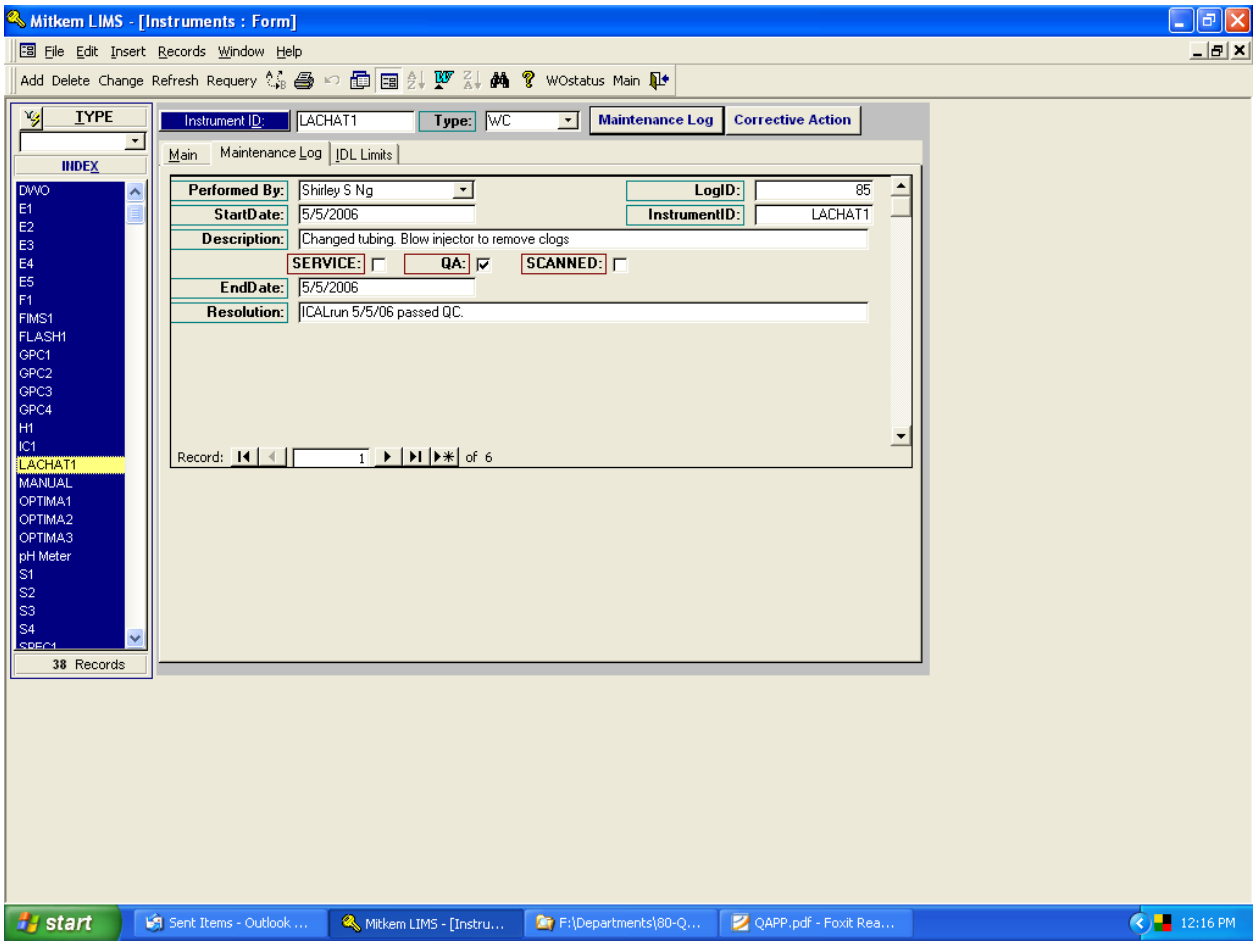
Spectrum Analytical, Inc. RI Division's laboratory personnel are familiar with the routine and non-routine maintenance requirements of the instruments they operate. This familiarity is based on education, hands-on experience and manufacturer's training courses. As needed, major equipment may under-go extensive maintenance or service by a contracted technician.

Instrument maintenance logs are kept for each instrument in the LIMS (figure 14-1). All employees have password protected access to the LIMS. The person performing the maintenance is required to provide the following information in the online log:

- Equipment identifier
- The inspection, maintenance, calibration or corrective action(s) performed.
- The trigger(s) for the maintenance action(s)
- The identity of the person(s) performing the maintenance
- The date on which the work was performed
- The need for a service call
- The condition of the equipment upon completion of the work (may include resolution of problems, date and type of ICAL run or other method of determining that the system is in good working order), and
- The presence of any scanned paperwork associated to the maintenance

Spectrum Analytical, Inc. RI Division maintains an inventory of replacement parts required for preventive maintenance and spare parts that often need replacement, such as filaments for GC/MS systems and the more mundane electrical fuses and GC column ferrules. To control cost, the appropriate supervisor shall decide the types and numbers of spare parts kept on hand for each equipment item.

Figure 14-1




Example of Instrument Maintenance Log

# 15.0 SPECIFIC ROUTINE PROCEDURES USED TO ASSESS DATA PRECISION, ACCURACY, COMPLETENESS, METHODS DETECTION LIMIT AND LINEAR DYNAMIC RANGE

These mathematical equations represent the means of calculating analytical figures of merit on a routine basis at Spectrum Analytical, Inc. RI Division. However, they may be supplanted with other calculations if requested by the client. Precision, accuracy and completeness are also discussed in Section 6.

## 15.1 Precision:

Precision is frequently determined by the comparison of replicates, where replicates result from an original sample that has been split for identical analyses. Standard deviations, *s*, of a sample are commonly used in estimating precision.

Sample standard deviation, *s*:

$$s = \sqrt{\frac{1}{n-1}\sum_{i=1}^{n}\left(x_i - \bar{x}\right)^2}$$

where a quantity, $x_i$ (e.g. a concentration), is measured *n* times with a mean, $\bar{x}$ .

The relative standard deviation, *RSD* (or sample coefficient of variation, *CV*), which expresses standard deviation as a percentage of the mean, is generally useful in the comparison of three or more replicates (although it may be applied in the case of n = 2).

$$\%RSD = 100\ (s\ /\ \bar{x}\ )$$

or

$$CV = 100\ (s\ /\ \bar{x}\ )$$

In which: *RSD* = relative standard deviation, or
*CV* = coefficient of variation
*s* = standard deviation
$\bar{x}$ = mean

For duplicates (samples that result when an original sample have been split into two for identical analyses), the relative percent difference (*RPD*) between the two samples may be used to estimate precision.

$$RPD = \frac{2(D_1 - D_2)}{(D_1 + D_2)} \times 100\%$$

In which: $D_1$ = first sample value
$D_2$ = second sample value (duplicate)

15.2 Accuracy:

The determination of accuracy of a measurement requires knowledge of the true or accepted value for the signal being measured. Accuracy may be calculated in terms of bias as follows:

$$Bias = X - T$$

$$\%Bias = 100\frac{(X - T)}{T}$$

In which: $X$ = average observed value of measurement
$T$ = "true" value

Accuracy also may be calculated in terms of the recoveries of analytes in spiked samples:

$$\%\,\mathrm{Recovery}(\%R) = 100 \times \frac{(SSR - SR)}{SA}$$

where: $SSR$ = spikes sample result
$SR$ = sample result
$SA$ = spike added

15.3 Completeness:

Determine whether a database is complete or incomplete may be quite difficult. To be considered complete, the data set must contain all QC check analyses verifying precision and accuracy for the analytical protocol. Less obvious is whether the data are sufficient to achieve the goals of the project. All data are reviewed in terms of goals in order to determine if the data set is sufficient.

Where possible, the percent completeness for each set of samples is calculated as follows:

$$\%\text{Completeness} = \frac{\text{valid data obtained}}{\text{total data planned}} \times 100$$

15.4 Method Detection Limit:

The method detection limit (MDL) is the minimum concentration of a substance that can be measured and reported with 99% confidence that the analyte concentration is not zero. It is computed as follows from data obtained by repeatedly determining an analyte in a given sample matrix:

1. Analyze at least seven samples of a homogeneous matrix spike that contains the analyte(s) of interest at concentrations of three to five times the expected MDL. The entire sample preparation and analysis protocol must be applied in each analysis; simply preparing one sample and repeating a measurement three or more times on the sample in not acceptable.

2. Upload the acceptable data into LIMS.

3. The LIMS will compute the standard deviation of the results for each analyte using the following equation:

$$MDL = t_{(n-1,\ \alpha=0.99)}\ (s)$$

Where *t* is the one-sided student's *t* value appropriate for the number of samples analyzed, *n*; $\alpha$ is the statistical confidence level; and *s* is the standard deviation.

The one-sided *t*-values are presented below:

| Number of samples | *t*-value |
|---|---|
| 7 | 3.14 |
| 8 | 2.996 |
| 9 | 2.90 |
| 10 | 2.82 |

4. The MDL is then checked against 40CFR136 requirements by the QA Department. If the MDL is acceptable then it is uploaded into the LIMS by either the QA Department or LIMS Administrator.

5. Immediately following the determination of the MDL, MDL check samples are analyzed at a concentration approximately equal to 2-3 x the new MDL for SW846 tests. The analyte of interest must be detected at this concentration, or the raising the MDL may be required. Once the MDL check is acceptable, the detection limit (DL) has been established.

6. An elevated MDL can be uploaded if necessary into the LIMS as long as documentation is available to show that the applicable method can produce an MDL at least that low. This can commonly occur for ICP

analysis in which extremely low MDLs can cause method compliance issues. When appropriate, the MDL study may be prepared and analyzed over several days to increase the variability of the preparation and/or analytical steps.

7. More detail on MDLs can be found in SOP 80.0005 Method Detection Limit Determination.

15.5 Linear Dynamic Range:

The linear dynamic range is the concentration range over which the instrument response is linear. It is determined by analyzing a series of standard solutions that extends beyond the non-linear calibration region at both the low and high extremes, and selecting that range of standards which demonstrates a linear relationship between instrument response and concentration.

For ICP analysis, the linear dynamic range is determined by analyzing each metal at 3 different concentrations. The concentration which produces results within a 10% error is determined to be the linear dynamic range. This procedure must be performed per individual method requirements.

ILM5.4 requires the analysis of the linear dynamic range be determined quarterly, with a 5 % error.

# 16.0 CORRECTIVE ACTION

An essential element of the QA Program, Corrective Action provides systematic, active measures taken in the resolution of problems and the restoration of analytical systems to their proper functioning.

Corrective actions for laboratory problems are described in Spectrum Analytical, Inc. RI Division's laboratory standard operating procedures (SOP). Personal experience often is most valuable in alerting the bench scientist to questionable results or the malfunctioning of equipment. Specific QC procedures are designed to help the analyst determine the need for corrective actions (see Section 11, Data Reduction, Validation and Reporting). Corrective actions taken by scientists in the laboratory help avoid the collection of poor quality data. The lab's corrective action program divides these issues into routine and non-routine corrective actions as described below.

Routine Corrective Action – A routine corrective action is taken when the out-of-control event encountered is one that is detected at the appropriate level in the QA process. Routine corrective actions are defined in the analytical SOP with specific steps to be taken as corrective action (i.e., low surrogate recovery, continuing calibration verifications, project specific protocols that do not meet acceptance criteria, etc.) Routine corrective actions must be documented as described in the analytical SOP, but do not require further documentation in the corrective action logbook. Examples of routine corrective action situations: surrogate/surrogates out, LCS out, CCV out, ICV out, IS area/areas out, typographical errors, random blank contamination, or false positive hit/spectral ID match corrected during data review.

Non-Routine Corrective Action – A non-routine corrective action is taken when the out-of-control event encountered is not typical for the method. For example, QC failures that passes through the final review to the client, procedural errors – not following the SOP, or a situation not being detected by normal QA procedures that could adversely impact the accuracy, precision, etc. of a result. Non-routine corrective actions must be documented in the Corrective Action Request (CAR) system, located within the LIMS. The analyst, using his/her own judgement, may deem any corrective action situation non-routine and formally document it in a CAR. When in doubt about a corrective action, the analysts are instructed to err on the side of formal CAR documentation. Examples of non-routine corrective action situations include: bad standard, expired standard mix being used, incorrect equation, "client-detected" problems, not following SOP protocols, using bad or contaminated lot of chemical/reagent/solvent, deciding to release data not conforming to SOP requirements, compound retention time outside of range, or improper library spectrum that leads to re-occurring mis-identification of compounds.

The essential steps in Spectrum Analytical, Inc. RI Division's corrective action system are:

1. Identify and define the problem.

2. Assign responsibility for investigating the problem. Usually this individual is the department supervisor.

3. Investigate and determine the root cause of the problem.

4. Determine a corrective action to eliminate the problem and prevent recurrence. Any changes that result from the corrective action investigation must be documented.

5. Assign and accept responsibility for implementing the corrective action.

6. Establish effectiveness of the corrective action and implement it.

7. Verify that the corrective action has eliminated the problem.

8. Both the laboratory and the QA Department need to monitor the corrective action to ensure it is effective.

9. Any corrective actions that cast doubt on the laboratory's compliance with its own policies and procedures may require an internal audit by the QA Department.

This scheme is generally accomplished through the use of Corrective Action Report Forms available to each of the laboratory areas within the LIMS system. Use of this report notifies the QA Department of a potential problem as described in SOP No. 80.0007. The QA Director initiates the corrective action by relating the problem to the appropriate laboratory managers and/or project managers who then investigate or assign responsibility for investigating the problem and determine its cause. Once determined, the QA Director will approve appropriate corrective action. Its implementation is later verified through an internal laboratory audit. Once the QA Director feels the system has returned to control, s/he will finalize the CAR using a password protected QA step.

Information contained on corrective action reports is kept confidential within Spectrum Analytical, Inc. RI Division and is generally limited to the individuals involved. Severe problems and difficulties may warrant special reports to the President of Spectrum Analytical Inc., who will ensure that the appropriate corrective actions are taken.

Nonconformance:

Any breech of standard protocols is a nonconformance item that is documented on the Corrective Action Request Form and management informed immediately. The following are nonconformance items:

1. Sample holding time exceeded.

2. Hoods, Class “1” weights, NIST Thermometers, balances, automatic pipettes, being used but not certified.

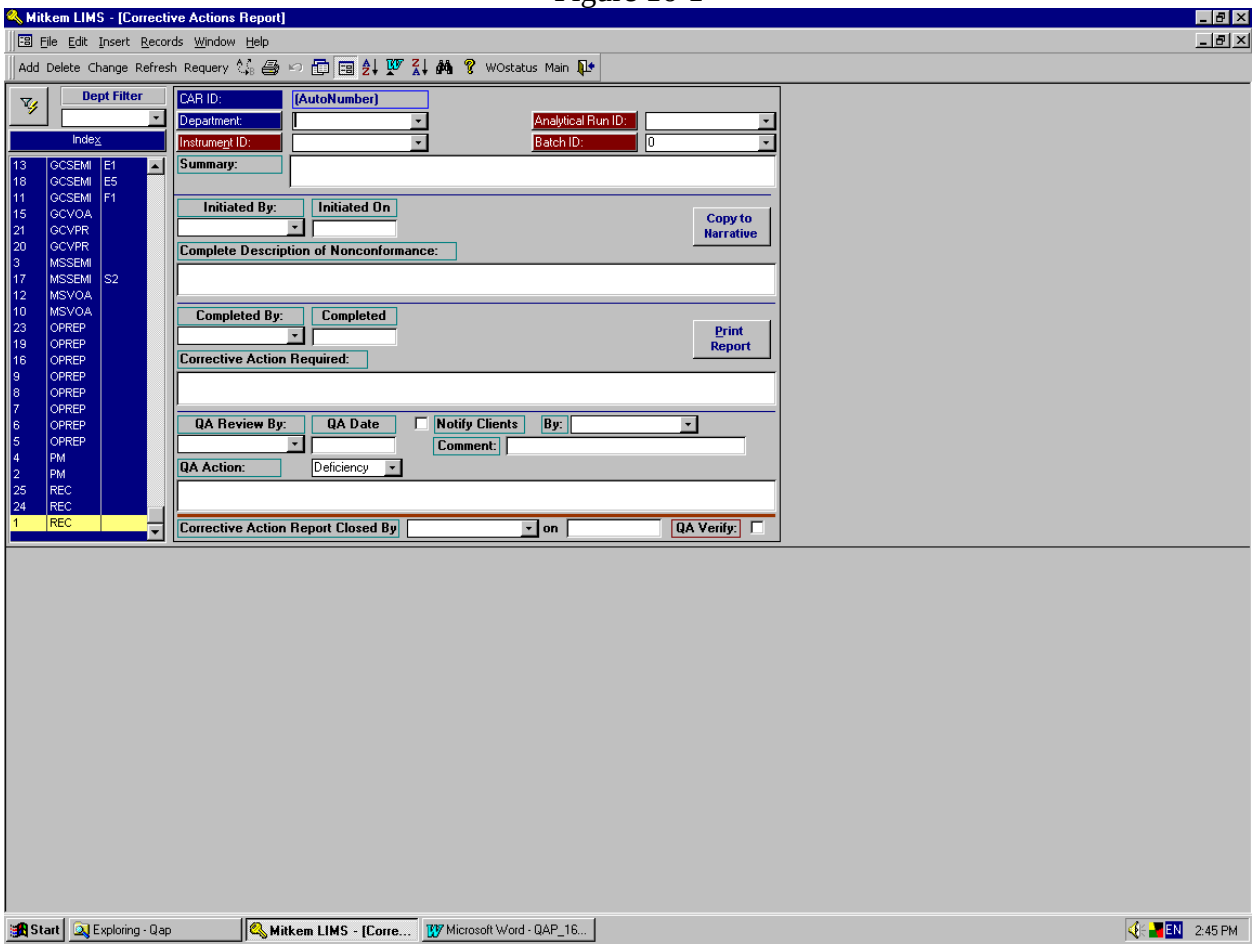
3. Expired standards being used.

4. Manual integration being misrepresented.

16.1 Client Complaints:

Spectrum Analytical, Inc. RI Division ensures client complaints are dealt with quickly and completely. The policies are stated in the laboratory Client Complaint Standard Operating procedure (SOP No. 80.0002).

Figure 16-1




Quality Assurance Corrective Action Request Form

# 17.0 QUALITY ASSURANCE REPORTS TO MANAGEMENT

The Spectrum Analytical, Inc. RI Division Quality Assurance Director submits a QA report annually to upper management. The report should be completed and submitted no later than the 15th of July in any calendar year.

The report contains detailed laboratory information and QA activities during the previous twelve months. Items to include are the status of internal and external audits, client complaints, quality control activities, resources and staffing. See the following pages for the report format.

Management will review the QA report and respond to outstanding issues. Management will add a review of the suitability of policies and procedures, and any other relevant issues**.** The response report is due within 30 days of the QA Report receipt.

A copy of the report is kept on file in the QA department.

In case of a severe problem or difficulty, a special report is prepared by the QA Director and submitted immediately to management.

Figure 17-1

**SPECTRUM ANALYTICAL, INC. RI DIVISION**
**Annual Quality Assurance Report to Management**

1. Status of Internal Audits.

2. Status of External Audits.

3. Identification of Quality Control issues in the laboratory.

4. Discussion of corrective action issues.

5. Proficiency Testing.

6. Changes in volume and type of work undertaken.

7. Client Feedback.

8. Reports from management and supervisory personnel.

# 18.0 SAFETY

Spectrum Analytical, Inc. RI Division maintains safety through a program managed by the Safety Officer and the Safety Committee. Responsibilities include many activities needed to comply with the Right-to-Know Laws.

- Training seminars with information on OSHA safety instruction for new employees.
- Introductory training to include location of fire extinguishers, first aid supplies, etc.
- Health and Safety manual review when hired.
- Annual Health and Safety Manual review and revision as needed.
- Monthly Safety Committee meetings.
- Centralized MSDS information.
- Maps with safety equipment and all exits noted.
- Posted safety rules.

If a chemical spill occurs, proper actions are described in Spectrum Analytical, Inc. RI Division's Contingency Plan. Additionally, the local fire department (North Kingstown) and hospital (Kent County) also have a copy in case a need arises. Each new hire is required to read the Contingency Plan and sign off on this. An annual meeting is held as a refresher for all employees. A copy of the Contingency Plan is located on the company Intranet and is available to all personnel.

Emergency equipment, such as spill control kits, fire extinguishers and fire blankets are located throughout the laboratory areas. The Contingency Plan has instructions for evacuation, notification of emergency authorities and regulatory personnel in the event of a chemical accident.

# 19.0 WASTE MANAGEMENT

## 19.1 Pollution Prevention

The waste management option of choice is to prevent pollution by minimizing the amount or types of chemical wastes that are generated. Spectrum Analytical, Inc. RI Division's ability to minimize waste generation is limited by the chemical analysis techniques that are required by the EPA or other authors of test methods. As new test methods are utilized in the laboratory, the type and volume of chemical waste generated by the new test is considered. Analysts and Supervisors are encouraged to look for ways to reduce the amount of chemical waste, or the type of chemical waste generated during the testing process; HOWEVER, no method is allowed to be modified without discussion among the Laboratory and/or Technical Director, QA Director and other management personnel to determine the affect of the change on the resulting data.

## 19.2. Waste Management

Spectrum Analytical, Inc. RI Division has identified and routinely disposes of chemical wastes in several hazardous waste streams. In general these are acids, caustics, solvent wastes and various laboratory waste solids. No laboratory chemical waste is disposed in the trash or dumped down the drain. All remaining sample volume following testing, and after contract-required disposal date has past, are disposed in one of these waste streams. These wastes are fully described in Spectrum Analytical Inc., RI Division's Contingency/Waste Management Plan and in the lab's Profile Log. New England Disposal Technologies is Spectrum Analytical, Inc. RI Division's waste hauler. Other hazardous wastes are identified and properly disposed according to these documents.

Continued compliance is monitored monthly by an outside consultant to ensure all RI DEM regulations are met. Key personnel attend an annual RCRA Facility Training, which focuses on the requirements for hazardous waste disposal and its proper documentation.

## 20.0 DEFINITIONS, ACRONYMS, ABBREVIATIONS:

ACCURACY: The closeness of agreement between an observed value and an accepted reference value.

ALIQUOT: A measured portion of a field sample, standard, or solution taken for sample preparation and/or analysis.

ANALYTICAL SERVICES BRANCH (ASB): The division of United States Environmental Protection Agency's (USEPA) Office of Superfund Remediation and Technology Innovation (OSRTI) responsible for the overall management of the Contract Laboratory Program (CLP).

ASTM: American Society for Testing and Materials, a developer and provider of voluntary consensus standards.

BATCH: A group of samples of the same matrix that are processed as a unit at the same time in the same location using the same method.. Unless defined differently by a specific analytical method (such as Oil & Grease by Method 1664), the maximum batch size is 20 samples.

BIAS: The deviation due to analytical or matrix effects of the measured value from a known spiked amount.

BLANK: A "clean" matrix analysis. Such as: Equipment Blank, Method Blank, and Trip Blank.

BREAKDOWN: A measure of the decomposition of certain analytes (DDT and Endrin) into by-products.

CAS: Chemical Abstracts Service, a registry where chemicals are assigned identification numbers.

CCB: Continuing Calibration Blank

CCV: Continuing Calibration Verification standard.

CLP: Contract Laboratory Program. A contract used by EPA to purchase analytical services. Also refers to the test protocols described in that contract. The CLP analyses can be used for EPA or for other clients. CLP-format data reports are arranged as described in the EPA CLP contract, including specified data report pages and all raw data.

CONTROL SAMPLE: A QC sample introduced into a process to monitor the performance of the system.

DL: Dilution, not used when the initial analysis is performed at dilution, but is used for a secondary dilution.

DoD: Department of Defense.

DUPLICATE: See Matrix Duplicate, Field Duplicate, and Matrix Spike Duplicate.

EQUIPMENT BLANK: A sample of analyte-free water that has been used during sample collection to measure any contamination introduced during sample collection.

ICB: Initial Calibration Blank

ICV: Initial Calibration Verification standard

IDL: Instrument Detection Limit. Statistical value similar to MDL, but with analyses performed on standards that have not been through the sample preparation process.

FIELD DUPLICATES: Independent samples that are collected as close as possible to the same point in space and time. They are two separate samples taken from the same source, stored in separate containers, and analyzed independently. These duplicates are useful in documenting the precision of the sampling process.

HT Holding Time. The maximum times that samples may be held prior to analysis and still be considered valid or not compromised (40CFR Part 136). DoD also clarifies the HT to mean the time elapsed from the time of sampling to the time of extraction or analysis , or from extraction to analysis…

LAB CONTROL SAMPLE (LCS): A blank spiked with compound(s) representative of the target analytes. This is used to document laboratory performance in a "clean" matrix.

LOD: Limit of Detection. The smallest amount of concentration of a substance that must be present in a sample in order to be detected at a high level of confidence (99%), per DoD.

LOQ: Limit of Quantitation (LOQ). The lowest concentration that produces a quantitative result within specified limits of precision and bias. The LOQ

is typically set at or above the concentration of the lowest initial calibration standard.

MATRIX: The component or substrate (e.g., water, soil, air, and oil) which contains the analyte of interest.

MATRIX DUP (DUP): A sample split by the laboratory that is used to document the precision of a method in a given sample matrix.

MATRIX SPIKE (MS): An aliquot of sample spiked with a known concentration of target analyte(s). The spiking occurs prior to sample preparation and analysis. A matrix spike is used to document the bias of a method in a given sample matrix.

MATRIX SPIKE DUP (MSD): Laboratory split samples spiked with identical concentrations of target analyte(s). The spiking occurs prior to sample preparation and analysis. They are used to document the precision and bias of a method in a given sample matrix.

MCL: Maximum Contaminant Level (MCL) is the highest concentration of a contaminant that is allowed in drinking water.

METHOD BLANK(MB): An analyte-free matrix to which all reagents are added in the same volumes or proportions as used in sample processing. The method blank should be carried through the complete sample preparation and analytical procedure. The method blank is used to document contamination resulting from the analytical process.

METHOD DETECTION LIMIT (MDL): The minimum concentration of a substance that can be measured and reported with 99% confidence that the analyte concentration is greater than zero and is determined from analysis of a sample in a given matrix type containing the analyte. For operational purposes, when it is necessary to determine the MDL in the matrix, the MDL should be determined by multiplying the appropriate one-sided 99% t-statistic by the standard deviation obtained from a minimum of seven analyses of a matrix spike containing the analyte of interest at a concentration estimated to be three to five times the MDL, where the t-statistic is obtained from standard references.

MSA: Method of Standard Additions

ND: Not Detected. Used in conjunction with the reporting limit.

ORGANIC-FREE REAGENT WATER: For volatiles, all references to water in the methods refer to water in which an interferent is not observed at the reporting limit of the compounds of interest. Organic-free reagent water

can be generated by passing tap water through a carbon filter bed containing about 1 pound of activated carbon. A water purification system may be used to generate organic-free deionized water. For semivolatiles and nonvolatiles, all references to water in the methods refer to water in which an interferent is not observed at the reporting limit of the compounds of interest.

PPB: Parts Per Billion, ug/L, ug/Kg

PPM: Parts Per Million, mg/L, mg/Kg

PQL: Practical Quantitation Limit. Equivalent to Reporting Limit.

PRECISION: The agreement among a set of replicate analyses.

PS: Post Spike. Spike added at the analysis level (as opposed to at the beginning of sample preparation) to determine interferences.

REPORTING LIMIT (RL): The lowest concentration that can be reliably achieved within specified limits of precision and accuracy during routine laboratory operating conditions. The RL is generally 5 to 10 times the MDL. However, it may be nominally chosen other than these guidelines to simplify data reporting. For many analytes the RL concentration is selected as the lowest non-zero standard in the calibration curve. Sample RLs are matrix-dependent, and are adjusted by the amount of sample analyzed, dilution, and percent moisture. Also see LOQ.

RE: Reextraction or Reanalysis

RPD: Relative Percent Difference, used to determine precision.

RRF: Relative Response Factor. Used for quantification with the internal standard procedure.

RT: Retention Time for a chromatographic peak, as calculated from the time of injection.

SAMPLE: A portion of material to be analyzed that is contained in single or multiple containers and identified by a unique sample number.

SAMPLE DELIVERY GROUP (SDG): A unit within a sample Case that is used to identify a group of samples for delivery.

SERIAL DILUTION (SD): A five-fold dilution of a sample. When corrected by the dilution factor, the diluted sample must agree with the original undiluted

sample within specified limits. Serial dilution may reflect the influence of interferents.

SAMPLE MANAGEMENT OFFICE (SMO) - A Contractor-operated facility operated under the SMO contract, awarded and administered by USEPA.

SOP: Standard Operating Procedure.

STANDARD ADDITION: The practice of adding a known amount of an analyte to a sample immediately prior to analysis. It is typically used to evaluate interferences.

STANDARD CURVE: A plot of concentrations of known analyte standards versus the instrument response to the analyte. Calibration standards are prepared by successively diluting a standard solution to produce working standards which cover the working range of the instrument. Standards should be prepared at the frequency specified in the appropriate method. The calibration standards should be prepared using the same type of acid or solvent and at the same concentration as will result in the samples following sample preparation. This is applicable to organic and inorganic chemical analyses.

SURROGATE: An organic compound that is similar to the target analyte(s) in chemical composition and behavior in the analytical process, but which is not normally found in environmental samples.

TRIP BLANK: A sample of analyte-free media taken from the laboratory to the sampling site and returned to the laboratory unopened. A trip blank is used to document contamination attributable to shipping and field handling procedures. This type of blank is useful in documenting contamination of volatile organics samples.

From EPA SW-846, Revision 4, 40CFR Part 136, DoD QSM and other sources.

# SPECTRUM ANALYTICAL, INC. RI DIVISION

# MAJOR INSTRUMENTATION and EQUIPMENT LIST

# APPENDIX A

# Laboratory Information System Equipment

1. **Data Collection**:

1.1. Seventeen- Hewlett Packard (HP) chem station software for collecting GC and GC/MS data (below) and one Perkin Elmer (PE) Total Chrom for collecting data from the GC-TCD/SCD.

- 5 GC-ECD (GCSEMI)
- 1 GC-FID (GCSEMI)
- 6 GC-MS (MSSEMI)
- 5 GC-MS (MSVOA)
- 1 GC-Hall/PID (GCVOA)
- 1 GC-FID/NPD (GCVOA)

1.2. Hardware varies but is x86 compatible

1.3. OS is Windows, Various Versions (9x, NT, 2000,Xp)

2. **Data Storage**:

2.1. Dell Poweredge servers (Windows 2003 server)

2.1.1. Bernoulli (primary file server, non-organic instrument data)
- Dual core Xeon processor
- 4 GB RAM
- 1 TB storage
- Symantec Backup Exec 12.5
- Tape drive Tandberg Data LTO-5 (1500-3000 GB)

2.1.2. Avogadro (organic instrument data)
- Dual P IV Xeon processors
- 2 GB RAM
- 105 GB storage
- Tape drive Tandberg LTO-2 (200-400 GB)

2.1.3. Planck (database server)
- Dual P IV Xeon processors
- 2 GB RAM
- 450 GB storage
- Tape drive Seagate LTO-1 (100-200 GB) not currently used

2.2. Tapes are for daily backup, long term archiving and data restoration

3. **Compound Identification**:

3.1. Fourteen - Target 4.14 chromatographic software

3.2. Hardware is Intel based for Target 4.14

3.3. OS is Windows Xp

4. **Forms Generation**:

4.1. In-house forms generation LIMS modules for SW-846, ILM and ISM metals

4.2. In-house forms generation LIMS modules for SW-846, OLC, OLM/ASP and SOM organics

4.3. Hardware varies but is x86 compatible

4.4. OS is Windows, Various Versions (2000 and Xp)

**Spectrum RI**
**Equipment List** **Department: Inorganics : Metals& Wet Chemistry**

| Equipment | Manufacturer | Serial # | Date Received | Date in Service | Condition New/Used | Equipment ID | Location |
|---|---|---|---|---|---|---|---|
| ICP/OES | Perkin Elmer | 077N3102302 | Nov-03 | Nov-03 | New | Optima3 | Metals |
| ICP/AES | Perkin Elmer | 069N8060801 | Nov-98 | Nov-98 | New | Optima2 | Metals |
| ICP/MS | ThermoScientific | SN01407C | Oct-08 | Dec-09 | New | X1 | Metals |
| Mercury Analyzer | Perkin Elmer | 1131 | Mar-00 | Mar-00 | Used | FIMS1 | Metals |
| Mercury Analyzer | Perkin Elmer | 101S7071002 | Feb-11 | Feb-11 | new | FIMS2 | Metals |
| GPR Centrifuge | Beckman Instruments | 7M149 | Apr-02 | Apr-02 | Used | Centrifuge | WC |
| Conductivity Meter | WTW Inolab Cond Level 1 | 3370010 | Apr-02 | May-02 | New | COND-1 | WC |
| Total Organic Carbon Analyzer | Tekmar/Dohrmann | US03035002 | Apr-03 | Apr-03 | Used | TOC1 | WC |
| Flow Injection Analyzer | Lachat Instruments | A83000-1020 | Apr-96 | Apr-96 | New | Lachat1 | WC |
| Ion Chromatograph | Dionex | 95030498E980802 | May-03 | May-03 | New | IC1 | WC |
| Spectrophotometer | Spectronic Instruments | 3SGD332010 | Apr-02 | Apr-02 | New | SPEC2 | WC |
| Spectrophotometer | Milton Roy Company | 3310004028 | Mar-06 | Mar-06 | New | SPEC3 | WC |
| Pensky Marten | Koehler 16200 | 5539 | June-95 | June-95 | New | FLASH1 | WC |
| Turbidity Meter | VWR® Model 800 | Tur800 2326 | April-12 | Feb-13 | Used | Turb1 | WC |

| | | | | | | | |
|---|---|---|---|---|---|---|---|
| COD Reactor | Hach Company | 990900019429 | Nov-03 | Nov-03 | New | COD1 | WC |
| COD Reactor | Hach Company | 950200012193 | Apr-02 | Apr-02 | New | COD2 | WC |
| Deionized Water Generator | Barnstead E-Pure D4641 | 1090001208384 | Jun-95 | Jun-95 | New | DI2 | WC |
| pH meter | Oakton Instruments | 875001 | Jun-12 | Jun-12 | new | WC-03 | WC |

**Spectrum RI**
**Balance List**

| Equipment | Manufacturer | Serial # | Date Received | Date in Service | Condition New/Used | Equipment ID |
|---|---|---|---|---|---|---|
| TOP-LOADING Balance | OHAUS | 1121230069 | 2000 | 2000 | New | TL10 |
| Analytical Balance | Denver A-250 | 0070742 | 2010 | 2010 | Used | AB-3 |
| TOP-LOADING Balance | OHAUS Voyager | F2921120391055 | 2001 | 2001 | New | TL9 |
| TOP-LOADING Balance | Denver | 0079896 | 2000 | 2000 | New | TL1 |
| TOP-LOADING Balance | OHAUS Precision Std. | C22427176 | 2002 | 2007 | New | TL6 |
| TOP-LOADING Balance | OHAUS Navigator | 1121122373 | 2002 | 2002 | New | TL11 |
| TOP-LOADING Balance | OHAUS | CD8910 | 2000 | 2000 | New | TL4 |
| TOP-LOADING Balance | OHAUS Navigator | 1122173423 | 2003 | 2003 | New | TL12 |
| TOP-LOADING Balance | OHAUS Scout Pro | 7126212230 | 2007 | 2007 | New | TL13 |
| | | | | | | |
| | | | | | | |
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| | | | | | | |
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**Spectrum RI**
**Equipment List** **Department: Organic Prep**

| Equipment | Manufacturer | Serial # | Date Received | Date in Service | Condition new/used | Equipment ID |
|---|---|---|---|---|---|---|
| TurboVap II | Caliper | TV0845N14899 | Jan-09 | Jan-09 | New | TV-4 |
| TurboVap II | Caliper | TV0902N15012 | Jan-09 | Jan-09 | New | TV-3 |
| TurboVap II | Caliper | 4364 | Mar-08 | Mar-08 | Used | TV-2 |
| TurboVap II | Caliper | Unable to view | Mar-08 | Mar-08 | Used | TV-1 |
| Shaker | Glas-Col | 412383 | Mar-08 | Mar-08 | New | N/A |
| Water Bath | Precision Scientific | 9508-005 | Dec-95 | Jan-96 | Used | N/A |
| Nitrogen Concentrator Bath | Organomations | 16526 | Jun-97 | Jun-97 | New | NZ1 |
| Deionized Water Generator | Barnstead E-Pure D4641 | 582941018789 | Jun-95 | Jun-95 | New | DI1 |
| Pressurized Fluid Extractor | Dionex | 98070129 | Jun-00 | Jun-00 | New | PFE1 |
| Gel Permeation Chromatograph | J2/AccuPrep | P26D031 | Jun-05 | Jul-05 | New | GPC3 |
| Gel Permeation Chromatograph | J2/AccuPrep | 06D-1196-4.1 | Jul-07 | Aug-06 | New | GPC4 |
| Misonex Ultrasonic Disruptor | Sonic Dismembrator Fisher Model 550 | Unable to view | | | New | OPH1 |
| Misonex Ultrasonic Disruptor | Sonic Dismembrator Fisher Model 550 | Unable to view | | | New | OPH2 |
| Misonex Ultrasonic Disruptor | Sonic Dismembrator Fisher Model 500 | Unable to view | | | New | OPH3 |

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|---|---|---|---|---|---|---|
| Misonex Ultrasonic Disruptor | Sonic Dismembrator Fisher Model 500 | Unable to view | | | New | OPH4 |
| Ultrasonic Cleaner FS30H | Fisher Scientific | RTB030721702 | Apr-07 | Apr-07 | New | N/A |
| Centrifuge Centra CL-2 | International Equipment Company | 42606943 | | | Used | N/A |

**Spectrum RI**
**Equipment List** **Department: GC-Semivolatiles**

| Equipment | Manufacturer | Serial # | Date Received | Date in Service | Condition New/Used | Equipment ID | Location |
|---|---|---|---|---|---|---|---|
| GC/ECD | Hewelett Packard | 3336A59890 | Oct-94 | Oct-94 | New | E2 | GC-SVOA |
| GC/ECD | Hewelett Packard | US00032017 | | | | E4 | GC-SVOA |
| GC/ECD | Hewelett Packard | US00037060 | | | | E5 | GC-SVOA |
| GC/ECD | Hewelett Packard | US00029100 | 13-Feb | 13-Feb | used | E6 | GC-SVOA |
| GC/FID | Hewelett Packard | US00001898 | | | | F1 | MS-SVOA |
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**Spectrum RI**
**Equipment List** **Department: Receiving**

| Equipment | Manufacturer | Serial # | Date Received | Date in Service | Condition New/Used | Equipment ID | Location |
|---|---|---|---|---|---|---|---|
| Dry Weight Oven | Thello | 600011006 | | | used | DWO | REC |
| Walk in Cooler | | Not Applicable | | | new | R1 | REC |
| Gyrotary Shaker table | New Brunswick Sci. Co. | unable to read | | | used | n/a | REC |
| pH meter | Oakton Instruments | 1446253 | Dec-08 | Dec-08 | new | WC-02 | REC |
| Kiln model TNF24-3 | Paragon Touch n Fire | 324341 | | | | n/a | WC |
| Stoppering tray dryer | FTS Systems Dura-Stop M | TD-12-90-133 | | | | n/a | WC |
| Freeze Dryer | FTS Systems Dura-Dry MP | unable to see | | | | n/a | WC |
| Dessicator | Sanplatec Corp | none | June-06 | June-06 | New | DryKeeper | REC |
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**Spectrum RI**
**Equipment List** **Department: SVOA**

| Equipment | Manufacturer | Serial # | Date Received | Date in Service | Condition New/Used | Equipment ID | Location |
|---|---|---|---|---|---|---|---|
| GC/MS | Hewelett Packard | US00011367 / US72821130 | Nov-99 | Nov-99 | Used | S3 | MS-SVOA |
| GC/MS | Hewelett Packard | CN10315002/ VS30945365 | May-03 | May-03 | New | S4 | MS-SVOA |
| GC/MS/FID | Hewelett Packard | CN107223014 / US73317299 | Jan-08 | Jan-08 | New | S5 | MS-SVOA |
| GC/MS | Hewelett Packard | CN10261100 | Nov-10 | Nov-10 | Used | S6 | MS-SVOA |
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**Spectrum RI**
**Equipment List** **Department: VOA**

| Equipment | Manufacturer | Serial # | Date Received | Date in Service | Condition New/Used | Equipment ID | Location |
|---|---|---|---|---|---|---|---|
| GC/MS | Hewelett Packard | 3336A55963 | | | | V1 | VOA |
| Auto sampler | OI | 13193 | | | | V1 | VOA |
| Concentrator | OI | J651460769 | | | | V1 | VOA |
| GC/MS | Hewelett Packard | 3336A58222 | | | | V2 | VOA |
| Auto sampler | OI | 13091 | | | | V2 | VOA |
| Concentrator | OI | H340460074 | | | | V2 | VOA |
| GC/FID/PID | Hewelett Packard | 2843A21041 | | | | V4 | VOA |
| Auto sampler | Tekmar/Dohrmann | 90312004 | | | | V4 | VOA |
| Concentrator | Tekmar/Dohrmann | 88341012 | | | | V4 | VOA |
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**Spectrum RI**
**Equipment List** **Department : VOA**

| Equipment | Manufacturer | Serial # | Date Received | Date in Service | Condition New/Used | Equipment ID | Location |
|---|---|---|---|---|---|---|---|
| GC/MS | Hewelett Packard | US00007055 | | | | V5 | VOA |
| Auto sampler | OI | 13462 | | | | V5 | VOA |
| Concentrator | OI | J651460769 | | | | V5 | VOA |
| GC/MS | Hewelett Packard | US00031343 | | | | V6 | VOA |
| Auto sampler | OI | B03745A407 | | | | V6 | VOA |
| Concentrator | OI | J651460769 | | | | V6 | VOA |
| GC | Hewelett Packard | 3140A37463 | | | | V7 | VOA |
| Auto sampler | Tekmar/Dohrmann | US01170015 | | | | V7 | VOA |
| GC/MS | Hewelett Packard | CN10411124 | Oct-10 | Nov-10 | NEW | V10 | VOA |
| Auto sampler | Tekmar/Dohrmann | US01157003 | Oct-10 | Nov-10 | USED | V10 | VOA |
| Concentrator | Tekmar/Dohrmann | US02021003 | Oct-10 | Nov-10 | NEW | V10 | VOA |
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# Weight Sets

**Laboratory weights for daily calibration use:**

1. WT1-Organic Prep Weight Set
2. WT2-Organic Prep 100g
3. WT3-Organic Prep 300g
4. WT4-Organic Prep 1kg
5. WT5-Inorganics Weight Set
6. WT6-VOA Weight Set
7. WT7-Unit 3 Weight Set

**NIST Class 1 Weight sets:**

1. W-01 Denver Instrument set: Serial number 98-121303 Class 1
2. W-03 Troemner set: Serial number 7283 Class 1

# Spectrum Analytical, Inc. Rhode Island Division

## CONFIDENTIALITY, ETHICS, and DATA INTEGRITY AGREEMENT

## APPENDIX B

# CONFIDENTIALITY, ETHICS, AND DATA INTEGRITY

The confidentiality, ethics, and data integrity agreement attached must be signed and dated by all new personnel associated with the data generated by Spectrum Analytical, Inc. Rhode Island Division. All said personnel will complete a training course and understand the information stated in the agreement. The course must include the ethical and legal responsibilities including the potential punishments and penalties for improper, unethical, or illegal actions. In addition, personnel are instructed on the importance of data confidentiality in both hard copy and digital forms. All personnel must fully understand this information before signing the agreement. A separate form is used for subcontractors and external auditors that request data for review.

Data Integrity training will be done on an annual basis. All employees are required to attend a training session or read a refresher document and sign off in hardcopy or through the digital SOP Database. All hard copy documents are stored in the employee's personnel file located in the QA Department.

All upper management personnel are required to sign a Non-disclosure Agreement which covers protecting confidentiality and proprietary rights. This Agreement is kept on file at the Spectrum Analytical, Inc., main offices in Agawam, Massachusetts.

**SPECTRUM ANALYTICAL, INC.**
**FEATURING HANIBAL TECHNOLOGY**
**Rhode Island Division**

## CONFIDENTIALITY, ETHICS AND DATA INTEGRITY AGREEMENT

I. I, _____ *(Name)*, state that I understand the standards of confidentiality, ethics and data integrity required of me with regard to the duties I perform and the data I report in connection with my employment at Spectrum Analytical, Inc. Rhode Island Division.

II. I agree that in the performance of my duties at Spectrum Analytical, Inc. Rhode Island Division. :

A. I shall not improperly use manual integrations to meet calibration or method QC criteria, such as peak shaving or peak enhancement.

B. I shall not intentionally misrepresent the date or time of analysis by resetting computer or instrument date/time.

C. I shall not falsify analytical results.

D. I shall not report analytical results without proper analysis documentation to support the results; dry-labbing.

E. I shall not selectively exclude data to meet QC criteria, such as calibration points, without technical or statistical justification.

F. I shall not misrepresent laboratory performance by presenting calibration data or QC limits within data reports that are not linked to the data set reported.

G. I shall not represent matrix interference as basis for exceeding acceptance criteria in interference-free matrices, such as method blanks and Laboratory Control Standards (LCS).

H. I shall not manipulate computer software for improper background subtraction or chromatographic baseline manipulations.

I. I shall not alter analytical conditions such as EM voltage, GC temperature program, etc. from standards analysis to sample analysis.

J. I shall not misrepresent QC samples such as adding surrogates after sample extraction, omitting sample preparation steps, or over-spiking/under-spiking.

K. I shall not report analytical results from the analysis of one sample for those of another.

L. I shall not intentionally represent another individual's work as my own.

III. I agree to report immediately any accidental or intentional reporting of non-authentic data by myself. Such report must be made to any member of Spectrum Analytical, Inc. Rhode Island Division Management or the QA Director (Hanibal Tayeh, Yihai Ding, Edward Lawler, Cinde Gomes, Sharyn Lawler) both orally and in writing.

IV. I agree to report immediately any accidental or intentional reporting of non-authentic data by other employees. Such report must be made to any member of Spectrum Analytical, Inc. Rhode Island Division Management or the QA Director (Hanibal Tayeh, Yihai Ding, Edward Lawler, Cinde Gomes, Sharyn Lawler) both orally and in writing.

V. Questions pertaining to confidentiality, ethics, and integrity may be posed to any of the above individuals.

VI. I agree not to divulge any pertinent confidential information including but not limited to data and any other information about a project to outside sources without the prior consent from the client.

I understand that failure to comply with the above confidentiality, ethics and data integrity agreement can result in my immediate dismissal from Spectrum Analytical, Inc. Rhode Island Division.
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_____ _____

*(Signature)* *(Date)*

_____

*(Print Name)*

## Training Session Record

**Please read, sign and follow the instruction (s) below.**

**Subject: Confidentiality, Ethics and Integrity Training to include proper laboratory practices with an understanding of examples and consequences for falsifying data or sharing confidential information. Falsifying data can lead to written warning, termination, business closure, and/or civil or criminal prosecution. It is my responsibility to report to my supervisor (anonymously if I prefer) any acts that could lead to the falsifying of data.**

**I agree that I understand the procedure referenced above and have attended a training session for its proper implementation.**

| Staff Member Name | Date | Signature | Staff Member Name | Date | Signature |
|---|---|---|---|---|---|
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## SUBCONTRACTORS

## CONFIDENTIALITY, ETHICS AND DATA INTEGRITY AGREEMENT

I. I, _____ *(Name)*, authorized representative of

_____(Subcontractor) state that I understand the standards of integrity required of me and the Subcontractor with regard to the duties performed and the data reported in connection with the analysis/analyses contracted by Spectrum Analytical, Inc. Rhode Island Division.

II. Subcontractor agrees that in the performance of analysis for Spectrum Analytical, Inc. Rhode Island Division:

A. Subcontractor shall not intentionally report data values or results that are not the actual values measured or observed;

C. Subcontractor shall not modify data values unless the modification can be technically justified through a measurable analytical process;

D. Subcontractor shall not intentionally report the dates and times of data analyses that are not the true and actual dates and times of analyses; and

D. Subcontractor shall not intentionally represent another's work as its own.

III. Subcontractor agrees to report immediately any accidental or intentional reporting of non-authentic data to Spectrum Analytical, Inc. Rhode Island Division.

IV. Subcontractor agrees not to divulge any pertinent information including but not limited to data and information about any Spectrum Analytical, Inc. Rhode Island Division projects to outside sources without the prior consent from Spectrum or its clients.

I understand that failure to comply with the above ethics and data integrity agreement can result in immediate termination of the subcontract relationship with Spectrum Analytical, Inc. Rhode Island Division.

_____ _____

*(Signature)* *(Date)*

_____

*(Name)*

_____

*(Title)*

# Confidentiality Agreement for External Audits

During the course of the laboratory audit/assessment certain information may become available to the auditor/assessor that is confidential.

All sample-related and project-related information at Spectrum Analytical, Inc. Rhode Island Division is confidential between Spectrum Analytical, Inc. Rhode Island Division and its client.

Any information obtained during the course of this audit/assessment may be used for audit/assessment purposes only.

No information obtained during the course of this audit/assessment may be disclosed by the auditor/assessor to any outside party, regardless of affiliation with the auditor/assessor.

Auditor/Assessor (signature): _____

(Print name): _____

(Date):_____

Company/organization name: _____

QAF.0014

# Spectrum Analytical, Inc. RI Division
# Resumes of Key Personnel

# APPENDIX C

# YIHAI DING
## Laboratory Director

Mr. Ding has experience in a wide variety of analytical chemistry techniques, including GC, GC/MS, HPLC and FTIR. His expertise includes the operation, calibration and maintenance of sophisticated analytical instrumentation, and the efficient operation of state of the art environmental service laboratories.

Mr. Ding's responsibilities as Laboratory Director at Spectrum Analytical, Inc. Featuring Hanibal Technology Rhode Island Division, involves the daily coordination of all laboratory sections to insure the production of high quality data meeting customer's technical and schedule requirements. His duties in this role include overseeing department supervisors and analysts in the daily calibration, maintenance and troubleshooting of analytical instruments, monitoring schedules and holding times, analysis of samples, review of sample and QC data. He also is involved with the implementation of Standard Operating Procedures, documentation of instrument and method QC criteria and development of new methods and implementation of new analytical technology.

Mr. Ding's prior experience includes research into the mechanisms and kinetics of various biochemical processes. A large portion of this research has required the analysis of reactants and products using state-of-the-art chemical instrumentation. Mr. Ding has also taught chemistry and biochemistry laboratory courses at the university level.

**EDUCATION**

**MIDDLE TENNESSEE STATE UNIVERSITY**
Murfreesbro, Tennessee
- Chemistry, MS

**JILIN UNIVERSITY**
Changchun, China
- Biochemistry, BS

**RELATED EXPERIENCE**

2005-present

**Spectrum Analytical, Inc., Featuring Hanibal Technology, Rhode Island Division (formerly Mitkem)**
- Laboratory Director

2005 **STL LABORATORIES**
Savannah, Georgia
- Supervisor of Semi-Volatile GC and GC/MS
- GC/MS Analyst
- GC/ECD Analyst

1998-2005 **MITKEM CORPORATION**
Warwick, Rhode Island
- GCMS Supervisor for both Volatile Organics and Semi-Volatile Organics Laboratories
- GC/MS Analyst

1994-1998 **MIDDLE TENNESSEE STATE UNIVERSITY**
Murfreesboro, Tennessee
- Researcher
- Laboratory Instructor, chemistry and biochemistry

1993-1994 **NATIONAL ENZYME ENGINEERING LAB**
Changchun, China
- Researcher

# SHARYN B. LAWLER

## Quality Assurance Director

Ms. Lawler has over twenty years of experience in the environmental laboratory industry. She has experience in implementation, operation and management of QA systems operating under USEPA, US Army Corps of Engineers and NELAC programs.

Ms. Lawler's responsibilities as Quality Assurance Director include development and implementation of the Quality Assurance Plan and Standard Operating Procedures. Her duties include interacting with federal and state regulatory officials in the acquisition and maintenance of laboratory certifications. She is also responsible for managing Spectrum Analytical, Inc. Rhode Island Division's document control program. Ms. Lawler performs both internal and external audits as well as overseeing the corrective action system, training program and evaluating QC check samples.

Previously Ms. Lawler was a senior data reviewer, where she was responsible for final QA/QC review of organic, metals and wet chemistry data. She insured final data met all method and in-house QC criteria prior to release to the customer, and that any issues were documented and described for inclusion in case narratives. A significant portion of this work involved review of full CLP-format data deliverables packages, both for standard as well as non-routine analyses. Prior to Spectrum Analytical Inc., Ms. Lawler worked for two CLP laboratories where she held positions including senior data review specialist, CLP Organics Task Manager and analyst in several laboratory sections.

**EDUCATION:**

**UNIVERSITY OF MASSACHUSETTS**
Amherst, Massachusetts
Independent Conc., Coastal Plant Ecology, BS

**RELATED EXPERIENCE:**

1997-Present

**Spectrum Analytical Inc., Featuring Hanibal Technology, RI Division (formerly Mitkem)**
- QA Director
- Senior Data Reviewer

1988-1997 **NATIONAL ENVIRONMENTAL TESTING**
Bedford, Massachusetts
- Senior Data Reviewer
- CLP Organic Task Manager

1983-1988 **CAMBRIDGE ANALYTICAL ASSOCIATES**
Boston, Massachusetts
- CLP Organic Task Manager
- Semivolatiles Analyst
- Preparation Laboratory Analyst

# EDWARD A. LAWLER

## Business Development Coordinator /Sr. Project Manager

Mr. Lawler has over thirty years of experience in environmental laboratory operations. He has extensive experience in managing laboratory workflow and in establishing and maintaining customer relationships. He also has considerable experience in a wide range of environmental chemical analyses, with a concentration in trace level volatile organics analysis.

As Business Development Coordinator, Mr. Lawler is responsible for securing contracts and BOA agreements with clients as well as pursuing new contracts and bids. He also works closely with lab staff to ensure they are aware of specific data deliverable requirements for new projects.

As Senior Project Manager, Mr. Lawler manages certain significant analytical testing programs, acting as principal technical liaison with the client. His extensive experience in laboratory data review allows him to ensure QA/QC criteria have been achieved, as well as preparing project narratives detailing these findings to the client.

Mr. Lawler's past responsibilities as Deputy Director for Quality Services included the prioritization of all analytical chemistry testing at Spectrum Analytical, Inc. Rhode Island Division. This included daily meetings with laboratory supervisors and managers to insure all technical and schedule requirements were met.

Mr. Lawler's previous experience includes various staff, management and senior management positions at several environmental testing laboratories. Direct project experience includes EPA CLP, Army MRD, Navy NEESA and NFESC, DOD HAZWRAP and New York DEC ASP programs. Mr. Lawler has also provided expert testimony at several Superfund trials including pre-trial consulting and trial witness services.

**EDUCATION:** **UNIVERSITY OF MASSACHUSETTS**
Amherst, Massachusetts
Environmental Sciences, BS 1977

**RELATED EXPERIENCE:**

| | |
|---|---|
| 1997- Present | **Spectrum Analytical Inc., Featuring Hanibal Technology, Rhode Island Division (formerly Mitkem)**<br>- Business Development Coordinator<br>- Senior Project Manager<br>- Deputy Director for Quality Services<br>- Operations Manager |
| 1989-1997 | **NATIONAL ENVIRONMENTAL TESTING, CAMBRIDGE DIVISION**<br>Bedford, Massachusetts<br>- Division Manager<br>- Proposal/Contract Manager<br>- Director of Project Management |
| 1983-1989 | **CAMBRIDGE ANALYTICAL ASSOCIATES, INC.**<br>Boston, Massachusetts<br>- Project Manager<br>- Volatile Organic Laboratory Manager |
| 1978-1983 | **ENERGY RESOURCES COMPANY, INC. - ERCO**<br>Cambridge, Massachusetts<br>- Volatile Organics (GC) Manager<br>- Analytical Chemist-Volatile Organics Lab<br>- Analytical Chemist-Organic Preparation Lab |
| 1978 | **LAPUCK LABORATORIES, INC.**<br>Watertown, Massachusetts<br>- Analytical Chemist-Wet Chemistry & Metals<br>- Microbiologist |

# SCOTT P. HUNTLEY

## IT Manager

Mr. Huntley has over twenty years experience in the environmental testing field. He has considerable experience in computer sciences and had been involved, throughout his career, in the setup and implementation of several Laboratory Information Management Systems (LIMS) and automated data reduction systems. Mr. Huntley's responsibilities include the set-up and validation of automated data transfer, reduction, storage, evaluation and reporting programs within Spectrum Analytical, Inc. RI Division's LIMS. He also is responsible for set-up of the electronic data delivery capabilities as well as the control charting capabilities of this system.

Previously Mr. Huntley has held several supervisory positions in environmental laboratories focusing on CLP and other DOD analytical programs. He has a wide range of experience in routine and state of the art analytical programs and methods. Mr. Huntley is experienced in the use of automated data transfer and reduction systems and laboratory automation techniques.

**EDUCATION:**

**RHODE ISLAND COLLEGE**
Providence, Rhode Island
Chemistry, BS
Computer Science, BS

**RELATED EXPERIENCE:**

| | |
|---|---|
| 1999-Present | **Spectrum Analytical, Inc., Featuring Hanibal Technology, RI Division (formerly Mitkem)**<br>MIS Senior Systems Analyst |
| 1996-1999 | **MITKEM CORPORATION**<br>Warwick, RI<br>- Senior Chemist<br>- Organic Lab Manager |
| 1991-1996 | **EA LABORATORIES**<br>Sparks, MD |

- Supervisor of Organic Chemists

1989-1991 **CEIMIC CORPORATION**
Narragansett, RI
- Night shift supervisor

1986-1989 **RI ANALYTICAL LABORATORIES**
Providence, RI
- GC Chemist

# Catherine L. Mosher

## Organics (SVOA/VOA) Department Manager

Ms. Mosher has experience in a wide variety of analytical chemistry techniques, including GC/FID and GC/MS. Her expertise includes the operation, calibration and maintenance of sophisticated, computer controlled instrumentation. Her expertise also includes analyses and QA review of forensics extended alkylated PAH and Biomarker analyses.

Ms. Mosher is employed as the Organics Department Manager in Spectrum Analytical Inc. Rhode Island Division, and oversees both the Volatile and Semivolatile departments. Ms. Mosher's responsibilities involve the coordination of organics analyses using GC/MS and GC instrumentation following both US EPA CLP and SW846 protocols. Her duties in this role include supervising analysts in the daily calibration, maintenance and troubleshooting of analytical instruments, monitoring schedules and holding times, analysis of samples, review of sample and QC data. She is involved with the implementation of Standard Operating Procedures, documentation of instrument and method QC criteria and development of new methods and implementation of new analytical technology. Ms. Mosher also insures the production of organic data is coordinated with other laboratory sections.

**EDUCATION**

**Community College of Rhode Island**
Warwick, Rhode Island
Certificate of Chemical Technology - 1991

**RELATED EXPERIENCE**

02/2007-Present

**Spectrum Analytical Inc., Featuring Hanibal Technology, Rhode Island Division (formerly Mitkem)**
- Manager, SVOA Department
- Senior Scientist, SVOA Laboratory

05/2005 – 12/2006

**Alpha Woods Hole Laboratories**
Raynham, MA
- Development of Volatile Air Laboratory

- Supervisor for Organics analyses including GC/MS VOA and SVOA, ECD's and FIDs
- Forensic Team Leader

03/1997 – 05/2005 **Woods Hole Group Laboratories**
Raynham, MA
- Forensic Team Leader
- GC/MS Group Leader

04/1996 – 03/1997 **Inchcape Testing**
New Bedford and Raynham, MA
- Semivolatile analyst
- Volatile analyst

09/1991 – 04/1996 **Energy and Environmental Engineering Inc.**
Somerville, MA
- Semivolatile GC/MS Supervisor
- GC-Pesticide/PCB analyst

01/1989 – 09/1991 **New England Testing Laboratory**
North Providence, RI
- Senior Chemical Technician - including Organic, Inorganic, Metals, and Microbiology analyses

10/1987 – 09/1988 **Rhode Island Analytical Laboratory**
Warwick, RI
- Chemical Technician

# HUIYAN HEATHER ZHAO-ANDERSON
## Inorganics Department Manager

Ms. Zhao-Anderson is employed as the Manager in Spectrum Analytical Inc. Rhode Island Division's Inorganics Department. Ms. Zhao-Anderson's responsibilities involve the coordination of metals and wet chemistry analyses using ICP/MS, ICP/AES and a variety of other instrumentation following both US EPA CLP and SW846 protocols. Her duties in this role include supervising analysts in the daily calibration, maintenance and troubleshooting of analytical instruments, monitoring schedules and holding times, analysis of samples, review of sample and QC data. She is involved with the implementation of Standard Operating Procedures, documentation of instrument and method QC criteria and development of new methods and implementation of new analytical technology. Ms. Zhao-Anderson also insures the production of inorganics organic data is coordinated with other laboratory sections. Prior to managing the inorganic department, Ms Zhao-Anderson was the department manager of our volatile organics laboratory for several years.

**EDUCATION**

**Yale University**
New Haven, CT
School of Forestry and
Environmental Study, MS 2005

**Peking University**
Beijing, China
Environmental Science and Economics
BS 2002

**RELATED EXPERIENCE**

09/2005 -Present

**Spectrum Analytical Inc., Featuring Hanibal Technology, Rhode Island Division (formerly Mitkem)**
- Manager, Inorganic Department
- Manager, VOA Department
- GC/MS Chemist, VOA Laboratory

# DAWNE SMART

## Data Reviewer, Project Manager, Data Reporting Supervisor

Ms. Smart's responsibilities as project manager involve the management of Spectrum Analytical Inc. Rhode Island Division's EPA Contract Laboratory Program (CLP) analytical services contract for ISM. This includes the daily oversight of incoming samples, maintenance of chain of custody documentation and communication records and resolution of any discrepancies or other issues involving CLP ISM sample assignments. Her responsibilities also include ongoing communication with EPA, sampler and CSC personnel, as well as monitoring data delivery schedules, writing project narratives and finalizing case communication.

Ms. Smart also is currently supervising the Data Reporting staff. She oversees the staff that generates data packages for all inorganic and organic fractions for different levels of report packages that will then go to data review personnel. Additionally, she and her staff are responsible for final report generation when all fractions of a project are completed, including bookmarking, pagination, final package posting to the website and hard copy report mailing if applicable.

Ms Smart also reviews sample and QC data, and completed CLP data packages for both organic and inorganic programs. Ms. Smart has extensive experience in Data Review as well as Quality Assurance. A significant portion of her previous employment included management of the Data Review department as well as the on-site QA Specialist for a major specialized laboratory.

**EDUCATION**

**COMMUNITY COLLEGE of RHODE ISLAND**
Warwick, Rhode Island
Certificate of Chemical Technology - 1991
Associate in Applied Science - 1997

**RELATED EXPERIENCE**

2007-Present

**Spectrum Analytical Inc., Featuring Hanibal Technology, Rhode Island Division (formerly Mitkem)**
- Data Reporting Supervisor
- ISM Contract manager

| | |
|---|---|
| | -Manager, Metals Department<br>-Supervisor, Inorganic Department |
| 1999 – 2007 | **ALPHA WOODS HOLE LABORATORIES**<br>Raynham, Massachusetts<br>-QA Specialist<br>-Manager, Data Review Department |
| 1996 – 1999 | **ANALYTICAL BALANCE COMPANY**<br>Middleboro, Massachusetts<br>- Department Head, Metals Analysis |
| 1995 – 1996 | **FOXBORO COMPANY**<br>West Bridgewater, Massachusetts<br>- Chemist |
| 1988 – 1995 | **NEW ENGLAND TESTING LABORATORY**<br>North Providence, RI<br>- Senior Laboratory Technician<br>- Laboratory Technician |
| 1987 – 1988 | **RHODE ISLAND ANALYTICAL LABORATORIES**<br>Warwick, RI<br>- Metals Preparation Technician<br>- Laboratory Assistant<br>- |

# AGNES R. HUNTLEY

## Project Manager

Ms. Huntley has gained extensive and diversified experience in environmental laboratories using U.S. EPA CLP and SW846 methodologies, as well as participating in US Navy and Army analytical services programs.

Ms. Huntley's responsibilities involve the management of Spectrum Analytical Inc. Rhode Island Division's EPA Contract Laboratory Program (CLP) analytical services contracts. This includes the daily oversight of incoming samples, maintenance of chain of custody documentation and communication records and resolution of any discrepancies or other issues involving CLP sample assignments. Her responsibilities also include ongoing communication with EPA, sampler and CSC personnel, as well as monitoring data delivery schedules, writing project narratives and finalizing case communication. Ms. Huntley has managed four contracts with the EPA, which included one Organics Low Concentration (OLC), two Organics Low/Medium Concentration (OLM) and one Inorganics Low/Medium Concentration (ILM) analytical services contracts. At present Ms. Huntley manages the Organics Multi-Media, Multi-Concentration (SOM01.2) Analytical Services Contract.

Previously, Ms. Huntley held the position of QA/QC Manager where her responsibilities included the development and implementation of Standard Operating Procedures, documentation of instrument and method performance using Method Detection Limit studies, and routine review of final laboratory data reports, review of analyst training and performance data and management of the corrective action system. Her duties also included interaction with federal and state regulatory officials in the acquisition and maintenance of laboratory certifications.

Prior experience includes management of the daily operations of the Organic Preparation Laboratory. Duties in this position included monitoring sample backlog, holding times, process work flow, and delivery due dates. Ms. Huntley also developed and implemented new test methods, trained laboratory staff, performed instrument maintenance and reviewed sample and QC data. Prior to joining Spectrum Analytical Inc. Ms. Huntley worked as an analytical chemist at NET Cambridge Division performing analyses under a wide variety of programs including Army COE, Navy NEESA, DOE HAZWRAP and EPA CLP.

**EDUCATION**

**SIMMONS COLLEGE**
Boston, Massachusetts
- Chemistry, BS
- Mathematics, BS

**RELATED EXPERIENCE**

| | |
|---|---|
| 1997-Present | **Spectrum Analytical, Inc., Featuring Hanibal Technology, Rhode Island Division (formerly Mitkem)**<br>- Project Manager, SOM Contract manager<br>- Supervisor, Sample Receiving Department |
| 1997-2008 | **MITKEM CORPORATION**<br>Warwick, Rhode Island<br>- CLP Project Manager<br>- QA/QC Manager<br>- Manager, Sample Preparation Laboratory |
| 1995-1997 | **NATIONAL ENVIRONMENTAL TESTING**<br>Bedford, Massachusetts<br>- Chemist, Organic Preparation |
| 1992-1995 | **SIMMONS COLLEGE CHEMISTRY DEPT.**<br>Boston, Massachusetts<br>- Teaching Assistant, Chemistry Department |

QAP Revision Page:

Rev 1 (02/01/2013): Included Facility floor plan, Updated Org Chart, updated equipment list, DW metals reporting requirements per 310 CMR 42

# APPENDIX F

EDV's Quality Assurance/Quality Control Plan
and
Dr. Maxine Wright-Walters Curriculum Vitae.

# QUALITY ASSURANCE/QUALITY CONTROL PLAN FOR DATA VALIDATION SERVICES


**EDV, INC**
**ENVIRONMENTAL DATA VALIDATION, INC**

**Corporate**
**1326 Orangewood Avenue**
**Pittsburgh, PA 15216**
**Phone-412-341-5281**
**Fax- 412-571-1932**

**Office Location**
**7712 Tuscarora Street**
**Pittsburgh, PA 15221**
**Phone-412-242-5200**
**Fax-412-242-5210**

**WEB PAGE: http://www.edv-inc.com**

# QUALITY ASSURANCE/QUALITY CONTROL PLAN FOR DATA VALIDATION SERVICES

# Table of Contents

# QUALITY ASSURANCE/QUALITY CONTROL PLAN FOR DATA VALIDATION SERVICES

## *OVERVIEW*

Environmental Data Validation Inc. (hereinafter referred to as EDV) is a certified small, woman-owned, disadvantaged, data validation and consulting business specializing in Environmental, Public Health and Scientific Research, Analytical data validation, Environmental consulting and Total environmental quality. Our motto is to deliver quality work on a timely basis. Established in 1990, EDV has kept its pace with changes and procedures in the environmental arena.

EDV is comprised of scientists and technical experts who specialize in environmental health and safety training & occupational health and safety consulting, building inspections, environmental site assessments, chemical and radiochemical data validation, environmental health and safety consulting, risk assessment, hazard assessment, exposure assessments, environmental health assessments, ecological risk assessments, epidemiological/environmental study design and quality consulting. Our consultants are from the academic arena or private sector and include; environmental scientists, industrial hygienists, epidemiologists, toxicologist, public health specialists and environmental engineers, chemists, biologists and health and safety specialists.

As part of our commitment to quality and the environment, EDV established an Environmental Management System based on the ISO 14000 standard and an Environmental Policy Statement; the blue print on which the company operates, and the basis for the environmental management system. The Environmental Policy Statement in integrated in our QA/QC program.

## ENVIRONMENTAL POLICY STATEMENT

Environmental Data Validation Inc is committed to developing, implementing, reviewing and maintaining an environmental management system, wherein the organizational structure, processes and resources are sufficient to continually measure, monitor and improve our environmental performance.

EDV understands that all activities, products or services can impact the environment. It is our policy to use practices and materials that can reduce, avoid or control pollution, which may include recycling, efficient use of resources and material substitution.

EDV will:

- adhere to all relevant environmental regulations and laws
- integrate this policy with its Quality policy
- seek to continually improve our overall environmental impact to our customers and the community
- adhere to integrity and high ethical standards

# QUALITY ASSURANCE/QUALITY CONTROL PLAN FOR DATA VALIDATION SERVICES

## INTRODUCTION

Quality Assurance (QA) plays a critical role in the generation and use of environmental data. QA activities ensure that the environmental sampling and analysis process is verified and documented so that the uncertainties in the resulting data can be controlled and quantified. In this way, the information gained from QA activities allows a data end user to determine whether the data are good enough to support their intended use.

Our motto is to deliver quality work on a timely basis. Our size and technical expertise has allowed us to accommodate our clients on very short notices and quick turnaround times. Our Quality Assurance/Quality Control program was established so as to give our clients formal documentation as to how we perform our validation efforts and the added security of knowing that their data is being handled professionally. As part of our commitment to quality and the environment, EDV established an Environmental Management System based on the ISO 14000 standard and an Environmental Policy Statement, the blue print on which the company operates, and the basis for the environmental management system. The Environmental Policy Statement in integrated in our QA/QC program.

# QUALITY ASSURANCE/QUALITY CONTROL PLAN FOR DATA VALIDATION SERVICES

## POLICY AND OBJECTIVES

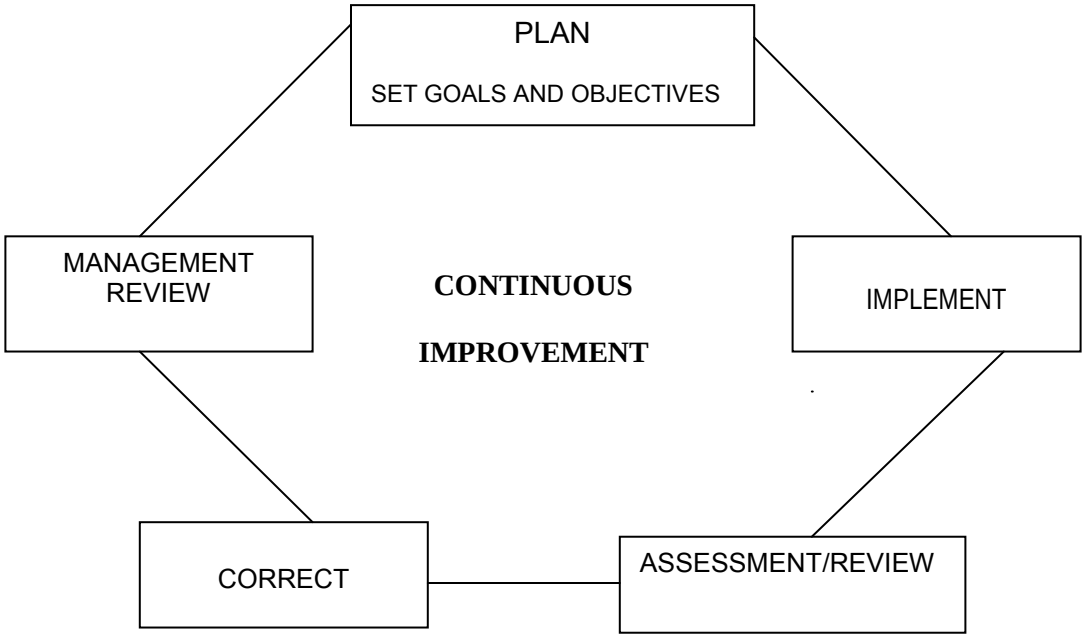
EDV's Quality Assurance/Quality Control (QA/QC) program was established to ensure quality and, validity to the work performed. The **quality assurance program** provides the structure, policies and responsibility for the execution of quality control and quality assessment operations, to assure our clients that defined standards and quality of a stated confidence level are met. The quality **control program** ensures maintenance of the controlled validation, review and data management process. The quality **assessment program** incorporates all the necessary elements to ensure that the quality control system is functioning effectively. To ensure that the highest standard of work is accomplished, EDV strictly adheres to QA/QC guidelines for data validation established by the EPA, in the National Functional Guidelines for Organic Analyses, and the National Functional Guidelines for Inorganic Analyses. Modifications to these guidelines established by various EPA regions or other governing bodies such as NEESA, DOE and AFCEE are utilized on a project specific basis. Our objective is to stay within the limits of data validation as we perform our tasks.

The satisfaction of our clients is most important to us; for this reason, we like to earn their confidence in the work performed by EDV. Our QA/QC program was so designed. This **Quality Assurance Project Plan** (QAPP) is designed based on the QA/QC program. It is important to us that our clients know, EDV's QA/QC system is in place so that their data can be accounted for, at all times, while it is in our hands and, that a thorough and complete job is done in validating the data.

It is the objective of the QAPP to ensure that quality results are produced by our validation efforts and that there is documentation every step of the way to verify this. It is also our objective and, policy to ensure that the results from the validation process are traceable. Our reports are written for easy understanding by the data end user.

## QA/QC PROGRAM CYCLE

EDV QA/QC program is based on Continuous Improvement and is reflected in the program's cycle for which the key elements of the system are listed below.



# QUALITY ASSURANCE/QUALITY CONTROL PLAN FOR DATA VALIDATION SERVICES

**Plan** - This is important so that the each department implement the quality policy in accordance with its guiding principles. Here objectives and goals/targets are identified.

**Implement** - This is necessary to effectively carry out the objectives of the QA/QC program,

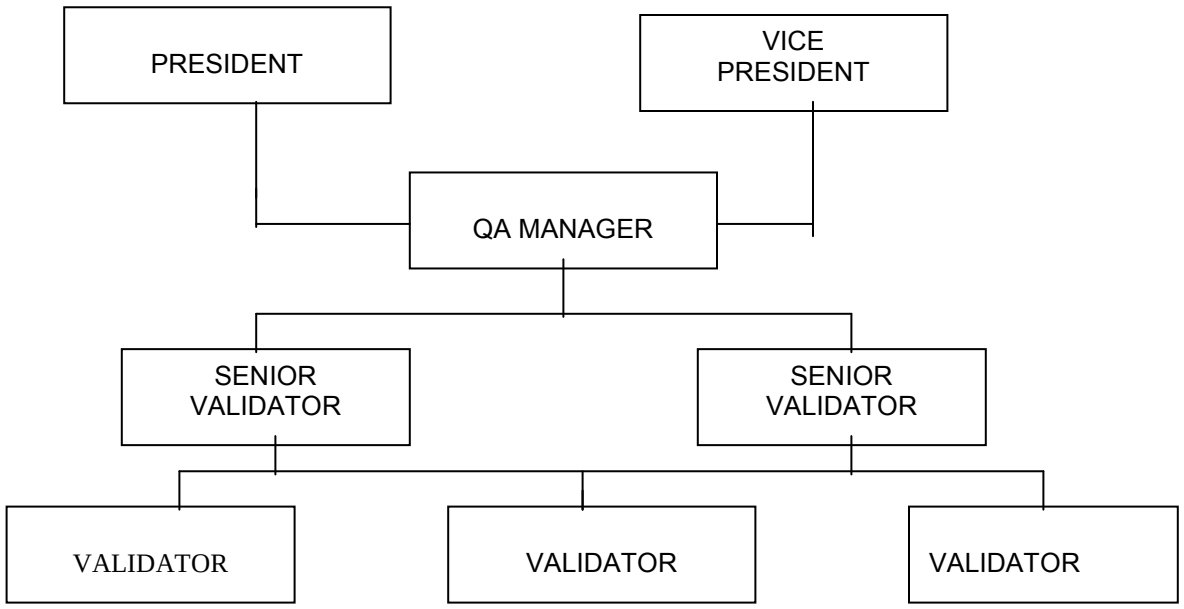
**Assess/Review** - This is where the policy and objectives of the program are reviewed.

**Correct** - This is the action necessary to ensure that the policy goals/targets and objectives are met.

**Management Review** - This is the overall assessment of the QA/QC program by management. From here deficiencies are corrected and continuous improvement enhanced.

# QUALITY ASSURANCE/QUALITY CONTROL PLAN FOR DATA VALIDATION SERVICES

**QA MANAGEMENT**




## QA Responsibilities and Reporting Relationships

The QA Management team is reflected above. Reporting goes up the chain of command, that is, the validators report to the senior validators who in turn report to the QA manager who reports to the president or vice president (in the event that the president is absent).

The QA manager is responsible for the overall QA/QC program and implements procedures, changes and corrective actions. The senior validators oversee or mentor the validators. The validators are responsible for data tracking, and overall smooth running of the QA/QC system on a day to day basis.

## QA Document Control

This is executed from the time the data package gets to EDV. It is highly important to have this so that we know where things are at all times. We understand the confidential nature of this subcontract and so, a data package or SDG will be assigned to a validator and remain in his/her possession until completed. The assigned validator is responsible for that package until its review is completed

A specific file cabinet will be designated for each subcontract. All documents pertaining to the subcontract will be stored here. The cabinet is fire proof and will be kept locked. The QA manager will hold the keys.

Only validators assigned to the subcontract will handle these data packages. All transfer of data packages will require a signature. When the review process is completed the Log-In notebook, shipping/mailing logbooks will be completed to reflect this. Once the report is received and approved by

the client, the data generated from the package will be backed up electronically and stored for two (2) years. A hard copy will also be stored.

**QA Program Assessment Procedures**

The QA program will be assessed periodically by the QA manager to ensure that all parameters are within control. Corrective action measures will be taken to remedy any out of control criteria. The program will be assessed to ensure that it is fulfilling its intended purpose. The goal of the program will be reviewed and such items as logbooks, worksheets, reports and re-submittals. The results of our assessment will be tallied and statistically assessed to see if there are any established trends.

## PERSONNEL

EDV hires qualified professionals. Each validator holds at least a bachelor's degree and has extensive laboratory experience. These validators are highly trained and are competent and fully experienced to work on any subcontract. Upon hire validators embark upon an extensive training program which includes such topics as: quality assurance project plan (QAPP), chain of custody, laboratory procedures, sample preparation methods, analytical methods, instrumentation, chromatogram interpretation and report writing.

**Training Progress**

Once every six months a refreshers training program is provided for our validators.

## FACILITIES AND EQUIPMENT

EDV has the necessary equipment to successfully perform on any subcontract. We are equipped with computers, calculators, adding machines, a typewriter, a copier, a fax machine and scientific calculators. Our computers have a battery operated backup system in the event of a power failure. Our fax machine is operational 24 hours per day, 7days per week.

These equipments are maintained on a schedule of once per year, or on the manufacturer's suggested schedule. All validators utilize a password to log on to the computer system. This password expires every thirty (30) days, at which point a new one must be selected. All electronic files are backed up on a daily basis. All file cabinets will be locked and the keys secured by the QA manager.

## *INTERNAL DATA VALIDATION (IDV) PROCEDURES*

## WHAT IS ANALYTICAL DATA VALIDATION

Analytical data validation is defined as an independent ***systematic*** process for reviewing a body of data against a set of criteria to provide assurance that the level of quality of the data are known and ***documented***. It consists of data screening, checking, auditing, verification and review. It serves two important management functions. First, it reviews the entire data collection, reduction and management process and identifies any errors in the flow of data from the point of generation to the final laboratory report. Second, it compares analytical precision as measured by laboratory duplicates, spikes and calibration standards against guidelines that are available from either analytical method or documents

such as EPA's Functional Guidelines

## DOCUMENT CONTROL

### Tracking Custody-and Storage

For the SDGs and Certificate of Analysis herein after referred to as Data Package, once received by EDV it will be stamped "received" and dated. The data package will then be logged in the Data Package Log-In notebook. It is also logged into the computer system. It will be logged by client's name, contract number, number of samples, sample matrix(ces), analysis type/parameter, date received, turnaround time and validation protocol to be utilized. All this will be done on the same day that the data package is received by EDV. For data packages received on a Saturday, the same process will be in effect.

Upon completion of the receipt process, the data package will then be distributed to tile relevant data validator(s), who must sign to the receipt of the data package and the number of samples in receipt. The validator will then check with the Data Log-In notebook as well as the computer log-ln for such information as turnaround time, validation protocol and any other pertinent information related to the Data Package. Based on the sample type/parameters, the validator will then obtain the required data validation protocol and worksheets to be used for the project. For this project, general data validation worksheets will be used.

The Data Package can be tracked at all times from the Data Log-In notebook or the computer Data Package Log-In The Data Package remains in the custody of the validator until the data review process is completed. The data packages, when not in use and at the end of each workday, will be locked in the designated file cabinet(s). No outdated SOPs will be utilized for validation.

### Logbook Maintenance and Archiving Procedures

Logbooks are maintained as per EDV's in house guidelines for Log Book Maintenance. The guidelines are:

No white outs or erasers of any form are to be used in the logbooks.
All errors must be lined through and the corrected item written above.
All corrections must be initialed and dated.
All entries in the logbooks must be signed
All logbook pages must have a heading and the pages sequentially numbered

All logbooks when full are labeled in bold letters across the cover as to the period for which it was used, that is, the start and end date of the logbook. The full (completed) logbook is stored in fire-proof cabinets, which are stored in the Data Storage room.

### SOPs Review, Distribution and Revision

All SOPs for the validation process are reviewed periodically and revised when necessary. The revised edition will clearly state what revision number it is. For every revision done, the same number is

assigned with the letter R# indicating revision number (e.g. SOP LG 3005 at second revision would be LG3005R2). The numbers are assigned in numeric order starting with the number one. The QA manager must approve the revised and original SOPs. A copy of each SOP is distributed to tile data validators for their files.

Documentation or Technical procedures will be revised as necessary. The QA manager will do all revision. Each revision will be stated on the document. Before any revision can take place management personnel must first discuss it. Once a consensus is reached then the QA manager will perform the revision.

## INTERNAL DATA VALIDATION (IDV) METHODOLOGY

**IDV Procedures**

Upon receipt of the data package from the client, the QA manager will check that the work Release is both technically and contractually correct in its entirety. EDV will verify/ that no conflicting information is present. If conflicting information is found EDV will immediately notify the client in writing (within 48 hrs of discovery) before proceeding further. The data package will be stamped "received" and dated. Once all conflicting information is resolved, the data package will then be logged in the Data Package Log-ln notebook. It will be logged by client's name, contract number, number of samples, sample matrix (ces), analysis type/parameter, date received, turnaround time and validation protocol to be utilized.

Upon completion of the receipt process, the data package will then be assigned and distributed to a qualified data validator to perform data validation on each applicable package. The data validator must sign to the receipt of the data package and the number of samples in receipt. **(Only the data validators listed in the Proposal will be allowed to work on the data** packages}. The validator at this point would have already been briefed on the requirements of this subcontract and will then check with the Data Package Log-In notebook for such information as turnaround time, validation protocol, and any other pertinent information related to the data package. The validators will also cross-check the information with the computer Log-In.

Data is generally validated according to the EPA's National Functional Guidelines for Organic Data Review, National Functional Guidelines for Inorganic Data Review, DOE Rocky Flats Plant "Radiochemical Data Validation Guidelines – Gross Alpha/Beta by Gas Proportional Counters", DOE Rocky Flats "Radiochemical Data Validation Guidelines-Analyses by High Resolution Gamma Spectroscopy" and any other relevant modifications of these protocols.

The extent of data validation will be at level IV, which is CLP. All our validation efforts will be documented on worksheets to allow traceability and ensure thoroughness. The worksheets will document any criteria out of limits. Flagging will be done according to the guidelines referenced above. The client will be notified in writing of any contract and or quality assurance criteria, which were not met within 48 hours of discovery. Any corrections made will be done in red ink by drawing a line through the incorrect item, writing the corrected item above, initialing and dating the item.

At the end of the IDV review process, the validation findings will be cross-checked by a secondary validator. If there are discrepancies that cannot be resolved, then a senior validator will check the data package for completeness and accuracy. In the event that the senior validator is unable to find a resolve then, the QA manager will check the package and make a decision. The QA manager will check

all data packages. All corrective action measures will be approved by the QA manager regardless of who initiated them. On this subcontract any one of the assigned validators can initiate a corrective measure after discussion with the QA manager.

**DATA VALIDATION REPORT**

The data validation report will be prepared based on findings. It will then be reviewed and approved by the Project/QA Manger.

'The data validation report will be in a narrative form and will describe justification of the proposed rejection of any results, problems encountered in the preparation of samples, during data validation and associated corrective actions ( including telephone logs for the analytical laboratory and EDV/client). A checklist that inventories the major types of documents received for each SDG from the laboratory, as well as any missing documents will be included in the data validation report. The final data validation report will be paginated and will contain the signature of the Project Manager documenting her review and approval of the data package.

**DELIVERY OF SUPPLIES/SERVICES (DELIVERABLES)**

The client will receive deliverables based on the turnaround times on the data packages. This could be 3, 7, 14 or 30 days.

**QA OVERSIGHT**

Data QA is assured through various steps that are in place. The purpose of QA is to ensure that the required elements of the quality control plan are met. Spot checks (internal audits) will be done on notebooks, worksheets and data summary tables. Corrective actions are in place for any inconsistencies found during this internal audit. When an internal audit is done, a report is generated and presented to every validator. (In the case of this subcontract, the report will only be presented to the validators assigned to this project)

Once every six months a performance audit will be done; the validators assigned to this subcontract will be given a set of data (of known results) to evaluate and generate a data validation report and data qualification summary, If the outcome is unsatisfactory, then, the deficient areas are identified and corrective action measures taken. These measures could include retraining.

Within the realm of QA, proper reporting procedures must be adhered to. QA reporting goes up the chain of command (see QA management chart). The QA manager has full responsibility of the QA program and has the power to designate responsibilities to each validator.

# QUALITY ASSURANCE/QUALITY CONTROL PLAN FOR DATA VALIDATION SERVICES

## Corporate Qualifications

**Maxine Walters**

Maxine Walters has 21 years extensive experience in analytical chemistry. Her expertise in data validation includes all types of parameters such as volatile target compounds (TCL), semi-volatile target compounds, pesticide/PCBs, dioxins & furans, conventional general/wet chemistry, TAL metals, leachate and reactivity characteristics (TCLP) priority pollutants-metals & organics; radiological parameters including gross alpha/beta, gamma spectroscopy parameters; thermal ionization mass spectroscopy, fluorometric uranium, alpha spectroscopy-strontium 89/90; alpha spectrometry- thorium-237, uranium-234, 238, neptunium-237, plutonium-238, 239, 240, americium-241 and curium-242, 243, 244 and, liquid scintillation counting parameters-tritium.

Her other experience includes QA/QC consulting for a variety of private sector clients as well as for state and federal programs, development of QAPPs, SAPs and SOPs for standard and non-standard methods, laboratory training, data usability assessment and general project management.

**Professional Qualifications**

Ms. Walters has 21 years experience in environmental/analytical chemistry. This includes 16 years extensive experience in analytical data validation (CLP and non-CLP), development of Data Quality Objectives, development of QA/QC and laboratory training programs, remedial investigation/feasibility studies (RI/FS), QAPPs and SAPs development. Ms Walters has 19 years project management experience and 9 years in depth research experience, which includes instrumentation and advance organic chemistry.

**Linda Wright**

Linda Wright has 15 years extensive experience in analytical chemistry. Her expertise in data validation includes all types of parameters such as volatile target compounds (TCL), semi-volatile target compounds, pesticide/PCBs, dioxins & furans, conventional general/wet chemistry, TAL metals, leachate and reactivity characteristics (TCLP) priority pollutants-metals & organics; radiological parameters including gross alpha/beta gamma spectroscopy parameters; thermal ionization mass spectroscopy, fluorometric uranium, alpha spectroscopy-strontium 89/90; alpha spectrometry- thorium-237, uranium-234, 238, neptunium-237, plutonium-238, 239, 240, americium-241 and curium-242, 243, 244 and, liquid scintillation counting parameters-tritium.

Her other experience includes QA/QC consulting for private sector clients as well as for state and federal programs, development of SOPs for standard and non-standard methods, laboratory training and chemical analyses.

**Professional Qualifications**

Ms. Wright has 15 years experience in environmental/analytical chemistry. This includes 12 years extensive experience in analytical data validation (CLP and non-CLP). Ms Wright has 7 years project management experience and 5 years environmental/radiochemical analytical chemistry experience, which includes instrumentation and advance organic chemistry.

# QUALITY ASSURANCE/QUALITY CONTROL PLAN FOR DATA VALIDATION SERVICES

**Gay Webber**

Gay Webber has 13 years extensive experience in environmental/analytical chemistry. Her expertise in data validation includes all types of parameters such as volatile target compounds (TCL), semi-volatile target compounds, pesticide/PCBs, PCB-congeners, dioxins & furans, conventional general/wet chemistry, TAL metals, leachate and reactivity characteristics (TCLP) priority pollutants-metals & organics; radiological parameters including gross alpha/beta, gamma spectroscopy parameters; thermal ionization mass spectroscopy, fluorometric uranium, alpha spectroscopy-strontium 89/90; alpha spectrometry- thorium-237, uranium-234, 238, neptunium-237, plutonium-238, 239, 240, americuium-241 and curium-242, 243, 244 and, liquid scintillation counting parameters-tritium.

**Professional Qualifications**

Ms. Webber has 12 years experience in environmental/analytical chemistry. This includes 7 years extensive experience in analytical data validation (CLP and non-CLP). Ms Webber has 7 years project management experience and 7 years radiochemical data validation experience, which includes instrumentation.

**Beverly King**

Beverly King has 15 years extensive experience in environmental/analytical chemistry. Her expertise in data validation includes all types of parameters such as volatile target compounds (TCL), semi-volatile target compounds, pesticide/PCBs, PCB-congeners, dioxins & furans, conventional general/wet chemistry, TAL metals, leachate and reactivity characteristics (TCLP) priority pollutants-metals & organics. Radiological parameters including gross alpha/beta and liquid scintillation counting parameter-tritium.

**Professional Qualifications**

Ms. King has 15 years experience in environmental/analytical chemistry. This includes 12 years extensive experience in analytical data validation (CLP and non-CLP). Ms. King has 7 years project management experience and 4 years radiochemical data validation experience, which includes instrumentation.

**Denise L. McGuire**

**Experience Summary**

Denise McGuire has 15 years extensive experience in analytical chemistry, laboratory audits and general QA/QC data management. Her expertise in data validation includes all types of parameters such as volatile target compounds (TCL), semi-volatile target compounds, pesticide/PCBs, PCB-congeners, dioxins & furans, conventional general/wet chemistry, TAL metals, TCLP, priority pollutants-metals & organics; radiological parameters including gross alpha/beta gamma spectroscopy parameters; thermal ionization mass spectroscopy, fluorometric uranium, alpha spectroscopy-strontium 89/90; alpha spectrometry- thorium-237, uranium-234, 238, neptunium-237, plutonium-238, 239, 240, americium-241 and curium-242, 243, 244 and, liquid scintillation counting parameters-tritium.

She has extensive experience in the environmental consulting and laboratory services field. This experience has included data validation experience interpreting organic, inorganic, radiological, and chemical warfare agent analytical data; managing and procuring subcontracted analytical laboratories; coordinating field sampling crews; generation and review of site-specific Field Sampling Plans, Quality

# QUALITY ASSURANCE/QUALITY CONTROL PLAN FOR DATA VALIDATION SERVICES

Assurance Project Plans (QAPPs), Standard Operating Procedures (SOPs), and Remedial Investigation/Feasibility Study (RI/FS) and Data Validation reports; field data collection and environmental sampling; training and supervision of technical personnel; and field and laboratory auditing. In addition, I have designed a data management system for all projects producing analytical data. The data management system ensures quality data by incorporating quality assurance procedures, data tacking, documentation, and multitask data usage.

# CURRICULUM VITAE

Maxine Wright-Walters, PhD

## Educational Background

| | |
|---|---|
| University of Pittsburgh<br><br>Graduate School of Public Health<br>Pittsburgh, PA | 2008 PhD. Environmental and Occupational Health (EOH)/Environmental Health Sciences (EHS)<br>Dissertation Topic: Exposure Concentrations of Pharmaceutical Estrogens and Xenoestrogens in Municipal Wastewater Treatment Plant Sources, the Aquatic Environment and an Aquatic Health Risk Assessment of Bisphenol-A: Implications for Wildlife and Public Health |
| Duquesne University<br>Pittsburgh, PA | 1997 MSc. Environmental Science & Management<br>Internship: Allegheny County Emergency Preparedness, and Response Center, Pittsburgh PA |
| New York Institute of Technology,<br>Old Westbury, NY | 1989 BS, Chemistry, |
| University of Technology<br>(College of Arts Science and Technology)<br>Jamaica W.I. | 1986 Diploma in Pharmacy<br>Thesis: Antimicrobial Properties of the *Mimosa Pudica* and its effect on the *neissera gonorrhea* organism. |

## *Additional Training*

| | |
|---|---|
| RAB Certified EMS Lead Auditor | 1998 |
| American Chemical Society's short course in Microwave Enhanced Chemistry | 1997 |
| ISO 14000 Lead Auditor | 1997 |
| ISO 9000 auditor | 1997 |
| PACS data Validation | 1997 |
| Radiochemistry | 1989 |
| Radioactivity safety | 1989 |
| OSHA 40hr Health and Safety | 1987 |
| Data Validation | 1987 |

## Employment History

1991-present — President/Project Manager, EDV, Inc., PA

Responsible for the day to day operation and management of this small environmental consulting business. Duties include: Recruiting and mentoring of staff, budgeting, marketing, environmental consulting to include development of Data Quality Objectives (DQOs), development of QA/QC and laboratory training programs and manuals, laboratory auditing, remedial investigation/feasibility studies (RI/FS), QAPPs and SAPs development. Environmental Health Assessments and Risk Assessments, ISO 9000 consulting to include implementation, training and auditing of quality systems, ISO 14000 consulting to include implementation, training and auditing of Environmental Management Systems (EMS). Environmental Health and Occupational Safety training and consulting. Laboratory consulting to include development of Good Laboratory Practices (GLP), methods development, auditing and training. Data validation of all types of parameters such as volatile target compounds (TCL), semi-volatile target compounds, pesticide/PCBs, dioxins & furans, conventional general/wet chemistry, TAL metals, leachate and reactivity characteristics (TCLP) priority pollutants-metals & organics; radiological parameters including gross alpha/beta,. gamma spectroscopy parameters; thermal ionization mass spectroscopy, fluorometric uranium,, alpha spectroscopy-strontium 89/90; alpha spectrometry- thorium-237, uranium-234, 238, neptunium-237, plutonium-238, 239, 240, americium-241 and curium-242, 243, 244 and, liquid scintillation counting parameters-tritium. QA/QC consulting under various programs such as CERCLA (superfund), RCRA and Brownfield. Sales, proposal writing, and general project management. Conduct training courses at college and professional levels in areas such as: QMS (ISO 900:2000), EMS (ISO 14001) implementation, Introduction to ISO 14001, ISO14001 Internal auditing, laboratory auditing, organic/inorganic and radiochemical data validation and many others.

1990-1991 — Senior Chemist, Ecotek LSI, GA

As a senior chemist responsibilities included; method development, troubleshooting, writing of SOPs for Sample Preparation laboratory and QC department, writing of training manuals; QC compliance and surveillance audits; radiological and chemical data validation for parameters such gross alpha/beta, . gamma spectroscopy parameters; thermal ionization mass spectroscopy, fluorometric uranium spectroscopy-strontium 89/90; alpha spectrometry- thorium-237, uranium-234, 238, neptunium-237, plutonium-238, 239, 240, americium-241 and curium-242, 243, 244 and, liquid scintillation counting parameters-tritium; volatile target compounds (TCL), semi-volatile target compounds, pesticide/PCBs, dioxins & furans, conventional general/wet chemistry, TAL metals, leachate and reactivity characteristics ( TCLP) priority pollutants-metals & organics.

1989-1990 — Chief Chemist/Safety Officer, Syosset Labs, NY.

Responsibilities for this position included Quality Control, research, method development and validations. Training of new chemists to ensure familiarity and understanding of USP and In House methods. Testing of raw materials, in-process and finished products to confirm non-compliant results obtained by other chemists. Monitor the set-up and testing of all stability samples. Familiar with FDA regulations. Write SOPs, implementation of a Health and Safety program. Ensure the general safety of the building and all its employees within as per OSHA guidelines.

1987-1989 QC Chemist, Nytest Environmental, Port Washington, NY.
As a QC chemist duties included; wet chemistry analysis, organic and inorganic sample extraction and preparation, preparation of base-neutral, acid and pesticide spikes. Analysis of organic compounds via GC/GCMS, data validation of organic compounds such as BNAs, VOAs, Pest/PCBs.

## *Research*

"Antimicrobial Properties of the *Mimosa Pudica* and its effect on the *neissera gonorrhea* organism." Researched the Mimosa Pudica for its antimicrobial properties and looked at its effects on the *neissera gonorrhea* organism. This research was done in 1986 at the Microbiology Department of the University of the West Indies. It was a requirement for final year pharmacy students at the College of Arts Science and Technology.

Research in Organic Chemistry, investigating the different pathways in the synthesis of organic compounds with emphasis on Opium compounds. This Research was done in 1985-1986 at the College of Arts Science and Technology-Pharmacy Department.

Instrumentation research, working specifically with the Gas Chromatograph in determining the relationship between peak areas and concentrations of compounds. This research was done in 1988 and funded by the Life Science Department, New York Institute of Technology.

## *Professional Training/Teaching*

Consad Research, Pittsburgh, PA
Risk Assessment Expert for Department of Labor (DOL) review of risk assessment best practices within various agencies of the Federal government. Consult on drafting an exposure factors and risk characterization handbook that will be used to assist DOL in its risk assessment practices. 2008

GlaxoSmithKline, Pittsburgh, PA
Implementation of a complete ISO 14001 EMS to include executive briefings, baseline assessment, identification of aspects and impacts and chemical inventory and waste management. Internal and Lead auditor EMS training. Environmental Health and Occupational Safety training and consulting. 2006.

United States Department of Energy -National Environmental Technology Laboratory (NETL)
ISO 14001 training course in Implementation, Identifying Aspects and Impacts and Internal and Lead auditing. Environmental Health and Safety training course. 2003.

Tech-Seal, WV
Implementation of a complete EHS program. Auditor internal auditor training. Implementation of an ISO 9000 Quality Management System.2002.

Jefferson Community College, OH
ISO14001/EHS Implementation Consulting and Auditing as part of an ISO9000/14000 Consortia provided by the college to local businesses in the Weirton, WV area. 1998-2002.

Cutler-Hammer Technology, Center, Pittsburgh, PA (A former Westinghouse/DOD facility)
Implementation of a complete ISO 14001 EMS to include; executive briefings, baseline assessment, identification of aspects and impacts, and waste management. Internal and Lead auditor EMS training. Environmental Health and Safety Implementation, training and consulting. Conducted Chemical inventory and audit. The site has been certified in ISO 9001 and 14001. 2001.

Cutler-Hammer, Horsehead, NY (A former Westinghouse/DOD facility):
Implementation of a complete ISO 14001 EMS to include executive briefings, baseline assessment, identification of aspects and impacts and waste management. Internal and Lead auditor EMS training. Environmental Health and Safety training and consulting. The site has been certified in ISO 9001 and 14001. 2001.

Curtiss-Wright, EMD, Cheswick, PA (A former Westinghouse/DOD facility)
Planned and implemented records management system for Marketing, Engineering, and Human Resources using standardized databases for all functions. 2001.

***Graduate Appointments***

Graduate Assistant: Research Assistant for the Center for Healthy Communities. 2008

Graduate Assistant: Research Assistant for the Community Awareness Allegheny River Stewardship Project. 2007-2008

Graduate Research Assistant: Teaching and Research Assistant for the department of Environmental and Occupational Health 2001-2007

***Public Teaching Experience (Public Courses)***

Organic Data Validation, 1999-2006
Environmental Health and Safety Program Implementation, 1997-2007
Inorganic/Inorganic Data Validation, 1999-present
Radiochemical Data Validation, 2000-2006
ISO 14001 Implementation, 2002-2005
Environmental Management Systems Auditing, 2000-2004
Quality Management Systems, 2002

***Academic Teaching Experience***

University of Pittsburgh, PA. Co-Presenter/Co-Instructor: Community Awareness Presentation of the Allegheny River Stewardship Project, Alle-Kiski Health Foundation, Heinz Endowments and Highmark Foundation, 2007

University of Pittsburgh, PA. Guest Lecturer. Exposure Assessment, 2007

University of Pittsburgh, PA. Guest Lecturer. Dose-response Assessment, 2007

University of Pittsburgh, PA. Guest lecturer. Exposure Assessment for Baseline Risk Assessment for Superfund Sites, 2005

University of Pittsburgh, PA. Guest Lecturer. Risk Assessment. 2004-2005

University of Pittsburgh, PA. Guest Lecturer. Risk Communication. 2005

University of Pittsburgh, PA. Guest Lecturer. Chemical Fate and Transport in the Environment, 2004-2005

Duquesne University, PA. Co-instructor. Environmental Management Systems, 1998

Jefferson Community College, OH. Guest Lecturer. ISO 14000 Implementation. 1998-1999

***Publication***

**Maxine Wright-Walters** and Conrad Volz. Exposure of aquatic receptors to Bisphenol A: Evidence that current risk models may not be sufficiently protective. Ohio River Basin Conference, Pittsburgh,2008.

**Maxine Wright-Walters** and Conrad Volz. Pharmaceutical Estrogens and Xeno Estrogens in Municipal Wastewater Treatment Plants: Implications for Wildlife and Humans. Third National Conference on Environmental Science

and Technology. North Carolina A&T State University on September, 2007.pp.80. Abstracts Issue.

**Maxine Wright-Walters** and Conrad Volz. Pharmaceutical Estrogens and Xeno Estrogens in Municipal Wastewater Treatment Plants: Implications for Wildlife and Humans. "Proceedings of the 2007 National Conference on Environmental Science and Technology", p 103-113. Springer 2009.

Volz, CD., Dabney, B.,Cohen, P., Cude, C., Dooly, I., Kyprianou, R., Malecki, K., Richter, W., Schulman, A., Shaw, S., Vanderslice, J., **Walters, M.**, and Vyas, V., September 2007. Handling Left Censored Water Contaminant Data for Descriptive Statistics and (CDC), Environmental Public Health Tracking Network (EPHT) from the Water Working Group, Non-Detect Subgroup.

R.S. Carruth; **M. Wright-Walters**; N. B. Sussman; B.D. Goldstein. The Use of Relative Risk Greater Than 2.0 in the American Court System. August 2004. International Society of Environmental Epidemiology (ISEE) Conference Proceedings, New York, NY.

**Maxine M. Wright-Walters**, Nancy B. Sussman, Roger S. Day, Russellyn S. Carruth and Bernard D. Goldstein An Alternative Approach to Determining the Legal Criterion of "More likely than Not" in the Absence of Statistical Significance December 2004. Society of Risk Analysis (SRA) Conference Proceedings, Baltimore, MD.

Charles Tomljanovic, **Maxine Wright-Walters** & Jules Stephensky Anthropogenic Electromagnetic Fields (EMFs) and Cancer: A Perspective. "Risk: Health Safety & Environment "- Vol 8. Pp 287-289. Summer 1997.

***Additional Skill***

Knowledge and ability to operate the following instruments: GC, GC/MS, ICPMS, HPLC, AA, Potentiometer, Osmometer, Ion Analyzer, UV/IR Spectrophotometer, Mass Spectrophotometer and GPC (automated and manual). Knowledge in ISO 9000, ISO 14000 and regulatory programs such as CWA, CAA, TSCA, FIFRA RCRA, NEPA and CERCLA. Familiar with FDA, DOD, DOE and other federal programs. Proficient in the use of Statistical programs such as SAS and Stata.

***Professional Affiliation***

Member of the American Chemical Society
Member of the Air and Waste Management Association.
Member of the American Society for Quality
Society of Risk Analysis

# HEALTH AND SAFETY PLAN

## REMEDIAL INVESTIGATION/CORRECTIVE ACTIONS
**245-265 & 271 HOLLENBECK STREET AND 50 BALFOUR DRIVE**
**ROCHESTER, NEW YORK**

**NYSDEC SITE #828188**

**Prepared by:** Day Environmental, Inc.
1563 Lyell Avenue
Rochester, New York 14606

**Project No.:** 4845S-13

**Date:** January 2014

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ATTACHMENTS

## 1.0 INTRODUCTION

Day Environmental, Inc. (DAY) prepared this Health and Safety Plan (HASP) to outline policies and procedures to protect workers and the public from potential environmental hazards during the remedial investigation to be conducted at, and in the vicinity of, the property addressed 245-265 and 271 Hollenbeck Street and 50 Balfour Drive, City of Rochester, County of Monroe, New York (the Site). The Project Locus map presented as Figure 1 shows general location of the Site.

Although the HASP focuses on the specific work activities planned for the Site, it must remain flexible due to the nature of this work. Conditions may change and unforeseen situations can arise that require deviations from the original HASP.

### 1.1 SITE HISTORY/OVERVIEW

The Site is located in the City of Rochester, Monroe County, New York, and it consists of approximately 6.95 acres of land located on three contiguous parcels including:

- 245-265 Hollenbeck Street: This parcel is improved with an approximate 8,000 square foot one-story, brick building that is currently vacant.

- 271 Hollenbeck Street: This parcel is currently vacant, but in the past it contained a railroad spur line and it was used for commercial and industrial purposes.

- 50 Balfour Drive: This parcel is improved with an approximate 134,000 square foot combined one-story and two-story concrete block building with two attached one-story metal buildings. Sheet metal fabrication, stamping, and assembly operations are currently conducted within this building, and in the past it was used for various operations including printing/lithographing, tool and die, electroplating, and manufacturing of kitchen machines and retail/industrial scales .

A Phase I Environmental Site Assessment (Phase I ESA) completed by DAY in 1997 identified the following recognized environmental conditions (RECs) for the Site.

- Historical industrial usage of the Site;
- Possible filling, dumping, ground disturbance;
- Former underground storage tanks; and
- Spillage/leaking and staining.

Subsequent to the Phase I ESA, various intrusive studies were conducted at the Site to evaluate the RECs. The findings of these studies are summarized below.

- Field evidence of petroleum-like and/or chemical-like odors, and elevated photoionization detector (PID) readings were encountered in select test borings.

- Select soil samples collected during the studies were analyzed for Target Compound List (TCL) Volatile Organic Compounds (VOCs), Semi-Volatile Organic Compounds (SVOCs), total petroleum hydrocarbons (TPH), Target Analyte List metals and cyanide. The results of this testing indicted that:
    - Soil samples BH-01(6-8'), BH-11(0-2'), BH-13(6-8'), BH-15(9-11'), TB-101(6.5') and TB-117(9.9') contained concentrations of tricholoethene (TCE) that exceeded the Soil Cleanup Objective (SCO) for Unrestricted Use as listed in table 6.8(a) of 6 NYCRR Part 375.
    - Soil sample TB-202(10.5'), contained concentrations of cis 1,2-dichloroethene (cis 1,2-DCE) that exceeded the Unrestricted Use SCO.
    - Soil sample BH-11(0-2') contained a concentration of tetrachloroethene (PCE) that exceeded the Unrestricted Use SCO.
    - Soil sample BH-01(6-8') contained a concentration of xylenes that exceeded the Unrestricted Use SCO.
    - Soil sample SS-1(0-2'), contained concentration of acetone that exceeded the Unrestricted Use SCO.
    - Soil samples BH-01(2-4'), BH-04(0-2'), TB-101(6.5'), TB-114(2.0'), and TB-211 (11.0') contained concentrations of zinc that exceeded the Unrestricted Use SCO.
    - Soil sample BH-01(2-4') contained a concentration of mercury that exceeded the Unrestricted Use SCO.
    - Soil sample BH-04(0-2') contained a concentration of lead that exceeded the Unrestricted Use SCO.
- Groundwater samples collected from the monitoring wells installed at the Site historically contained concentrations of the following VOCs that exceeded New York State Department of Environmental Conservation (NYSDEC) Standard or Guidance Values on one, or more, occasion:.
    - monitoring well MW-1: PCE, TCE, and cis 1,2-DCE
    - monitoring well MW-2: TCE, and cis 1,2-DCE
    - monitoring well MW-3: PCE, TCE, cis 1,2-DCE, and Vinyl Chloride (VC)
    - monitoring well MW-4: TCE, trans 1,2-dichloroethene (trans 1,2-DCE), cis 1,2-DCE, and VC
    - monitoring well MW-5: TCE, trans 1,2-DCE, cis 1,2-DCE, and VC
    - monitoring well MW-6: TCE, trans 1,2-DCE, Cis 1,2-DCE, and VC
    - monitoring well MW-7 (bedrock well): PCE, TCE, Cis 1,2-DCE, and VC
    - monitoring well MW-8: cis 1,2-DCE, and VC
    - monitoring well MW-9: TCE, cis 1,2-DCE, and VC
    - monitoring well MW-10: TCE, cis 1,2-DCE, and VC
    - monitoring well MW-11: TCE, and cis 1,2-DCE
    - monitoring well MW-12: TCE, cis 1,2-DCE, and VC
    - monitoring well MW-13: TCE and cis 1,2-DCE

- monitoring well MW-14: TCE, cis 1,2-DCE, and VC
- monitoring well MW-16: TCE, trans 1,2-DCE cis 1,2-DCE, and VC
- monitoring well MW-17: TCE, trans 1,2-DCE, Cis 1,2-DCE, and VC
- monitoring well MW-18: cis 1,2-DCE and VC

## 1.2 PLANNED ACTIVITIES COVERED BY HASP

This HASP is intended to be used during intrusive environmental studies and subsequent remedial activities (if any) conducted at the Site that have the potential to encounter contaminated materials. Currently, identified activities to be completed at the Site that have the potential to encounter contaminated materials include:

- Site Preparation Activities
- Advancement of test borings and installation of groundwater monitoring wells
- Soil, Groundwater and Soil Vapor sample collection
- Handling of Investigation Derived Waste (IDW)

This HASP can be modified to cover other site activities as deemed appropriate. The owner of the property, its contractors, and other site workers will be responsible for the development and/or implementation of health and safety provisions associated with site activities.

## 2.0 KEY PERSONNEL AND MANAGEMENT

The Project Manager (PM) and Site Safety Officer (SSO) are responsible for formulating health and safety requirements, and implementing the HASP.

### 2.1 PROJECT MANAGER

The PM has the overall responsibility for the project and will coordinate with the SSO to ensure that the goals of the project are attained in a manner consistent with the HASP requirements.

### 2.2 SITE SAFETY OFFICER

The SSO has responsibility for administering the HASP relative to site activities, and will be in the field while activities are in progress. The SSO's operational responsibilities will be monitoring, including personal and environmental monitoring, ensuring personal protective equipment (PPE) maintenance, and identification of protection levels. The air monitoring data obtained by the SSO will be available for review by regulatory agencies and other on-site personnel.

### 2.3 EMPLOYEE SAFETY RESPONSIBILITY

Each employee is responsible for personal safety as well as safety of others in the area. The employee will use the equipment provided in a safe and responsible manner as directed by the SSO.

### 2.4 KEY SAFETY PERSONNEL

The following individuals are anticipated to share responsibility for health and safety of DAY representatives at the Site.

| | |
|---|---|
| DAY Project Manager | Nathan Simon |
| DAY Site Safety Officer | Charles Hampton, Christie Sobol or Will Batiste |

## 3.0 SAFETY RESPONSIBILITY

Contractors, consultants, state or local agencies, or other parties, and their employees, involved with this project will be responsible for their own safety while on-site. Their employees will be required to understand the information contained in this HASP, and must follow the recommendations that are made in this document. As an alternative, contractors, consultants, state or local agencies, or other parties, and their employees, involved with this project can utilize their own health and safety plan for this project as long as it is found acceptable to the New York State Department of Health (NYSDOH), NYSDEC and/or the Monroe County Department of Public Health (MCDPH).

## 4.0 JOB HAZARD ANALYSIS

There are many hazards associated with environmental work on a site, and this HASP discusses some of the anticipated hazards for this Site. The hazards listed below deal specifically with those hazards associated with the management of potentially contaminated media (e.g. soil, fill, groundwater, etc.).

### 4.1 CHEMICAL HAZARDS

Chemical substances can enter the unprotected body by inhalation, skin absorption, ingestion, or injection (i.e., a puncture wound, etc.). A contaminant can cause damage to the point of contact or can act systemically, causing a toxic effect at a part of the body distant from the point of initial contact.

A list of selected constituents that have been detected at the Site at concentrations that exceed soil or groundwater standards, criteria and guidance (SCG) values are presented below. This list also presents the Occupational Safety and Health Administration (OSHA) permissible exposure limits (PELs), National Institute for Occupational Safety and Health (NIOSH) recommended exposure limits (RELs), and NIOSH immediately dangerous to life or health (IDLH) levels.

| CONSTITUENT | OSHA PEL | NIOSH PEL | IDLH |
|---|---|---|---|
| Tetrachloroethene (PCE) | 678 $mg/m^3$ | Minimize workplace exposure concentrations | 1017 $mg/m^3$ |
| Trichloroethene (TCE) | 537 $mg/m^3$ | 134.25 $mg/m^3$ | 5370 $mg/m^3$ |
| trans 1,2- Dichloroethene (trans 1,2-DCE) | 790 $mg/m^3$ | 790 $mg/m^3$ | 3970 $mg/m^3$ |
| cis 1,2- Dichloroethene (cis 1,2-DCE) | 790 $mg/m^3$ | 790 $mg/m^3$ | 3970 $mg/m^3$ |
| Vinyl Chloride | 2.56 $mg/m^3$ | NA | NA |
| Acetone | 2400 $mg/m^3$ | 590 $mg/m^3$ | 2500 $mg/m^3$ |
| 2-hexanone | 410 $mg/m^3$ | 4.10 $mg/m^3$ | 6560 $mg/m^3$ |
| Methylene Chloride | 86.7 $mg/m^3$ | NA | 7981 $mg/m^3$ |
| n-Butylbenzene | NA | NA | NA |
| n-propylbenzene | NA | NA | NA |
| Isopropylbenzene | 245 $mg/m^3$ | 245 $mg/m^3$ | 4428 $mg/m^3$ |
| 1,2,4-Trimethylbenzene | NA | 125 $mg/m^3$ | NA |
| Naphthalene | 50 $mg/m^3$ | 50 $mg/m^3$ | 1310 $mg/m^3$ |
| Toluene | 1074 $mg/m^3$ | 375 $mg/m^3$ | 1885 $mg/m^3$ |

| | | | |
|---|---|---|---|
| Total Xylenes[1] | 435 mg/m$^3$ | 435 mg/m$^3$ | 3906 mg/m$^3$ |
| Lead | 0.05 mg/m$^3$ | 0.05 mg/m$^3$ | 100 mg/m$^3$ |
| Mercury | 0.1 mg/m$^3$ | 0.05 mg/m$^3$ | 10 mg/m$^3$ |
| Zinc | 5 mg/m$^3$ | 5 mg/m$^3$ | 500 mg/m$^3$ |

NA = Not Available

The potential routes of exposure for these analytes and chemicals include inhalation, ingestion, skin absorption and/or skin/eye contact. The potential for exposure through any one of these routes will depend on the activity conducted. The most likely routes of exposure for the anticipated environmental activities at the Site include inhalation and skin/eye contact.

## 4.2 Physical Hazards

There are physical hazards associated with this project, which might compound the chemical hazards. Hazard identification, training, adherence to the planned environmental measures, and careful housekeeping can prevent many problems or accidents arising from physical hazards. Potential physical hazards associated with this project and suggested preventative measures include:

- Slip/Trip/Fall Hazards – Some areas may have wet or frozen surfaces that will greatly increase the possibility of inadvertent slips. Caution must be exercised when using steps and stairs due to slippery surfaces in conjunction with the fall hazard. Good housekeeping practices are essential to minimize the trip hazards.
- Small Quantity Flammable Liquids – Small quantities of flammable liquids will be stored in "safety" cans and labeled according to contents.
- Electrical Hazards – Electrical devices and equipment shall be de-energized prior to working near them. All extension cords will be kept out of water, protected from crushing, and observed regularly to ensure structural integrity. Temporary electrical circuits will be protected with ground fault circuit interrupters. Only qualified electricians are authorized to work on electrical circuits. Heavy equipment (e.g., excavator, backhoe, drill rig) shall not be operated within 10 feet of high voltage lines, unless proper protection form the high voltage lines is provided by the appropriate utility company.
- Noise – Work around large equipment often creates excessive noise. The effects of noise can include:

- Workers being startled, annoyed, or distracted.
- Physical damage to the ear resulting in pain, or temporary and or/permanent hearing loss.
- Communication interference that may increase potential hazards due to the inability to warn of danger and proper safety precautions to be taken.

[1] Exposure limits for Total Xylenes was based on the lowest limits among the Xylene isomers (o-Xylene, m-Xylene, p-Xylene).

Proper hearing protection will be worn as deemed necessary. In general, feasible administrative or engineering controls shall be utilized when on-site personnel are subjected to noise exceeding an 8-hour time weighted average (TWA) sound level of 90 decibels on the A-weighted scale (dBA). In addition, whenever employee noise exposures equal or exceed an 8-hour TWA sound level of 85 dBA, employers shall administer a continuing, effective hearing conservation program as described in the OSHA Regulation 29 Code of Federal Rules (CFR) Part 1910.95.

- Heavy Equipment – Each morning before start-up, heavy equipment will be checked to ensure safety equipment and devices are operational and ready for immediate use.
- Subsurface and Overhead Hazards – Before any excavation activity, efforts will be made to determine whether underground utilities and potential overhead hazards will be encountered. Underground utility clearance must be obtained prior to subsurface work.

## 4.2 ENVIRONMENTAL HAZARDS

Environmental factors such as weather, wild animals, insects, snakes and irritant plants can pose a hazard when performing outdoor tasks. The SSO shall make reasonable efforts to alleviate these hazards should they arise.

### *4.3.1 Heat Stress*

The combination of warm ambient temperature and protective clothing increases the potential for heat stress. In particular,

- Heat rash
- Heat cramps
- Heat exhaustion
- Heat stroke

Site workers will be encouraged to increase consumption of water or electrolyte-containing beverages such as Gatorade® when the potential for heat stress exists. In addition, workers are encouraged to take rests whenever they feel any adverse effects that may be heat-related. The frequency of breaks may need to be increased upon worker recommendation to the SSO.

### *4.3.2 Exposure to Cold*

With outdoor work in the winter months, the potential exists for hypothermia and frostbite. Protective clothing greatly reduces the possibility of hypothermia in workers. However, personnel will be instructed to wear warm clothing and to stop work to obtain more clothing if they become too cold. Employees will also be advised to change into dry clothes if their clothing becomes wet from perspiration or from exposure to precipitation.

## 5.0 SITE CONTROLS

To prevent migration of contamination caused through tracking by personnel or equipment, work areas, and personal protective equipment staging/decontamination areas will be specified prior to beginning operations.

### 5.1 SITE ZONES

In the area where contaminated materials present the potential for worker exposure (work zone), personnel entering the area must wear the mandated level of protection for the area. A "transition zone" shall be established where personnel can begin and complete personal and equipment decontamination procedures. This can reduce potential off-site migration of contaminated media. Contaminated equipment or clothing will not be allowed outside the transition zone (e.g., on clean portions of the Site) unless properly containerized for disposal. Operational support facilities will be located outside the transition zone (i.e., in a "support zone"), and normal work clothing and support equipment are appropriate in this area. If possible, the support zone should be located upwind of the work zone and transition zone.

### 5.2 GENERAL

The following items will be requirements to protect the health and safety of workers during implementation of activities that disturb contaminated material.

- Eating, drinking, chewing gum or tobacco, smoking, or any practice that increased the probability of hand to mouth transfer and ingestion of contamination shall not occur in the work zone and/or transition zone during disturbance of contaminated material.
- Personnel admitted in the work zone shall be properly trained in health and safety techniques and equipment usage.
- No personnel shall be admitted in the work zone without the proper safety equipment.
- Proper decontamination procedures shall be followed before leaving the Site.

## 6.0 PROTECTIVE EQUIPMENT

This section addresses the various levels of PPE, which are or may be required at this job site. Personnel entering the work zone and transition zone shall be trained in the use of the anticipated PPE to be utilized.

### 6.1 ANTICIPATED PROTECTION LEVELS

The following table summarizes the protection levels (refer to Section 6.2) anticipated for tasks to be implemented during this project.

| TASK | PROTECTION LEVEL | COMMENTS/MODIFICATIONS |
|---|---|---|
| Site mobilization | D | |
| Site preparation | D | |
| Intrusive work | C/Modified D/D | Based on air monitoring, and SSO discretion. |
| Decontamination Area | Modified D/D | |
| Site breakdown and demobilization | D | |

It is anticipated that work conducted as part of this project will be performed in Level D or modified Level D PPE. If conditions are encountered that require Level A or Level B PPE, the work will immediately be stopped. The appropriate government agencies (e.g., NYSDEC, NYSDOH, MCDPH, etc.) will be notified and the proper health and safety measures will be implemented (e.g., develop and implement engineering controls, upgrade in PPE, etc.). If conditions are encountered that require Level C PPE, the work will be temporarily suspended and the work site will be evaluated to limit exposure prior to implementing Level C PPE.

### 6.2 PROTECTION LEVEL DESCRIPTIONS

This section lists the minimum requirements for each protection level. Modifications to these requirements can be made upon approval of the SSO. If Level A, Level B, and/or Level C PPE is required, Site personnel that enter the work zone and/or transition zone must be properly trained and certified in the use of those levels of PPE.

#### *6.2.1 Level D*

Level D consists of the following:

- Safety glasses
- Hard hat when working with heavy equipment
- Steel-toed or composite-toed work boots

- Protective gloves during sampling or handling of potentially contaminated media
- Work clothing as prescribed by weather

### *6.2.2 Modified Level D*

Modified Level D consists of the following:

- Safety glasses with side shields
- Hard hat when working with heavy equipment
- Steel-toed or composite-toed work boots
- Protective gloves during sampling or handling of potentially contaminated media
- Outer protective wear, such as Tyvek coverall [Tyveks (Sarans) and polyvinyl chloride (PVC) acid gear will be required when workers have a potential to be exposed to impacted liquids or impacted particulates]

### *6.2.3 Level C*

Level C consists of the following:

- Air-purifying respirator with appropriate cartridges
- Outer protective wear, such as Tyvek coverall [Tyveks (Sarans) and PVC acid gear will be required when workers have a potential to be exposed to impacted liquids or particulates]
- Hard hat when working with heavy equipment
- Steel-toed or composite-toed work boots
- Nitrile, neoprene, or PVC overboots, if appropriate
- Nitrile, neoprene, or PVC gloves, if appropriate
- Face shield (when projectiles or splashes pose a hazard)

### *6.2.4 Level B*

Level B protection consists of the items required for Level C protection with the exception that an air-supplied respirator is used in place of the air-purifying respirator. Level B PPE is not anticipated to be required during this project. If the need for level B PPE becomes evident, activities in the affected area will be stopped until conditions are further evaluated, and any necessary modifications to the HASP have been approved by the PM and SSO. Subsequently, the appropriate safety measures (including Level B PPE) must be implemented prior to commencing site activities.

### *6.2.5 Level A*

Level A protection consists of the items required for Level B protection with the addition of a fully encapsulating, vapor-proof suit capable of maintaining positive pressure. Level A PPE is not anticipated to be required during this project. If the need for level A PPE becomes evident, activities in the affected area will be stopped until conditions are further evaluated, and any necessary modifications to the HASP have been approved by the PM and SSO. Subsequently, the appropriate safety measures (including Level A PPE) must be implemented prior to commencing site activities.

## 6.3 RESPIRATORY PROTECTION

Any respirator used will meet the requirements of the OSHA 29 CFR 1910.134. Both the respirator and cartridges specified shall be fit-tested prior to use in accordance with OSHA regulations (29 CFR 1910). Air purifying respirators shall not be worn if contaminant levels exceed designated respirator cartridge use concentrations. The workers will wear respirators with approval for: organic vapors less than 1,000 ppm; and dusts, fumes and mists with a TWA less than 0.05 milligrams per cubic meter ($mg/m^3$).

No personnel who have facial hair, which interferes with respirator sealing surface, will be permitted to wear a respirator and will not be permitted to work in areas requiring respirator use.

Only workers who have been certified by a physician as being physically capable of respirator usage shall be issued a respirator. Personnel unable to pass a respiratory fit test or without medical clearance for respirator use will not be permitted to enter or work in areas that require respirator protection.

## 7.0 DECONTAMINATION PROCEDURES

This section describes the procedures necessary to ensure that both personnel and equipment are free from contamination when they leave the work site.

### 7.1 PERSONNEL DECONTAMINATION

Personnel involved with activities that involve disturbing contaminated media will follow the decontamination procedures described herein to ensure that material which workers may have contacted in the work zone and/or transition zone does not result in personal exposure and is not spread to clean areas of the Site. This sequence describes the general decontamination procedure. The specific stages can vary depending on the Site, the task, and the protection level, etc.

1. Leave work zone and go to transition zone
2. Remove soil/debris from boots and gloves
3. Remove boots
4. Remove gloves
5. Remove Tyvek suit and discard, if applicable
6. Remove and wash respirator, if applicable
7. Go to support zone

### 7.2 EQUIPMENT DECONTAMINATION

In order to reduce the potential for cross-contamination of samples collected during this project, the following procedures will be implemented to ensure that the data collected (primarily the laboratory data) is acceptable.

It is anticipated that most of the materials used to assist in obtaining samples will be disposable one-time use materials (e.g., sampling containers, bailers, rope, pump tubing, latex gloves, etc.). However, when equipment must be re-used (e.g., drill rigs, static water level indicator, split spoon samplers, etc.), it will be decontaminated by at least one of the following methods:

- Steam clean the equipment within a dedicated decontamination area; or
- Rough wash in tap water; wash in mixture of tap water and Alconox-type soap; double rinse with deionized or distilled water; and air dry and/or dry with clean paper towel.

The decontamination area will be set-up in a location to minimize disturbance to properties surrounding the work area.

### 7.3 DISPOSAL

Disposable clothing will be disposed in accordance with applicable regulations. Liquids (e.g., decontamination water, etc.) or solids (e.g., soil) generated by remedial activities will be disposed in accordance with applicable regulations.

## 8.0 AIR MONITORING

During activities that have the potential to disturb contaminated soil, fill material, or groundwater, air monitoring will be conducted in order to determine airborne particulate and contamination levels. This ensures that respiratory protection is adequate to protect personnel against the chemicals that are encountered and that chemical contaminants are not migrating off-site. Additional air monitoring may be conducted at the discretion of the SSO. Readings will be recorded and be available for review.

The following chart describes the direct reading instrumentation that will be utilized and appropriate action levels.

| Monitoring Device | Action Level | Response/Level of PPE |
|---|---|---|
| PID Volatile Organic Compound Meter | < 1 ppm in breathing zone, sustained 5 minutes | Level D |
| | 1-25 ppm in breathing zone, sustained 5 minutes | Cease work, implement measures to reduce air emissions when the work is performed, etc. If levels can not be brought below 1 ppm in the breathing zone, then upgrade PPE to Level C |
| | 26-250 ppm in breathing zone, sustained 5 minutes | Level B, Stop work, evaluate the use of engineering controls, etc. |
| | >250 ppm in breathing zone | Level A, Stop work, evaluate the use of engineering controls, etc. |
| RTAM Particulate Meter | < 100 $\mu g/m^3$ over an integrated period not to exceed 15 minutes. | Continue working |
| | > 100 $\mu g/m^3$ | Cease work, implement dust suppression, change in way work performed, etc. If levels can not be brought below 150 $\mu g/m^3$, then upgrade PPE to Level C |

### 8.1 PARTICULATE MONITORING

During activities where contaminated materials (e.g., soil, fill, etc.) may be disturbed, air monitoring will include real-time monitoring for particulates using a real-time aerosol monitor (RTAM) particulate meter at the perimeter of the work zone in accordance with the Final DER-10 Technical Guidance for Site Investigation and Remediation dated May 2010. DER-10 uses an action level of 100 $\mu g/m^3$ (0.10 $mg/m^3$) over background conditions for an integrated period not

to exceed 15 minutes. If the action level is exceeded, or if visible dust is encountered, then work shall be discontinued until corrective actions are implemented. Corrective actions may include dust suppression, change in the way work is performed, and/or upgrade of personal protective equipment.

## 8.2 VOLATILE ORGANIC COMPOUND MONITORING

During activities where contaminated materials may be disturbed, a PID will be used to monitor total VOCs in the ambient air. The PID will prove useful as a direct reading instrument to aid in determining if current respiratory protection is adequate or needs to be upgraded. The SSO will take measurements before operations begin in an area to determine the amount of VOCs naturally occurring in the air. This is referred to as a background level. Levels of VOCs will periodically be measured in the air at active work sites, and at the transition zone when levels are detected above background in the work zone.

## 8.3 COMMUNITY AIR MONITORING PLAN

During activities that have the potential to disturb contaminated soil, fill material, or groundwater, this Community Air Monitoring Plan (CAMP) will be implemented. The CAMP includes real-time monitoring for VOCs and particulates (i.e., dust) at the downwind perimeter of each designated work area when activities with the potential to release VOCs or dust are in progress at the Site. This CAMP is based on the NYSDOH Generic CAMP included as Appendix 1A of the NYSDEC document titled *"DER-10, Technical Guidance for Site Investigation and Remediation"* dated May 2010. The CAMP is not intended for use in establishing action levels for worker respiratory protection. Rather, its intent is to provide a measure of protection for the downwind community (i.e., off-site receptors including residences/businesses and on-site workers not directly involved with the subject work activities) from potential airborne contaminant releases as a direct result of project activities.

Continuous monitoring will be conducted during ground intrusive activities involving potentially contaminated soil, fill material or groundwater. Ground intrusive activities include, but are not limited to, test pitting or trenching, advancement/installation of test borings or monitoring wells, etc.

Periodic monitoring for VOCs will be conducted during non-intrusive activities involving potentially contaminated soil, fill material or groundwater where deemed appropriate (e.g., during collection of soil samples or groundwater samples, etc.).

### *8.3.1 VOC Monitoring, Response Levels, and Actions*

VOCs must be monitored at the downwind perimeter of the immediate work area (i.e., the work zone) on a continuous basis or as otherwise specified. Upwind concentrations should be measured at the start of each workday and periodically thereafter to establish background conditions. The monitoring work should be performed using equipment appropriate to measure the types of contaminants known or suspected to be present. The equipment should be calibrated at least daily for the contaminant(s) of concern or for an appropriate surrogate. The equipment

should be capable of calculating 15-minute running average concentrations, which will be compared to the levels specified below.

- If the ambient air concentration of total organic vapors at the downwind perimeter of the work area or exclusion zone exceeds 5 ppm above background for the 15-minute average, work activities must be temporarily halted and monitoring must be continued. If the total organic vapor level readily decreases (per instantaneous readings) below 5 ppm over background, work activities can resume with continued monitoring.

- If total organic vapor levels at the downwind perimeter of the work area or exclusion zone persist at levels in excess of 5 ppm over background but less than 25 ppm, work activities must be halted, the source or vapors identified, corrective actions taken to abate emissions, and monitoring continued. After these steps, work activities can resume provided that the total organic vapor level 200 feet downwind of the exclusion zone or half the distance to the nearest potential receptor or residential/commercial structure, whichever is less (but in no case less than 20 feet), is below 5 ppm over background for the 15-minute average.

- If the organic vapor level is above 25 ppm at the perimeter of the work area, activities must be shutdown.

The 15-minute readings must be recorded and made available for NYSDEC and NYSDOH personnel to review. Instantaneous readings, if any, used for decision purposes should also be recorded.

### *8.3.2 Particulate Monitoring, Response Levels, and Actions*

Particulate concentrations should be monitored continuously at the upwind perimeter of the work zone at temporary particulate monitoring stations. Upwind concentrations should be measured at the start of each workday and periodically thereafter to establish background conditions. The particulate monitoring should be performed using real-time monitoring equipment capable of measuring particulate matter less than 10 micrometers in size (PM-10) and capable of integrating over a period of 15 minutes (or less) for comparison to the airborne particulate action level. The equipment must be equipped with an audible alarm to indicate exceedance of the action level. In addition, fugitive dust migration should be visually assessed during work activities.

- If the downwind PM-10 particulate level is 100 micrograms per cubic meter ($\mu g/m^3$) greater than background (upwind perimeter) for the 15-minute period or if airborne dust is observed leaving the work area, then dust suppression techniques must be employed. Work may continue with dust suppression techniques provided that downwind PM-10 particulate levels do not exceed 150 $\mu g/m^3$ above the upwind level and provided that no visible dust is migrating from the work area.

- If, after implementation of dust suppression techniques, downwind PM-10 particulate levels are greater than 150 $\mu g/m^3$ above the upwind level, work must be stopped and a re-evaluation of activities initiated. Work can resume provided that dust suppression

measures and other controls are successful in reducing the downwind PM-10 particulate concentration to within 150 $\mu g/m^3$ of the upwind level and in preventing visible dust migration.

Readings will be recorded and made available for review.

## 9.0 EMERGENCY CONTINGENCY PLAN

This section presents the emergency contingency plan (ECP) describing the procedures to be performed in the event of an emergency (e.g., fire, spill, tank/drum release, etc.). To provide first-line assistance to field personnel in the case of illness or injury, the following items will be made immediately available on the Site:

- First-aid kit;
- Portable emergency eye wash; and
- Supply of clean water.

### 9.1 EMERGENCY TELEPHONE NUMBERS

The following telephone numbers are listed in case there is an emergency at the Site:

| | |
|---|---|
| Fire/Police Department: | 911 |
| Poison Control Center: | (800) 222-1222 |
| NYSDEC | |
| Region 8: Environmental Remediation | (585) 226-5349 |
| Spill Hotline | (800) 457-7362 |
| NYSDOH | |
| Public Health Duty Officer | (866) 881-2809 |
| MCDPH | |
| Public Health Engineering | (585) 753-5476 |
| OBI, LCC | |
| Justin Kretzmann | (585) 266-3060 x167 |
| Michael McAlpin | (585) 287-9342 |
| DAY ENVIRONMENTAL, INC. | |
| Nathan Simon | (585) 454-0210 x109 |
| Raymond Kampff | (585) 454-0210 x108 |
| NEAREST HOSPITAL: | Rochester General Hospital<br>1425 Portland Avenue, Rochester, NY 14621<br>(585) 922-4000 (Main)<br>(585) 922-2000 (Emergency Department) |
| Directions to the Hospital: | Head east on Balfour Drive toward Hollenbeck Street for approximately 0.1 miles. Turn left on Hollenbeck Street and proceed approximately 0.3 miles. Turn right on Norton Street and proceed approximately 1.2 |

miles and then turn left on Carter Street. Turn right into Rochester General Hospital after approximately 0.3 miles (Figure 1).

## 9.2 EVACUATION

During activities involving potential disturbance of contaminated soil, fill material, or groundwater, a log of each individual entering and leaving the Site will be kept for emergency accounting practices. Although unlikely, it is possible that a site emergency could require evacuating personnel from the Site. If required, the SSO will give the appropriate signal for site evacuation (i.e., hand signals, alarms, etc.).

All personnel shall exit the Site and shall congregate in an area designated by the SSO. The SSO shall ensure that all personnel are accounted for. If someone is missing, the SSO will alert emergency personnel. The appropriate government agencies will be notified as soon as possible regarding the evacuation, and any necessary measures that may be required to mitigate the reason for the evacuation.

## 9.3 MEDICAL EMERGENCY

In the event of a medical emergency involving illness or injury to one of the on-site personnel, Emergency Medical Services (EMS) and the appropriate government agencies should be notified immediately. The area in which the injury or illness occurred shall not be entered until the cause of the illness or injury is known. The nature of injury or illness shall be assessed. If the victim appears to be critically injured, administer first aid and/or cardio-pulmonary resuscitation (CPR) as needed. If appropriate, instantaneous real-time air monitoring shall be done in accordance with air monitoring outlined in Section 8.0 of this HASP.

## 9.4 CONTAMINATION EMERGENCY

It is unlikely that a contamination emergency will occur; however, if such an emergency does occur, the specific work area shall be shut down and immediately secured. If an emergency rescue is needed, notify Police, Fire Department and EMS units immediately. Advise them of the situation and request an expedient response. The appropriate government agencies shall be notified immediately. The area in which the contamination occurred shall not be entered until the arrival of trained personnel who are properly equipped with the appropriate PPE and monitoring instrumentation as outlined in Section 8.0 of this HASP.

## 9.5 FIRE EMERGENCY

In the event of a fire on-site, all non-essential site personnel shall be evacuated to a safe, secure area. The Fire Department will be notified immediately, and advised of the situation and the identification of any hazardous materials involved. The appropriate government agencies shall be notified as soon as possible.

The four classes of fire along with their constituents are as follows:

Class A: Wood, cloth, paper, rubber, many plastics, and ordinary combustible materials.

Class B: Flammable liquids, gases and greases.

Class C: Energized electrical equipment.

Class D: Combustible metals such as magnesium, titanium, sodium, potassium.

Small fires on-site may be actively extinguished; however, extreme care shall be taken while in this operation. Approaches to the fire shall be done from the upwind side if possible. Distance from on-site personnel to the fire shall be close enough to ensure proper application of the extinguishing material but far enough away to ensure that the personnel are safe. The proper extinguisher shall be utilized for the Class(es) of fire present on the site. If possible, the fuel source shall be cut off or separated from the fire. Care must be taken when performing operations involving the shut-off of valves and manifolds, if present.

Examples of proper extinguishing agent as follows:

Class A: Water
Water with 1% AFFF Foam (Wet Water)
Water with 6% AFFF or Fluorprotein Foam
ABC Dry Chemical

Class B: ABC Dry Chemical
Purple K
Carbon Dioxide
Water with 6% AFFF Foam

Class C: ABC Dry Chemical
Carbon Dioxide

Class D: Metal-X Dry Powder

No attempt shall be made against large fires these shall be handled by the Fire Department.

### 9.6 SPILL OR AIR RELEASE

In the event of a spill or air release of hazardous materials on-site, the specific area of the spill or release shall be shut down and immediately secured. The area in which the spill or release occurred shall not be entered until the cause can be determined and site safety can be evaluated. Non-essential site personnel shall be evacuated to a safe and secure area. The appropriate government agencies shall be notified as soon as possible. The spilled or released material shall be immediately indentified and appropriate containment measures shall be implemented, if

possible. Real-time air monitoring shall be implemented as outlined in Section 8.0 of this HASP. If the materials are unknown, Level B protection is mandatory. If warranted, samples of the materials shall be acquired to facilitate identification.

### 9.7 LOCATING CONTAINERIZED WASTE AND/OR UNDERGROUND STORAGE TANKS

In the event that unanticipated containerized waster (e.g., drums) and/or underground storage tanks (USTs) are located during investigation and/or subsequent remedial activities, the work must be stopped in the specific area until site safety can be evaluated and addressed. Non-essential Site personnel shall not work in the immediate area until conditions including possible exposure hazards are addressed. The appropriate government agencies shall be notified as soon as possible. The SSO shall monitor the area as outlined in Section 8.0 of this HASP.

Prior to handling, unanticipated containers will be visually assessed by the SSO to gain as much information as possible about their contents. As a precautionary measure, personnel shall assume that unlabelled containers and/or tanks contain hazardous materials until their contents are characterized. To the extent possible based upon the nature of the containers encountered, actions may be taken to stabilize the area and prevent migration (e.g., placement of berms, etc.). Subsequent to initial visual assessment and any required stabilization, properly trained personnel will sample, test, remove, and dispose of any containers and/or tanks, and their contents. After visual assessment and air monitoring, if the material remains unknown, Level B protection (or higher) is mandatory.

## 10.0 ABBREVIATIONS

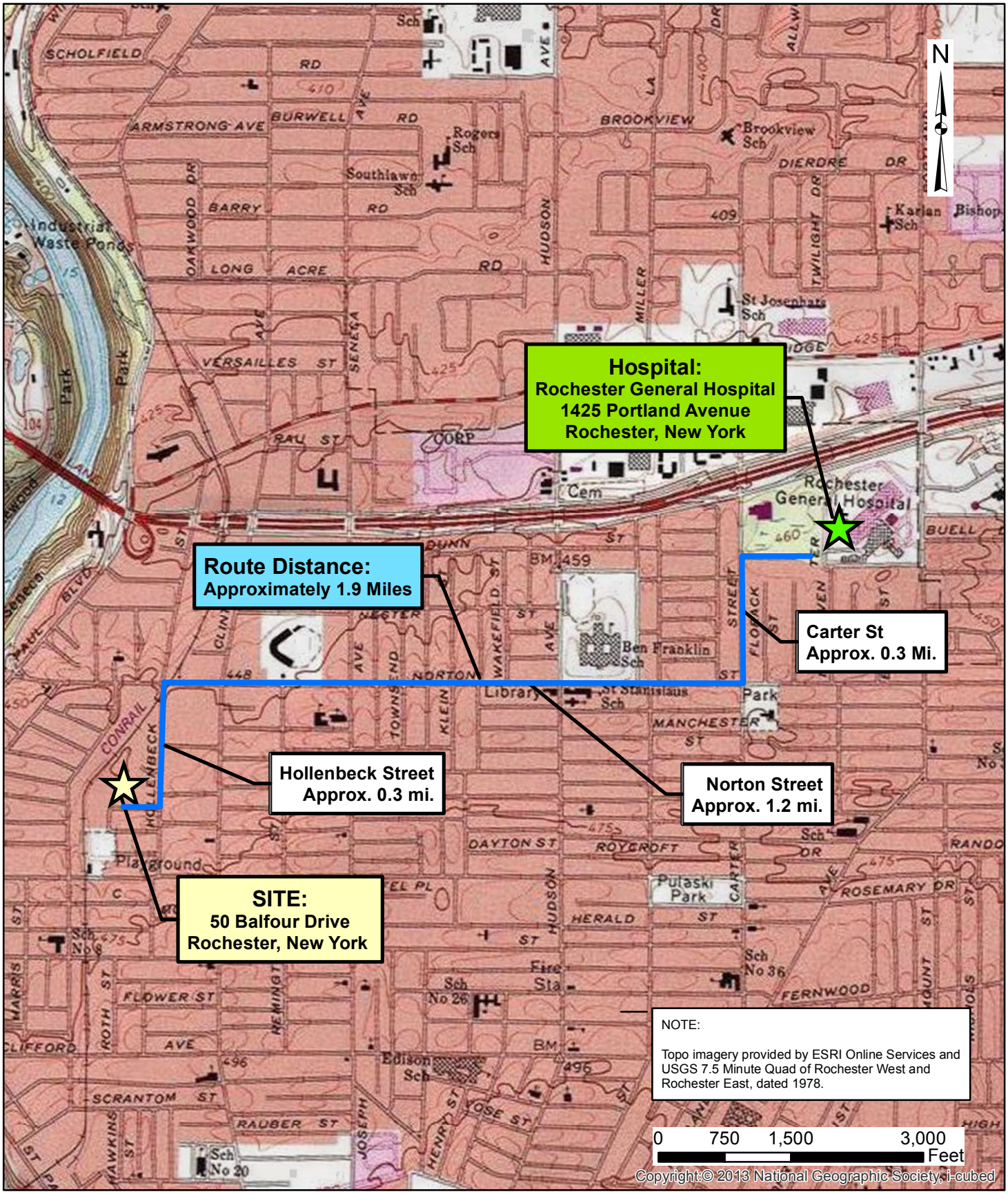
| | |
|---|---|
| AFFF | Aqueous Film Forming Foams |
| bgs | Below Ground Surface |
| CAMP | Community Air Monitoring Program |
| CFR | Code of Federal Regulations |
| cis 1,2-DCE | cis 1,2-dichloroethene |
| CPR | Cardio-Pulmonary Resuscitation |
| DAY | Day Environmental, Inc. |
| dBA | Decibels on the A-Weighted Scale |
| ECP | Emergency Contingency Plan |
| EMS | Emergency Medical Service |
| ESA | Environmental Site Assessment |
| HASP | Health and Safety Plan |
| IDLH | Immediately Dangerous to Life or Heath |
| IDW | Investigative Derived Waste |
| MCDPH | Monroe County Department of Public Health |
| $mg/m^3$ | Milligram Per Meter Cubed |
| NIOSH | National Institute for Occupational Safety and Health |
| NYSDEC | New York State Department of Environmental Conservation |
| NYSDOH | New York State Department of Health |
| OSHA | Occupational Safety and Health Administration |
| PCE | Tetrachloroethene |
| PEL | Permissible Exposure Limit |
| Phase I ESA | Phase I Environmental Site Assessment |
| PID | Photoionization Detector |
| PM | Project Manager |
| PM-10 | Particulate Matter Less Than 10 Micrometers In Diameter |
| PPE | Personal Protection Equipment |
| ppm | Parts Per Million |
| PVC | Polyvinyl Chloride |
| QAPP | Quality Assurance Project Plan |
| REC | Recognized Environmental Condition |
| REL | Recommended Exposure Limit |
| RTAM | Real-Time Aerosol Monitor |
| SCG | Standards, Criteria and Guidance |
| SCO | Soil Cleanup Objective |
| SSO | Site Safety Officer |
| SVOC | Semi-Volatile Organic Compound |
| TAL | Target Analyte List |
| TCE | Trichloroethene |
| TIC | Tentatively Identified Compound |
| TCL | Target Compound List |
| Trans 1,2 DCE | trans 1,2-dicloroethene |
| TPH | Total Petroleum Hydrocarbons |
| TWA | Time-Weighted Average |

| | |
|---|---|
| UST | Underground Storage Tank |
| $\mu g/m^3$ | Micrograms Per Meter Cubed |
| VC | Vinyl Chloride |
| VOC | Volatile Organic Compound |

**ATTACHMENT 1**

**Figure 1 – Route for Emergency Services**

Last Date Saved: 20 Dec 2013 Document Path: E:\Aerial Locus Maps\4845S-13Mcalpn - RTH.mxd




| | | | |
|---|---|---|---|
| Date: 12-20-2013 | DAY ENVIRONMENTAL, INC.<br>Environmental Consultants<br>Rochester, New York 14606<br>New York, New York 10170 | Project Title: 50 balfour drive<br>ROCHESTER, NEW YORK<br>HEALTH AND SAFETY PLAN | Project No.: 4845S-13 |
| Drawn By: RJM | | Drawing Title: Route to Emergency Services | FIGURE 1 |
| Scale: AS NOTED | | | |