

**QUALITY ASSURANCE PROJECT PLAN
FOR
KISER PLATING SITE
REMOVAL ACTION
INDIANAPOLIS, MARION COUNTY, INDIANA
NPL STATUS: NON-NPL**

Prepared for

UNITED STATES ENVIRONMENTAL PROTECTION AGENCY

Prepared by

WESTON SOLUTIONS, INC.

June 10, 2013 (Revision 1)

U.S. EPA Contract No. EP-S5-06-04
TDD Number: S05-0001-1304-020

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QAPP Worksheet #1
Title and Approval Page

Site Name/Project Name: Kiser Plating Removal Action Site

Site Location: 401 E. Howard Street, Muncie, Delaware County, Indiana

Quality Assurance Project Plan (QAPP) for the Kiser Plating Site Removal Action
Document Title

United States Environmental Protection Agency (U.S. EPA) Region V
Lead Organization

Greg Roussos, Weston Solutions, Inc. (WESTON®) Superfund Technical Assessment and Response Team (START)

Preparer's Name and Organizational Affiliation

711 E Monument Ave, Suite 201, Dayton, Montgomery County, Ohio, (937) 531-4400
Greg.Roussos@WestonSolutions.com

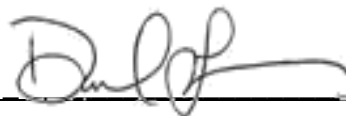
Preparer's Address, Telephone Number, and E-mail Address

June 10, 2013 (Revision 0)

Preparation Date (Day/Month/Year)

Investigative Organization's Project Manager: _____

Signature – Dan Liebau



Dan Liebau, WESTON START, 6/10/2013

Printed Name/Organization/Date Printed

Investigative Organization's Project QA Officer: _____

Signature – Lisa Graczyk

Lisa Graczyk, WESTON START, 6/10/2013

Printed Name/Organization/Date

Lead Organization's Project Manager: _____

Signature – Shelly Lam

Shelly Lam, United States Environmental Protection Agency (U.S. EPA),

Printed Name/Organization/Date

Approval Signatures: _____

Signature

Printed Name/Title/Date

Approval Authority

Other Approval Signatures: _____

Signature

Printed Name/Title/Date

Document Control Number: 2135-2E-BHHG

QAPP Worksheet #2 QAPP Identifying Information

Site Name/Project Name: Kiser Plating Removal Action Site
Site Location: 401 E Howard Street, Muncie, Delaware County, Indiana (Figure 1)
Site Number/Code: B5XK
Operable Unit: Not Applicable (NA)
Contractor Name: Weston Solutions, Inc.
Contractor Number: EP-S5-06-04
Contract Title: Superfund Technical Assessment and Response Team
Work Assignment Number: S05-0001-1304-020

1. Identify guidance used to prepare QAPP:
Uniform Federal Policy for Quality Assurance Project Plans
2. Identify regulatory program:
U.S. EPA Region V, Emergency Response Branch
3. Identify approval entity:
U.S. EPA Region V
4. Indicate whether the QAPP is a generic or a project-specific QAPP (circle one)
5. List dates of scoping sessions that were held:
The scoping meeting was conducted on June 6, 2013. Shelly Lam (U.S. EPA), Richie Byrd and Toben Vieweg (Environmental Restoration, LLC), and Keith Hughes, and Dan Liebau (WESTON START) were present during scoping meeting.

6. List dates and titles of QAPP documents written for previous site work, if applicable:

| Title | Approval Date |
|----------------|---------------|
| Not Applicable | |
| | |
| | |

7. List organizational partners (stakeholders) and connection with lead organization:
1) The Indiana Department of Environmental Management
8. List data users:
U.S. EPA Region V, On-Scene Coordinator (OSC)
9. If any required QAPP elements and required information are not applicable to the project, then circle the omitted QAPP elements and required information on the attached table. Provide an explanation for their exclusion below:

Identify where each required QAPP element is located in the QAPP (provide section, worksheet, table, or figure number) or other project planning documents (provide complete document title, date, section number, page numbers, and location of the information in the document). Circle QAPP elements and required information that are not applicable to the project. Provide an explanation in the QAPP.

| Required QAPP Element(s) and Corresponding QAPP Section(s) | Required Information | Crosswalk to Worksheet No. or Related Documents |
|--|---|---|
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| Required QAPP Element(s) and Corresponding QAPP Section(s) | Required Information | Crosswalk to Worksheet No. or Related Documents |
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| Required QAPP Element(s) and Corresponding QAPP Section(s) | Required Information | Crosswalk to Worksheet No. or Related Documents |
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| Required QAPP Element(s) and Corresponding QAPP Section(s) | Required Information | Crosswalk to Worksheet No. or Related Documents |
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COC – Chain-of-Custody

PQO – Project Quality Objectives

QA – Quality Assurance

QAPP – Quality Assurance Project Plan

QC – Quality Control

SOP – Standard Operating Procedure

QAPP Worksheet #3 Distribution List

| QAPP Recipients | Title | Organization | Telephone Number | E-mail Address | DCN |
|------------------------|---|--------------------------------|-------------------------|-----------------------------------|--------------|
| Shelly Lam | OSC | U.S. EPA | 317-417-0980 | lam.shelly@epamail.epa.gov | 2135-2E-BHHG |
| Dan Liebau | Project Manager | WESTON | 906-523-5569 | daniel.liebau@westonsolutions.com | 2135-2E-BHHG |
| Lisa Graczyk | Project QA Officer | WESTON | 312-424-3339 | lgraczyk@css-dynamac.com | 2135-2E-BHHG |
| Keith Hughes | Site Leader | WESTON | 618-922-9985 | khughes@pe-engrs.com | 2135-2E-BHHG |
| Richie Byrd | ERRS Response Manager and Sample Management Coordinator | Environmental Restoration, LLC | 317-371-7674 | r.byrd@erllc.com | 2135-2E-BHHG |
| TBD | Pace Analytical Services | Pace Analytical Services | 317-875-5894 | TBD | 2135-2E-BHHG |

Notes:

DCN – Document Control Number
ERRS – Emergency and Rapid Response Services
OSC – On-Scene Coordinator
QA – Quality Assurance
QAPP – Quality Assurance Project Plan
TBD – To Be Determined
U.S. EPA – United States Environmental Protection Agency
WESTON – Weston Solutions, Inc.

QAPP Worksheet #4
Project Personnel Sign-Off Sheet

Organization: Weston Solutions, Inc.

| Project Personnel | Title | Telephone Number | Signature | Date QAPP Read |
|--------------------------|---|-------------------------|------------------|-----------------------|
| Shelly Lam | U.S. EPA OSC | 317-417-0980 | | June 2013 |
| Dan Liebau | Project Manager | 906-523-5569 | | June 2013 |
| Lisa Graczyk | Project QA Officer | 312-424-3339 | | June 2013 |
| Richie Byrd | ERRS RM and Sample Management Coordinator | 317-371-7674 | | June 2013 |

Notes:

ERRS – Emergency and Rapid Response Services

OSC – On-Scene Coordinator

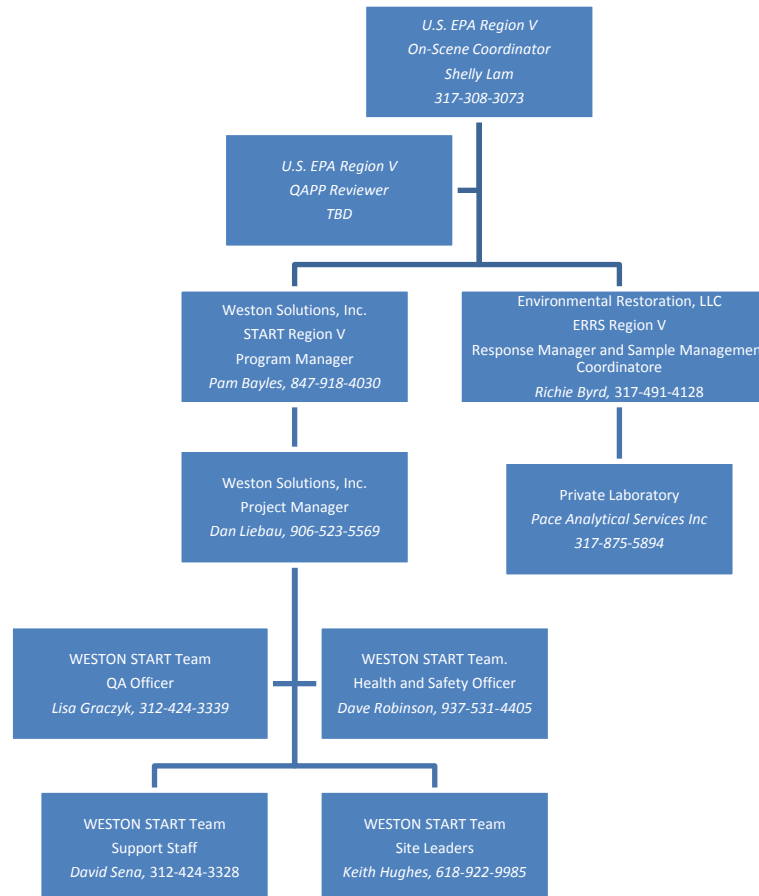
QA – Quality Assurance

QAPP – Quality Assurance Project Plan

RM – Response Manager

U.S. EPA – United States Environmental Protection Agency

QAPP Worksheet #5 Project Organizational Chart



ERRS – Emergency and Rapid Response Services
QAPP – Quality Assurance Project Plan
START – Superfund Technical Assessment and m
U.S. EPA – United States Environmental Protection Agency
WESTON – Weston Solutions, Inc.

**QAPP Worksheet #6
Communication Pathways**

| Communication Drivers | Responsible Entity | Name | Phone Number | Procedure (Timing, Pathways, etc.) |
|--|------------------------------|--------------|---------------------|--|
| Project scope changes | U.S. EPA OSC | Shelly Lam | 317-308-3073 | The OSC will inform the WESTON PM of any project scope changes. The WESTON PM will in turn inform the program manager of the changes. |
| Management of required project tasks for START | WESTON START Project Manager | Dan Liebau | 906-523-5569 | The WESTON PM will inform the appropriate WESTON project staff (field and non-field) of tasks to complete and the required completion date. The WESTON project staff will communicate with the PM of task progress and resources/information |
| Delays or changes to START field work | WESTON START Site Leader | Keith Hughes | 618-922-9985 | The WESTON Site Leader will inform the OSC and of any delays or changes to field work while on site. The WESTON Site Leader will inform the WESTON PM of delays or changes to field work by email or telephone. |
| Daily START field updates | WESTON START Site Leader | Keith Hughes | 618-922-9985 | The WESTON Site Leader will inform the OSC of daily field progress while on site. The WESTON Site Leader will provide the WESTON Project Manager with any pertinent information contained in the daily. |
| Management of required project tasks for ERRS | ERRS Response Manager | Richie Byrd | 317-371-7674 | The ERRS RM will inform the appropriate ERRS project staff (field and non-field) of tasks to complete and the required completion date. The ERRS project staff will communicate with the RM of task progress and resources/information required to complete tasks. |
| Delays or changes to ERRS field work | ERRS Response Manager | Richie Byrd | 317-371-7674 | The ERRS RM will inform the OSC and of any delays or changes to field work while on site. |

| Communication Drivers | Responsible Entity | Name | Phone Number | Procedure (Timing, Pathways, etc.) |
|---|---|--|---|--|
| Daily ERRS field updates | ERRS Response Manager | Richie Byrd | 317-371-7674 | The RM will inform the OSC of daily field progress while on site. |
| Reporting of Laboratory Data Quality Issues | Sample Management Coordinator | Richie Byrd | 317-491-4128 | The Sample Management Coordinator will inform the OSC of any issues related to data quality upon receipt of samples or during analyses. |
| Recommendations to stop work and initiation of corrective actions | OSC/ERRS RM/ WESTON START QA Officer | Shelly Lam / Richie Byrd / Lisa Graczyk | 317-308-3073 / 317-491-4128/ 312-424-3339 | The OSC, ERRS QA Officer and WESTON START QA Officer all have the authority to stop work and initiate corrective actions should there be a reason to do so. Whoever stops the work or initiates corrective actions will inform the other interested parties such as the WESTON Site Leader, ERRS RM and OSC. The Site Leader will inform the WESTON PM of stop work orders and corrective actions. |
| Distribution of analytical data | Sample Management Coordinator | Richie Byrd | 317-491-4128 | The Sample Management Coordinator will receive all deliverables from the laboratory and distribute them to the OSC. The data will not be distributed further until it has been reviewed and validated by an ERRS QA Officer. The OSC will distribute the validated data to other interested parties. |
| Approval of QAPP Amendments | OSC | Shelly Lam | 317-308-3073 | Approval of all QAPP amendments will be by the OSC prior to the changes being implemented. |

Notes:

ERRS – Emergency and Rapid Response Services
OSC – On-Scene Coordinator
PM – Project Manager
QA – Quality Assurance
QAPP – Quality Assurance Project Plan

SMC – Sample Management Coordinator
START – Superfund Technical Assessment and Response Team
U.S. EPA – United States Environmental Protection Agency
WESTON – Weston Solutions, Inc.

QAPP Worksheet #7
Personnel Responsibilities and Qualifications Table

| Name | Title | Organizational Affiliation | Responsibilities | Education and Experience Qualifications |
|---------------|--------------------------|-----------------------------------|--|--|
| Shelly Lam | OSC | U.S. EPA Region V | The OSC or her designee has overall project authority and directs the WESTON Project Manager regarding the tasks required to meet project objectives. The OSC is also responsible for reviewing and approving the project-specific QAPP (and any amendments) prior to its implementation. | Federal OSC |
| Pamela Bayles | Region V Program Manager | WESTON START | The START Program Manager is responsible for ensuring the quality of work performed under the Region V START III contract. The START Program Manager interfaces directly with the U.S. EPA Contracting Officer and Project Officer, and has overall responsibility and direction for task assignments. | M.E.M. (Masters in Environmental Management), Air and Water Resources; B.S., Biology; over 19 years experience |
| Dan Liebau | Project Manager | WESTON START | The project manager is responsible for managing all START aspects of the project, WESTON project personnel, and WESTON subcontractors. The project manager interfaces directly with the U.S. EPA OSC regarding all START project tasks. | TBD |
| Richie Byrd | Response Manager | ER LLC | The response manager is responsible for managing all ERRS aspects of the project, ERRS project personnel, and ERRS subcontractors. The response manager interfaces directly with the U.S. EPA OSC regarding all ERRS project tasks. | TBD |
| Lisa Graczyk | START QA Officer | WESTON START | The START QA Officer reviews the project QAPP and has overall responsibility for START project QA. The QA Officer will also perform a compliance check of all data reviewed and validated by the START Chemist. | B.S. Chemistry; over 21 years experience |

| Name | Title | Organizational Affiliation | Responsibilities | Education and Experience Qualifications |
|---------------|---------------------------|-----------------------------------|---|--|
| Richie Byrd | ERRS QA Officer | ER LLC | The ERRS QA Officer reviews the project QAPP and has overall responsibility for ERRS project QA. The QA Officer will also perform a compliance check of all data reviewed and validated by the ERRS Chemist. | TBD |
| Dave Robinson | Health and Safety Officer | WESTON START | The health and safety officer approves the Health and Safety Plan and provides guidance to WESTON field personnel on health and safety issues. | Over 29 years experience in Industrial Hygiene, Health and Safety, Environmental Science |
| Keith Hughes | Site Leader | WESTON START | The site leader manages the field team and all START work performed in the field. The site leader interfaces directly with the WESTON project manager regarding field tasks and any issues that arise while in the field. | B.S. Environmental Science; over 16 years experience |
| Greg Roussos | QAPP Preparer | WESTON START | The QAPP preparer is responsible for preparing the site-specific QAPP and is in close communication with the Project Manager, Sample Management Coordinator, and QA officer regarding all aspects of project-specific requirements. | B.S. Biology; over 2 years experience |

Notes:

QA – Quality Assurance
QAPP – Quality Assurance Project Plan
QC – Quality Control
ER – Environmental Restoration
ERRS – Emergency and Rapid Response Services
H&S – Health and Safety
HASP – Health and Safety Plan
OSC – On-Scene Coordinator
PM – Project Manager

SMC – Sample Management Coordinator
START – Superfund Technical Assessment and Response Team
TBD – To Be Determined
U.S. EPA – United States Environmental Protection Agency
WESTON – Weston Solutions, Inc.

QAPP Worksheet #8
Special Personnel Training Requirements Table

| Project Function | Specialized Training – Title or Description of Course | Training Provider | Training Date | Personnel/Groups Receiving Training | Personnel Titles/ Organizational Affiliation | Location of Training Records/Certificates¹ |
|---------------------------|---|--------------------------|----------------------|---|---|--|
| Field Sampling Activities | 40-Hour OSHA HAZWOPER Training and Recurrently Annual 8-hour refreshers | WESTON, ER LLC | Various | U.S. EPA OSC, WESTON START Site Leader, WESTON START site personnel, ERRS personnel | U.S. EPA, WESTON START, ER LLC personnel | WESTON's web-based EHS Track for WESTON START personnel; on-site for WESTON START and ERRS personnel |

Notes:

EHS – Environmental Health and Safety

ER – Environmental Restoration

ERRS – Emergency and Rapid Response Services

HAZWOPER – Hazardous Waste Operations and Emergency Response

OSHA – Occupational Safety and Health Administration

QAPP – Quality Assurance Project Plan

U.S. EPA – United States Environmental Protection Agency

WESTON – Weston Solutions, Inc.

QAPP Worksheet #9 Project Scoping Session Participants Sheet

| Project Name: Kiser Plating Removal Action Site Projected Date(s) of Sampling: Beginning week of 6/17/2013 Project Manager: Dan Liebau | | | | Site Name: Kiser Plating Site Location: 401 E. Howard Street, Muncie, Delaware County, Indiana | |
|---|-----------------|----------------------|--------------|--|---------------------|
| Date of Session: April 6, 2012 Scoping Session Purpose: To plan sampling at the Site during the removal | | | | | |
| Name | Title | Affiliation | Phone # | E-mail Address | Project Role |
| Shelly Lam | U.S. EPA OSC | U.S. EPA Region V | 317-417-0980 | lam.shelly@epamail.epa.gov | U.S. EPA OSC |
| Dan Liebau | PM | WESTON START | 937-602-3089 | randy.kirkland@westonsolutions.com | WESTON PM |
| Keith Hughes | Site Leader | WESTON START | 618-922-9985 | khughes@pe-engrs.com | START Field Lead |
| Richie Byrd | RM | ER LLC | 317-371-7674 | r.byrd@erllc.com | ERRS RM |
| | | | | | |

Notes:

ER – Environmental Restoration
OSC – On-Scene Coordinator
PM – Project Manager
QAPP – Quality Assurance Project Plan
RM – Response Manager
START -
U.S. EPA – United States Environmental Protection Agency
WESTON – Weston Solutions, Inc.

Comments/Decisions: During a scoping session meeting, the OSC, ERRS RM, and START discussed:

1) Preliminary soil sampling

- A grid pattern for soil sampling will be used to determine the extent of VOC contamination. Soil samples will be analyzed for Toxicity Characteristic Leachate Procedure (TCLP) VOCs and total target analyte list (TAL) VOCs. Action levels for TCLP VOC analytical results will be the screening criteria in 40 CFR, Part 261, Subpart C. Action levels for total VOC analytical results will be the 2009 Indiana Department of Environmental Management (IDEM) Industrial and Residential Soil Closure Levels for Direct Contact. Soil will be excavated and removed where the representative grid sample exceeds any action level. Soil will be disposed of as hazardous waste if the representative grid sample exceeds the TCLP VOC action level or the IDEM Industrial Soil Closure Level for Direct Contact. Soil to be disposed will also be analyzed for TCLP SVOCs and TCLP metals.

2) Residential vapor sampling

- A public meeting will be held on October 3, 2012, to inform the public of the threat of vapor intrusion and planned removal actions. Up to 170 parcels, residential and commercial, will be offered to be sampled for VOCs in subsurface vapor or soil gas and indoor air. Action levels will

be specified by the Agency for Toxic Substance and Disease Registry (ATSDR). If action levels are exceeded, the installation of vapor mitigation systems and post-installation proficiency sampling may be conducted.

3) Confirmation soil sampling

- Confirmation soil samples will be collected from the sidewalls and floor of excavation areas. Confirmation soil samples will be analyzed for total TAL VOCs. Additional excavation will occur where the confirmation soil sample exceeds IDEM Residential Soil Closure Levels for Direct Contact.

Action Items: ERRS will procure the laboratory and equipment needed for sampling.

Consensus Decisions: See above

QAPP Worksheet #10 Problem Definition

The problem to be addressed by the project: Site assessment analytical results indicated that hazardous substances as defined by CERCLA Section 101(14) and pollutants and contaminants as defined by CERCLA Section 101(33) are present at the Site and represent an actual or potential exposure threat to nearby human populations. Concentrations of hazardous substances exceed relevant screening criteria. Possible exposure routes include dermal contact with contaminated subsurface soil during excavation activities and inhalation of contaminated air that has migrated through subsurface soil and groundwater (known as vapor intrusion). Potential human receptors include future Site workers and nearby residents. Based on site assessment analytical results, hazardous substances identified at the Site at concentrations exceeding the relevant screening criteria include cadmium in subsurface soil and the following substances in soil gas: 1,2,4-trimethylbenzene; 1,1-dichloroethene; trans-1,2-dichloroethene; ethylbenzene; m,p-xylene; tetrachloroethene; trichloroethene; and vinyl chloride. TCLP cadmium was detected in subsurface soil at a concentration exceeding its 40 CFR 261.24(b) regulatory limit. The other contaminants were detected in soil gas at concentrations exceeding their respective VISLs. The Agency for Toxic Substances and Disease Registry (ATSDR) has studied the toxicological effects of all these hazardous substances except for 1,2,4-trimethylbenzene. Information about each substance except 1,2,4-trimethylbenzene is provided below from the ATSDR ToxFAQs.

During the site assessment, a soil sample collected near the surface contained TCLP cadmium at a concentration of 15 mg/L, which exceeds its 40 CFR 261.24(b) regulatory limit of 1 mg/L. Cadmium in soil near the surface could migrate through the soil and into groundwater. Based on analytical results for soil gas samples collected during the site assessment, the following hazardous substances were detected at concentrations exceeding their respective VISLs: 1,2,4-trimethylbenzene; 1,1-dichloroethene; trans-1,2-dichloroethene; ethylbenzene; m,p-xylene; tetrachloroethene; trichloroethene; and vinyl chloride. Some of these hazardous substances were detected at concentrations greatly exceeding their VISLs. For example, the VISL for trichloroethene is 3.9 ppbv. Trichloroethene was detected in soil gas sample at a concentration of 82,000 ppbv, which is more than 20,000 times the VISL. Trichloroethene is a hazardous substance with the potential to cause cancer in humans. Volatilization of hazardous substances in Site soil may pose a threat to nearby residents. Contamination in subsurface soil could migrate off the Site to residential properties through the vapor intrusion pathway, causing potential exposure of nearby human populations to hazardous substances, pollutants, or contaminants. Residential properties are located directly west of the Site.

The environmental questions being asked: What is the extent of VOC contamination in on-site soil? Have soil cleanup objectives been achieved? What is the extent of VOC contamination in subslab vapor beneath off-site residential and commercial buildings? What is the extent of VOC contamination in indoor air in off-site residential and commercial buildings? Are vapor mitigation systems required in homes or businesses?

The possible classes of contaminants and the affected matrices: VOCs in soil, soil-gas, subslab vapor, and indoor air.

The rationale for inclusion of chemical and non-chemical analyses: Historical sampling and analytical laboratory analysis of on-site soil, on-site groundwater, and on-site soil gas (see above).

Project decision conditions (“If..., then...” statements): If soil action levels exceeding the IDEM 2009 Residential Closure Levels for Direct Contact are exceeded within the excavation, additional excavation may be conducted. If soil gas, subslab vapor, or indoor air action levels specified by the ATSDR are exceeded, the installation of a vapor mitigation system, and post-installation proficiency sampling may be conducted.

Notes:

ATSDR – Agency for Toxic Substance and Disease Registry

µg/m³ – Microgram per cubic meter

µg/L – Micrograms per liter

CERCLA – Comprehensive Environmental Response, Compensation, and Liability Act

DCE - Dichloroethene

ERRS – Emergency and Rapid Response Services

IDEM – Indiana Department of Environmental Management

OSWER - Office of Solid Waste and Emergency Response

ppbv - parts per billion by volume

QAPP – Quality Assurance Project Plan

RAL - Removal Actions Level

RCRA - Resource Conservation Recovery Act

RSL – Regional Screening Level

TCLP - Toxicity Characteristic Leachate Procedure

START – Superfund Technical Assessment and Response Team

U.S. EPA – United States Environmental Protection Agency

UST – Underground storage tank

VOC – Volatile organic compound

VISL – Vapor Intrusion Screening Level

WESTON – Weston Solutions, Inc.

QAPP Worksheet #11
Project Quality Objectives/Systematic Planning Process Statements

Who will use the data? U.S. EPA Region V

What will the data be used for? Soil analytical data will be used to determine the extent of contamination in soil and to determine if soil in the excavation exceeds action levels, requiring additional excavation. Soil gas, subslab vapor, and indoor air analytical data will be used to determine if vapor at residential properties nearby the Site contain VOCs above site-specific action levels. This will determine whether vapor mitigation systems will need to be installed.

What type(s) of data are needed? (target analytes, analytical groups, field screening, on-site analytical or off-site laboratory techniques, sampling techniques): Preliminary and confirmation soil samples collected during the removal action will be submitted to an offsite laboratory for total TAL VOC analysis using U.S. EPA SW-846 Method 8260B. Preliminary soil samples will also be submitted for TCLP TAL VOC analysis using U.S. EPA SW-846 Method 1311/8260B. During preliminary soil sampling, soil borings will be field-screened using a MultiRAE photoionization detector (PID) to identify areas of highest VOC contamination. Equipment rinsate samples will be collected from drilling equipment during preliminary soil sampling activities and submitted for total and TCLP VOC analysis.

Subslab vapor samples collected during the removal action will be submitted to an offsite laboratory for the total VOC analysis using U.S. EPA Method TO-15 Gas Chromatography/Mass Spectrometry (GC/MS).

How “good” does the data need to be in order to support the environmental decision? The reporting limits need to be sufficient to compare VOC results to specific screening criteria.

How much data are needed? (number of samples for each analytical group, matrix, and concentration): The quantity of preliminary soil data will need to be sufficient to determine the extent of contamination within the site boundary. An estimated 44 extent of contamination soil samples will be collected.

The quantity of confirmation soil data will be sufficient to be representative of the soil remaining within the excavation. An estimated 80 confirmation soil samples will be collected.

The quantity of air sample data will need to be sufficient to be representative of the sub-slab vapor, indoor air, and ambient air in residential and commercial properties in close proximity to the Site. An estimate of up to 100 sub-slab vapor, indoor air, and ambient air samples will be collected.

The data will be collected in accordance with Worksheet # 17.

Who will collect and generate the data? WESTON START will collect the data. START will determine the location of the subslab sample within basements of the residential properties. The analytical data will be generated by the Pace Analytical.

How will the data be reported? The sample results will be reported by the ERRS-procured commercial laboratory in a summary report submitted to the ERRS Sample Management Coordinator by e-mail. The ERRS Sample Management Coordinator will distribute the summary report to the U.S. EPA OSC, who will in turn distribute the summary report to WESTON and any other interested parties. The ERRS QA Officer will perform a compliance check of all data received from the laboratory.

How will the data be archived? WESTON will maintain a copy of all Site-related data and files for a period of 10 years in accordance with its policies. In addition, WESTON will give a copy of all data to the U.S. EPA OSC, who will archive the data in the U.S. EPA's records center.

Notes:

ATSDR – Agency for Toxic Substance and Disease Registry
ERRS – Emergency and Rapid Response Services
GC/MS – Gas Chromatography/Mass Spectrometry
IDEM – Indiana Department of Environmental Management
OSC – On-Scene Coordinator
PPE – Personal Protective Equipment
QA – Quality Assurance

QAPP – Quality Assurance Project Plan
QC – Quality Control
RISC - Risk Integrated System of Closure
START – Superfund Technical Assessment and Response Team
U.S. EPA – United States Environmental Protection Agency
VOC – Volatile organic compound
WESTON – Weston Solutions, Inc.

QAPP Worksheet #12A
Measurement Performance Criteria Table

| Matrix | Soil | | | | |
|-------------------------------------|--|--------------------------------|--|--|--|
| Analytical Group¹ | Total VOCs | | | | |
| Concentration Level | High/Medium/Low | | | | |
| Sampling Procedure | Analytical Method/SOP² | Data Quality Indicators | Measurement Performance Criteria | QC Sample and/or Activity Used to Assess Measurement Performance | QC Sample Assesses Error for Sampling (S), Analytical (A) or both (S&A) |
| See Worksheet #17 | U.S. EPA SW-846 Method 8260B | Laboratory Precision | RPD \leq 20% | Laboratory Duplicates | A |
| | | Overall Accuracy/Bias | % Recovery as determined by laboratory | Laboratory Control Samples | A |
| | | Accuracy/Bias Contamination | Any detection of target analytes in the blank | Laboratory Blanks | A |
| | | Representativeness | NA | Adherence to surface soil sampling SOP (WSOP-302) for collection procedures and quantity of samples to collect | S |
| | | Sensitivity | Quantitation limit at MDLs for individual metals compounds | Method Detection Limit Study | A |
| | | Completeness | 90% of samples collected and analytical data received | Project manager assesses completeness of samples collected; laboratory project manager assess completeness of analytical requirements per the QAPP | S&A |

Notes:

¹If information varies within an analytical group, separate by individual analyte.

²Reference number from QAPP Worksheet #23 (see Section 3.2).

% - Percent

\leq - Less than or equal to

MDL - Method Detection Limit

NA - Not Applicable

QAPP - Quality Assurance Project Plan

QC - Quality Control

RPD - Relative Percent Difference

SOP - Standard Operating Procedure

VOC - Volatile organic compound

QAPP Worksheet #12B
Measurement Performance Criteria Table

| Matrix | Vapor | | | | |
|-------------------------------------|--|--------------------------------|--|--|--|
| Analytical Group¹ | Total VOCs | | | | |
| Concentration Level | High/Medium/Low | | | | |
| Sampling Procedure | Analytical Method/SOP² | Data Quality Indicators | Measurement Performance Criteria | QC Sample and/or Activity Used to Assess Measurement Performance | QC Sample Assesses Error for Sampling (S), Analytical (A) or both (S&A) |
| See Worksheet #17 | U.S. EPA Method TO-15 | Laboratory Precision | RPD \leq 20% | Laboratory Duplicates | A |
| | | Overall Accuracy/Bias | % Recovery as determined by laboratory | Laboratory Control Samples | A |
| | | Accuracy/Bias Contamination | Any detection of target analytes in the blank | Laboratory Blanks | A |
| | | Representativeness | NA | Adherence to subslab vapor sampling SOP, U.S. EPA SOP 2082 – Construction and Installation of Permanent Sub-Slab Soil Gas Wells. | S |
| | | Sensitivity | Quantitation limit at MDLs for individual metals compounds | Method Detection Limit Study | A |
| | | Completeness | 90% of samples collected and analytical data received | Project manager assesses completeness of samples collected; laboratory project manager assess completeness of analytical requirements per the QAPP | S&A |

Notes:

¹If information varies within an analytical group, separate by individual analyte.

²Reference number from QAPP Worksheet #23 (see Section 3.2).

% - Percent

\leq – Less than or equal to

MDL – Method Detection Limit

NA – Not Applicable

QAPP – Quality Assurance Project Plan

QC – Quality Control

RPD – Relative Percent Difference

SOP – Standard Operating Procedure

VOC – Volatile organic compound

QAPP Worksheet #12C Measurement Performance Criteria Table

| Matrix | Soil | | | | |
|-------------------------------------|--|--------------------------------|--|--|--|
| Analytical Group¹ | TCLP VOCs | | | | |
| Concentration Level | High/Medium/Low | | | | |
| Sampling Procedure | Analytical Method/SOP² | Data Quality Indicators | Measurement Performance Criteria | QC Sample and/or Activity Used to Assess Measurement Performance | QC Sample Assesses Error for Sampling (S), Analytical (A) or both (S&A) |
| See Worksheet #17 | U.S. EPA SW-846 Method 1311/8260B | Laboratory Precision | RPD \leq 20% | Laboratory Duplicates | A |
| | | Overall Accuracy/Bias | % Recovery as determined by laboratory | Laboratory Control Samples | A |
| | | Accuracy/Bias Contamination | Any detection of target analytes in the blank | Laboratory Blanks | A |
| | | Representativeness | NA | Adherence to surface soil sampling SOP (WSOP-302) for collection procedures and quantity of samples to collect | S |
| | | Sensitivity | Quantitation limit at MDLs for individual metals compounds | Method Detection Limit Study | A |
| | | Completeness | 90% of samples collected and analytical data received | Project manager assesses completeness of samples collected; laboratory project manager assess completeness of analytical requirements per the QAPP | S&A |

Notes:

¹If information varies within an analytical group, separate by individual analyte.

²Reference number from QAPP Worksheet #23 (see Section 3.2).

% - Percent

\leq - Less than or equal to

MDL – Method Detection Limit

NA – Not Applicable

QAPP – Quality Assurance Project Plan

QC – Quality Control

RPD – Relative Percent Difference

SOP – Standard Operating Procedure

TCLP – Toxicity Characteristic Leaching Procedure

VOC – Volatile organic compound

QAPP Worksheet #13
Secondary Data Criteria and Limitations Table

| Secondary Data | Data Source (Originating Organization, Report Title, and Date) | Data Generator(s) (Originating Org., Data Types, Data Generation/Collection Dates) | How Data Will Be Used | Limitations on Data Use |
|----------------|--|---|--------------------------|----------------------------|
| Not Applicable | | | | |

Notes:

QAPP – Quality Assurance Project Plan

QAPP Worksheet #14

Summary of Project Tasks

Sampling Tasks:

1. Sampling of soil to determine extent of excavation
2. Equipment rinsate sampling
3. Sampling of subslab vapor or soil gas
4. Sampling of indoor air
5. Sampling of soil in the finished excavation

Analysis Tasks:

An ERRS-procured commercial laboratory will prepare and process the samples for the following:

1. Total TAL VOCs
2. TCLP VOCs, TCLP SVOCs & TCLP Metals for disposal analysis
3. TO 15 (VOCs)
4. Total Metals

Quality Control Tasks:

1. Perform sample collection procedures per SOPs.
2. Collect duplicate, equipment blanks, and matrix spike / matrix spike duplicate samples as necessary.
3. Laboratory to perform laboratory QC procedures. QC procedures include analyzing blanks, laboratory control sample, and matrix spike samples.
4. The WESTON QA Officer will perform a compliance check of all data received from the laboratory.

Secondary Data:

Not applicable.

Data Management Tasks:

Compare sub-slab and indoor air vapor results to site-specific screening criteria provided by the ATSDR. Compare soil results to IDEM 2009 Residential Closure Levels for Direct Contact for soil at industrial properties.

Documentation and Records:

Sampling locations will be documented and all sample collection data will be recorded in field logbooks. COCs, air bills, and sample

logs will be prepared and retained for each sample. A copy of all finalized documents and analytical data will be retained in a central file area.

Assessment/Audit Tasks:

Assessment of field activities will be carried out by the Project Manager through frequent contact with the site leader. Audits will be carried out as directed and approved by the U.S. EPA OSC.

Data Review Tasks:

The laboratory will review all analytical data for completeness and quality. The analytical data will then be submitted to the U.S. EPA's ERRS contractor for distribution to the U.S. EPA OSC. The U.S. EPA OSC will in turn distribute the analytical data to WESTON and any other parties. A case narrative describing any quality control issues with the analyses will be submitted with the final data report. In addition, the laboratory will qualify data in accordance with its quality policies. The ERR RM (or his designee) will also perform a compliance check of all data received from the laboratory.

Notes:

ASTDR – Agency for Toxic Substance and Disease Registry

COC – Chain of Custody

ERRS – Emergency and Rapid Response Services

OSC – On-Scene Coordinator

QA – Quality Assurance

QAPP – Quality Assurance Project Plan

QC – Quality Control

RM – Response Manager

RSL – Regional Screening Level

SOP – Standard Operating Procedure

U.S. EPA – United States Environmental Protection Agency

VOC – Volatile organic compound

WESTON – Weston Solutions, Inc.

QAPP Worksheet #15 Reference Limits and Evaluation Table

| Matrix | Sub-slab Vapor | | | | | | |
|-------------------------------------|---------------------|--|--|--------------------------------------|-----------------------------|---|-----------------------|
| Analytical Group¹ | Total VOCs | | | | | | |
| Concentration Level | High/Medium/ Low | | | | | | |
| Analyte | CAS Number | Project Action Limit (ppbv) | Project Quantitation Limit (ppbv) | Analytical Method¹ | | Achievable Laboratory Limits² | |
| | | | | MDLs (ppb) | Method QLs (ppb) | MDLs (ppbv) | QLs (ppbv) |
| Acetone | 67-64-1 | 134,450 | 134,450 | TBD | TBD | 0.065 | 2.0 |
| Chloroform | 67-66-3 | 0.9 | 0.9 | TBD | TBD | 0.08 | 0.5 |
| 1,2-Dichlorobenzene | 95-50-1 | 350 | 350 | TBD | TBD | 0.2 | 0.5 |
| cis-1,2-Dichloroethene | 156-59-2 | - | - | TBD | TBD | 0.060 | 0.5 |
| trans-1,2-Dichloroethene | 156-60-5 | - | - | TBD | TBD | 0.076 | 0.5 |
| 2-Hexanone | 591-78-6 | - | - | TBD | TBD | 0.048 | 2.0 |
| p-Isopropyltoluene | 99-87-6 | - | - | TBD | TBD | - | - |
| Propylbenzene | 103-65-1 | - | - | TBD | TBD | - | - |
| Tetrachloroethylene | 127-18-4 | 3 | 3 | TBD | TBD | 0.086 | 0.5 |
| Trichloroethylene | 79-01-6 | 9 | 9 | TBD | TBD | 0.048 | 0.5 |
| 1,2,4-Trimethylbenzene | 95-63-6 | 15 | 15 | TBD | TBD | 0.19 | 0.5 |
| Vinyl chloride | 75-01-4 | - | - | TBD | TBD | 0.36 | 0.5 |
| m,p-Xylene | - | 500 | 500 | TBD | TBD | 0.12 | 1.0 |
| o-Xylene | 95-47-6 | 500 | 500 | TBD | TBD | 0.086 | 0.50 |

Notes:

¹Analytical MDLs and QLs are those documented in validated methods.

²Achievable MDLs and QLs are limits that an individual laboratory can achieve when performing a specific analytical method.

CAS – Chemical Abstract Service

MDL – Method Detection Limit

ppbv – Parts per billion by volume

QAPP – Quality Assurance Project Plan

QL – Quantitation Limit

VOC – Volatile organic compound

QAPP Worksheet #15 - Continued Reference Limits and Evaluation Table

| Matrix | Indoor Air / Ambient Air | | | | | | |
|-------------------------------------|-----------------------------|--|--|--------------------------------------|----------------------------------|---|-----------------------|
| Analytical Group¹ | Total VOCs | | | | | | |
| Concentration Level | High/Medium/ Low | | | | | | |
| Analyte | CAS Number | Project Action Limit (ppbv) | Project Quantitation Limit (ppbv) | Analytical Method¹ | | Achievable Laboratory Limits² | |
| | | | | MDLs (ppbv) | Method QLs (ppbv) | MDLs (ppbv) | QLs (ppbv) |
| Acetone | 67-64-1 | 13,445 | 13,445 | TBD | TBD | 0.065 | 2.0 |
| Chloroform | 67-66-3 | 0.09 | 0.09 | TBD | TBD | 0.08 | 0.5 |
| 1,2-Dichlorobenzene | 95-50-1 | 35 | 35 | TBD | TBD | 0.2 | 0.5 |
| cis-1,2-Dichloroethene | 156-59-2 | - | - | TBD | TBD | 0.060 | 0.5 |
| trans-1,2-Dichloroethene | 156-60-5 | - | - | TBD | TBD | 0.076 | 0.5 |
| 2-Hexanone | 591-78-6 | - | - | TBD | TBD | 0.048 | 2.0 |
| p-Isopropyltoluene | 99-87-6 | - | - | TBD | TBD | - | - |
| Propylbenzene | 103-65-1 | - | - | TBD | TBD | - | - |
| Tetrachloroethylene | 127-18-4 | 0.3 | 0.3 | TBD | TBD | 0.086 | 0.5 |
| Trichloroethylene | 79-01-6 | 0.9 | 0.9 | TBD | TBD | 0.048 | 0.5 |
| 1,2,4-Trimethylbenzene | 95-63-6 | 1.5 | 1.5 | TBD | TBD | 0.19 | 0.5 |
| Vinyl chloride | 75-01-4 | - | - | TBD | TBD | 0.36 | 0.5 |
| m,p-Xylene | - | 50 | 50 | TBD | TBD | 0.12 | 1.0 |
| o-Xylene | 95-47-6 | 50 | 50 | TBD | TBD | 0.086 | 0.50 |

Notes:

¹Analytical MDLs and QLs are those documented in validated methods.

²Achievable MDLs and QLs are limits that an individual laboratory can achieve when performing a specific analytical method.

CAS – Chemical Abstract Service

MDL – Method Detection Limit

ppbv – Parts per billion by volume

QAPP – Quality Assurance Project Plan

QL – Quantitation Limit

VOC – Volatile organic compound

QAPP Worksheet #15 - Continued Reference Limits and Evaluation Table

| Matrix | Soil Gas / Vapor |
|-------------------------------|---------------------|
| Analytical Group ¹ | Total VOCs |
| Concentration Level | High/Medium/ Low |

| Analyte | CAS Number | Project Action Limit (ppbv) | Project Quantitation Limit (ppbv) | Analytical Method ¹ | | Achievable Laboratory Limits ² | |
|--------------------------|------------|--------------------------------|---|--------------------------------|-------------------------|--|---------------|
| | | | | MDLs (ppbv) | Method QLs (ppbv) | MDLs (ppbv) | QLs (ppbv) |
| Acetone | 67-64-1 | 134,450 | 134,450 | TBD | TBD | 0.065 | 2.0 |
| Chloroform | 67-66-3 | 0.9 | 0.9 | TBD | TBD | 0.08 | 0.5 |
| 1,2-Dichlorobenzene | 95-50-1 | 350 | 350 | TBD | TBD | 0.2 | 0.5 |
| cis-1,2-Dichloroethene | 156-59-2 | - | - | TBD | TBD | 0.060 | 0.5 |
| trans-1,2-Dichloroethene | 156-60-5 | - | - | TBD | TBD | 0.076 | 0.5 |
| 2-Hexanone | 591-78-6 | - | - | TBD | TBD | 0.048 | 2.0 |
| p-Isopropyltoluene | 99-87-6 | - | - | TBD | TBD | - | - |
| Propylbenzene | 103-65-1 | - | - | TBD | TBD | - | - |
| Tetrachloroethylene | 127-18-4 | 3 | 3 | TBD | TBD | 0.086 | 0.5 |
| Trichloroethylene | 79-01-6 | 9 | 9 | TBD | TBD | 0.048 | 0.5 |
| 1,2,4-Trimethylbenzene | 95-63-6 | 15 | 15 | TBD | TBD | 0.19 | 0.5 |
| Vinyl chloride | 75-01-4 | - | - | TBD | TBD | 0.36 | 0.5 |
| m,p-Xylene | - | 500 | 500 | TBD | TBD | 0.12 | 1.0 |
| o-Xylene | 95-47-6 | 500 | 500 | TBD | TBD | 0.086 | 0.50 |

Notes:

¹Analytical MDLs and QLs are those documented in validated methods.

²Achievable MDLs and QLs are limits that an individual laboratory can achieve when performing a specific analytical method.

CAS – Chemical Abstract Service

MDL – Method Detection Limit

NA – Not Applicable

ppbv – Parts per billion by volume

QAPP – Quality Assurance Project Plan

QL – Quantitation Limit

VOC – Volatile organic compounds

QAPP Worksheet #15 - Continued Reference Limits and Evaluation Table

| | |
|-------------------------------------|---------------------|
| Matrix | Soil |
| Analytical Group¹ | Total VOCs |
| Concentration Level | High/Medium/ Low |

| Analyte | CAS Number | Project Action Limits ³ (mg/kg) | Project Quantitation Limit (mg/kg) | Analytical Method ¹ | | Achievable Laboratory Limits ² | |
|--------------------------|------------|---|------------------------------------|--------------------------------|--------------------------|---|----------------|
| | | | | MDLs (mg/kg) | Method QLs (mg/kg) | MDLs (mg/kg) | QLs (mg/kg) |
| Acetone | 67-64-1 | 35,000 / 51,000 | 35,000 / 51,000 | TBD | TBD | 9.6 | 50 |
| Chloroform | 67-66-3 | 3 / 4.7 | 3 / 4.7 | TBD | TBD | 0.6 | 5.0 |
| 1,2-Dichlorobenzene | 95-50-1 | 2,800 / 3,900 | 2,800 / 3,900 | TBD | TBD | 1.1 | 10 |
| cis-1,2-Dichloroethene | 156-59-2 | 110 / 140 | 110 / 140 | TBD | TBD | 0.8 | 5.0 |
| trans-1,2-Dichloroethene | 156-60-5 | 180 / 230 | 180 / 230 | TBD | TBD | 1.0 | 5.0 |
| 2-Hexanone | 591-78-6 | - | - | TBD | TBD | 2.4 | 10 |
| p-Isopropyltoluene | 99-87-6 | - | - | TBD | TBD | 0.9 | 5.0 |
| Propylbenzene | 103-65-1 | 1,600 / 2,200 | 1,600 / 2,200 | TBD | TBD | 1.0 | 10 |
| Tetrachloroethylene | 127-18-4 | 9.9 / 16 | 9.9 / 16 | TBD | TBD | 1.6 | 5.0 |
| Trichloroethylene | 79-01-6 | 4.9 / 24 | 4.9 / 24 | TBD | TBD | 0.9 | 5.0 |
| 1,2,4-Trimethylbenzene | 95-63-6 | 1,800 / 170 | 1,800 / 170 | TBD | TBD | 1.1 | 5.0 |
| Vinyl chloride | 75-01-4 | 1.5 / 6.4 | 1.5 / 6.4 | TBD | TBD | 1.7 | 10 |
| m,p-Xylene | - | 690 / 890 | 690 / 890 | TBD | TBD | 1.6 | 5.0 |
| o-Xylene | 95-47-6 | - | - | TBD | TBD | 0.9 | 5.0 |

Notes:

¹Analytical MDLs and QLs are those documented in validated methods.

²Achievable MDLs and QLs are limits that an individual laboratory can achieve when performing a specific analytical method.

³Project Action Limits include both the 2009 IDEM Residential and Industrial Soil Closure Level for Direct Contact.

CAS – Chemical Abstract Service

MDL – Method Detection Limit

mg/kg – milligram per kilogram

NA – Not Applicable

QAPP – Quality Assurance Project Plan

QL – Quantitation Limit

VOC – Volatile organic compound

QAPP Worksheet #15 - Continued Reference Limits and Evaluation Table

| Matrix | Soil | | | | | | |
|-------------------------------------|---------------------|--|---|--------------------------------------|----------------------------------|---|-----------------------|
| Analytical Group¹ | TCLP VOCs | | | | | | |
| Concentration Level | High/Medium/ Low | | | | | | |
| Analyte | CAS Number | Project Action Limit (mg/L) | Project Quantitation Limit | Analytical Method¹ | | Achievable Laboratory Limits² | |
| | | | | MDLs (mg/L) | Method QLs (mg/L) | MDLs (mg/L) | QLs (mg/L) |
| Benzene | 71-43-2 | 0.5 | 0.5 | TBD | TBD | 0.0008 | 0.005 |
| Carbon tetrachloride | 56-23-5 | 0.5 | 0.5 | TBD | TBD | 0.0017 | 0.005 |
| Chlorobenzene | 108-90-7 | 100 | 100 | TBD | TBD | 0.0008 | 0.005 |
| Chloroform | 67-66-3 | 6.0 | 6.0 | TBD | TBD | 0.0009 | 0.005 |
| 1,4-Dichlorobenzene | 106-46-7 | 7.5 | 7.5 | TBD | TBD | 0.0007 | 0.01 |
| 1,2-Dichloroethane | 107-06-2 | 0.5 | 0.5 | TBD | TBD | 0.0012 | 0.005 |
| 1,1-Dichloroethene | 75-35-4 | 0.7 | 0.7 | TBD | TBD | 0.0008 | 0.005 |
| Methyl ethyl ketone | 78-93-3 | 200 | 200 | TBD | TBD | 0.0036 | 0.01 |
| Tetrachloroethylene | 127-18-4 | 0.7 | 0.7 | TBD | TBD | 0.0013 | 0.005 |
| Trichloroethylene | 79-01-6 | 0.5 | 0.5 | TBD | TBD | 0.0009 | 0.005 |
| Vinyl chloride | 75-01-4 | 0.2 | 0.2 | TBD | TBD | 0.0009 | 0.002 |

Notes:

¹Analytical MDLs and QLs are those documented in validated methods.

²Achievable MDLs and QLs are limits that an individual laboratory can achieve when performing a specific analytical method.

CAS – Chemical Abstract Service
MDL – Method Detection Limit
mg/L – Milligrams per liter
QAPP – Quality Assurance Project Plan

QL – Quantitation Limit
TCLP – Toxicity Characteristic Leachate Procedure
VOC – Volatile organic compound

**QAPP Worksheet #16
Project Schedule/Timeline Table**

| Activities | Organization | Dates (Month Day, Year) | | Deliverable | Deliverable Due Date |
|--|-----------------|-----------------------------------|--------------------------------|------------------------|----------------------|
| | | Anticipated Date(s) of Initiation | Anticipated Date of Completion | | |
| QAPP Preparation | WESTON | June 10, 2013 | July 31, 2013 | QAPP | TBD (final) |
| Preliminary Soil Sampling | WESTON | June 17, 2013 | June 11, 2013 | Samples to laboratory | TBD |
| Sub-slab and Soil Gas / Vapor, Indoor Air, and Ambient Air Sampling for VOCs | WESTON | June 18, 2013 | TBD | Samples to laboratory | TBD |
| Confirmation Soil Sampling | WESTON | TBD | TBD | Samples to laboratory | TBD |
| Laboratory Analysis | Pace Analytical | June 17, 2013 | TBD | Laboratory Data Report | TBD |
| Final Project Report | WESTON | TBD | TBD | Final Report | TBD |

Notes:

QAPP – Quality Assurance Project Plan

TBD – To Be Determined

WESTON – Weston Solutions, Inc.

QAPP Worksheet #17 Sampling Design and Rationale

Describe and provide a rationale for choosing the sampling approach (e.g., grid system, biased statistical approach):

Preliminary soil sampling is based on a grid system. The project site will be gridded and marked into 25 foot by 25 foot grids. There will be a total of 51 grids within the excavation site boundary (See Figure 2). The center of each grid will be sampled at a depth of 2 to 4 feet below ground surface using a Geoprobe. Soil samples will be collected using poly scoop and 4 ounce jars. The analytical results will be used to determine which grids will require soil excavation.

U.S. EPA will possibly sample subslab or indoor vapor at up to 100 residential and commercial properties within ½ mile of the Site (see Figure 3). The residential properties sampled will be the closest in proximity to the Site and based upon access granted by the property owners. At locations where it is not feasible to collect subslab vapor samples, such as in residences without basements, an indoor air sample will be collected within the home. In addition, a soil gas/vapor sample will be taken adjacent to the foundation of the property. Where sub-slab and soil gas/vapor samples will be collected, an indoor air sample will be collected in conjunction in order to determine whether potential contaminants in air at the residence originate from subslab vapor or from another source within the residence. Ambient air samples will be collected periodically near sub-slab, soil gas/vapor, and/or indoor air sampling locations. The ambient air samples will be secured within the area of sampling. The analytical results will be used to determine background conditions during the sampling events.

U.S. EPA will sample soil from the floor of the excavation areas and the sidewalls, if present. This method will be used to adequately represent any hot spots that exist in the excavation that may require additional excavation. However, U.S. EPA may determine to leave contaminated soil on site should confirmation sampling results indicate further soil contamination. The decision to discontinue excavation activities will be at the sole discretion of the U.S. EPA OSC.

Describe the sampling design and rationale in terms of what matrices will be sampled, what analytical groups will be analyzed and at what concentration levels, the sampling locations (including QC, critical, and background samples), the number of samples to be taken, and the sampling frequency (including seasonal considerations) [Refer to Worksheet #18 for details]:

Preliminary soil samples will be collected at a depth of 2 to 4 feet below ground surface within 51 25 by 25 foot grids. The soil samples will be screened with X-Ray Fluorescence prior to and then analyzed for total metals and total VOCs for because the site has documented hydrocarbon solvent and metals contamination. Low, medium, and high concentrations are anticipated.

The sub-slab vapor probe will be placed at a feasible location on the lowest level and near the center of the property of nearest to the groundwater plume. The subslab vapor probe will be installed in accordance with U.S. EPA SOP 2082. The subslab vapor sample will be collected over a 24-hour period using a 6-liter summa canister. The locations sampled will be marked clearly on a Site drawing and noted in the field logbook/field collection sheets. This sampling method was chosen in order to best characterize vapor volatilizing from a groundwater plume

impacted by the Site. The sub-slab vapor samples will be analyzed for total VOCs. Low, medium, and high concentrations are anticipated.

For properties without a basement area, a soil gas/vapor sample will be collected adjacent to the property foundation. The sample will be collected for the portion of the property nearest the groundwater plume. This sampling method was chosen in order to best characterize vapor volatilizing from a groundwater plume impacted by the Site for locations without a basement level. The soil gas/vapor samples will be analyzed for total VOCs. Low, medium, and high concentrations are anticipated.

Where sub-slab or soil gas/vapor samples will be collected, an indoor air sample will be collected in conjunction in order to determine whether potential contaminants in air at the residence originate from subslab vapor or from another source within the residence. The indoor air samples will be analyzed for total VOCs. Low concentrations are anticipated.

Ambient air samples will be collected periodically near sub-slab, soil gas/vapor, and/or indoor air sampling locations. The ambient air samples will be secured within the area of sampling. The analytical results will be used to determine background conditions during the sampling events. The ambient air samples will be analyzed for total VOCs. Low concentrations are anticipated.

Soil samples collected in the sidewalls and floor of the excavation areas will be analyzed for total TAL VOCs because the site has documented hydrocarbon solvent contamination. Low and potentially medium concentrations are anticipated.

Notes:

bgs – below ground surface

ERRS – Emergency and Rapid Response Services

QA – Quality Assurance

QAPP – Quality Assurance Project Plan

QC – Quality Control

SOP – Standard operating procedure

START – Superfund Technical Assessment and
Response Team

U.S. EPA – United States Environmental Protection
Agency

WESTON – Weston Solutions, Inc.

QAPP Worksheet #18
Sampling Locations and Methods/SOP Requirements Table

| Sampling Location/ ID Number | Matrix | Depth | Analytical Group | Concentration Level | Number of Samples (identify field duplicates) | Sampling SOP Reference¹ | Rationale for Sampling Location |
|---|------------------|---|---------------------------------|--------------------------------|--|---|--|
| On-site soil (preliminary soil boring samples) / KP-SB01(X- X)-GGG-MMDDYY through KP-SBXX(X- X)-GGG-MMDDYY | Soil | Subsurface up to 4 feet below grade surface | Total TAL VOCs, TCLP VOCs | High/Medium/ Low | Up to 105 samples ^{2,3} | See Worksheet #17 | See Worksheet #17 |
| Residential / commercial properties / KP-SG-123Main- MMDDYY through KP-SG-999Clay- MMDDYY | Soil gas | Subsurface surface | Total VOCs | Total VOCs | Up to 200 samples ⁴ | See Worksheet #17 | See Worksheet #17 |
| Residential properties / KP-SS-123Main- MMDDYY through KP-SS-999Clay- MMDDYY | Subslab Vapor | Immediately below basement slab | Total VOCs | High/Medium/ Low | Up to 60 samples ⁴ | See Worksheet #17 | See Worksheet #17 |
| Residential properties / KP-IA-123Main- MMDDYY through KP-IA-999Clay- MMDDYY | Indoor Air | Breathing level in central location of home, preferably in basement | Total VOCs | High/Medium/ Low | Up to 263 samples ⁴ | See Worksheet #17 | See Worksheet #17 |

| Sampling Location/ ID Number | Matrix | Depth | Analytical Group | Concentration Level | Number of Samples (identify field duplicates) | Sampling SOP Reference ¹ | Rationale for Sampling Location |
|---|-------------|---|---------------------|------------------------|--|---|---------------------------------------|
| Residential properties / KP-AA01- MMDDYY through TC-AAXX- MMDDYY | Ambient Air | Ambient air at or above breathing level, dependent upon sample security needs | Total VOCs | Low | Up to 60 samples ⁴ | See Worksheet #17 | See Worksheet #17 |
| Sidewalls and bottom of excavations / KP- CS-GGG-L- MMDDYY through KP-CS-GGG-L- MMDDYY | Soil | Soil surface | Total VOCs | High/Medium/ Low | Up to 154 samples ² | See Worksheet #17 | See Worksheet #17 |

Notes:

¹Specify the appropriate letter or number from the Project Sampling SOP References table (Worksheet #21).

²MS/MSD and field duplicate samples will be collected at a frequency of 1 for every 20 samples.

³Equipment rinsate samples will be collected at a frequency of 1 for every 20 preliminary soil samples.

⁴Field duplicate samples will be collected at a frequency of 1 for every 20 samples.

ID – Identification

QAPP – Quality Assurance Project Plan

SOP– Standard Operating Procedure

TAL – Target Analyte List

TCLP – Toxicity Characteristic Leaching Procedure

VOC – Volatile Organic Compound

QAPP Worksheet #19
Analytical SOP Requirements Table

| Matrix | Analytical Group | Concentration Level | Analytical and Preparation Method/SOP Reference¹ | Sample Volume | Containers (number, size, and type) | Preservation Requirements (chemical, temperature, light protected) | Maximum Holding Time (preparation/analysis) |
|---------------------------|-------------------------|----------------------------|--|--------------------------|--|---|---|
| Vapor | Total VOCs | High/Medium/Low | U.S. EPA TO-15 | Approximately 5.5 liters | 6 liter summa canister | None | 30 days from collection to analysis |
| Soil | Total VOCs | High/Medium/Low | U.S. EPA SW-846 Method 8260B | Approximately 3 oz | Three Encore™ sampling devices and one 2oz container | MeOH and NaHSO ₄ | 48 days from collection to preservation in laboratory |
| Soil | TCLP VOCs | High/Medium/Low | U.S. EPA SW-846 Method 1131/8260B | Approximately 4 oz | 4-oz glass container | None | 14 days for extraction, 14 days for analysis |
| Equipment Rinsate (Water) | Total VOCs | Low | U.S. EPA SW-846 Method 8260B | Approximately 3 oz | Three 40mL VOA | HCl | 14 days from collection to analysis |

Notes:

Worksheet #19 content to be verified when ERRS selects and awards the analytical laboratory.

¹Specify the appropriate reference letter or number from the Analytical SOP References table (Worksheet #23).

HCl – Hydrochloric Acid

mL – Milliliter

MeOH – Methanol

NaHSO₄ – Sodium Bisulfate
Oz - Ounce
QAPP – Quality Assurance Project Plan
SOP – Standard Operating Procedure
VOC – Volatile organic compound

QAPP Worksheet #20

Field Quality Control Sample Summary Table

| Matrix | Analytical Group | Concentration Level | Analytical and Preparation SOP Reference¹ | No. of Sampling Locations² | No. of Field Duplicates | No. of MS/MSDs³ | No. of Field Blanks | No. of Equip. Blanks⁴ | Total No. of Samples to Lab |
|---------------|-------------------------|----------------------------|---|--|--------------------------------|-----------------------------------|----------------------------|---|------------------------------------|
| Soil | Total VOCs | High/Medium/Low | U.S. EPA SW-846 Method 8260B | 186 | 43 | 9 | 30 | 5 | 273 |
| Soil | TCLP VOCs | High/Medium/Low | U.S. EPA SW-846 Method 1131/8260B | 186 | 43 | 9 | 0 | 2 | 240 |
| Vapor | Total VOCs | High/Medium/Low | U.S. EPA TO-15 | 557 | 29 | 0 | 0 | 0 | 586 |

Notes:

¹Specify the appropriate reference letter or number from the Analytical SOP References table (Worksheet #23).

²If samples will be collected at different depths at the same location, count each discrete sampling depth as a separate sampling location or station.

³MS/MSDs are not additional samples, but are instead investigative samples on which MS analyses are performed.

⁴Equipment blanks listed will be equipment rinsate samples collected from preliminary soil sampling drilling.

MS – Matrix Spike

MSD – Matrix Spike Duplicate

QAPP – Quality Assurance Project Plan

SOP – Standard Operating Procedure

VOC – Volatile organic compound

QAPP Worksheet #21
Project Sampling SOP References Table

| Reference Number | Title, Revision Date and/or Number | Originating Organization | Equipment Type | Modified for Project Work? (Y/N) | Comments |
|----------------------------------|---|---------------------------------|--|---|--|
| WSOP-302 | WESTON START SOP 302 – Surface Soil Sampling | WESTON | Shovel, disposable gloves and scoop, sample jars and sample documentation | N | |
| WSOP-304 | WESTON START SOP 302 – Surface Soil Sampling | WESTON | Geoprobe, disposable gloves and scoop, sample jars and sample documentation | N | |
| SOP 1704 SOP 2042 SOP 2082 | U.S. EPA SOP 1704 – Summa Canister Sampling U.S. EPA SOP 2042 – Soil Gas Sampling U.S. EPA SOP 2082 – Construction and Installation of Permanent Sub-Slab Soil Gas Wells. | WESTON | Summa canisters, drill, vapor sampling ports, tubing, vacuum, modeling clay, cement powder, summa-tubing connectors, ppbRAE, MultiRAE, hand tools, flash light, and others | N | Ohio EPA’s May 2010 “Sample Collection and Evaluation of Vapor Intrusion to Indoor Air” will be used as guidance |

Notes:
EPA – Environmental Protection Agency
SOP – Standard Operating Procedure
START – Superfund Technical Assessment Response Team
QAPP – Quality Assurance Project Plan
WESTON – Weston Solutions, Inc.

QAPP Worksheet #22

Field Equipment Calibration, Maintenance, Testing, and Inspection Table

| Field Equipment | Calibration Activity | Maintenance Activity | Testing Activity | Inspection Activity | Frequency | Acceptance Criteria | Corrective Action | Responsible Person | SOP Reference ¹ |
|--|----------------------|----------------------|------------------|---------------------|-----------|---------------------|-------------------|--------------------|----------------------------|
| Not Applicable – Field screening not planned | | | | | | | | | |

Notes:

¹Specify the appropriate reference letter or number from the Project Sampling SOP References table (Worksheet #21).

SOP – Standard Operating Procedure

QAPP – Quality Assurance Project Plan

QAPP Worksheet #23
Analytical SOP References Table

| Reference Number | Title, Revision Date, and/or Number | Definitive or Screening Data | Analytical Group | Instrument | Organization Performing Analysis | Modified for Project Work? (Y/N) |
|-----------------------------------|---|-------------------------------------|-------------------------|-------------------------------------|---|---|
| U.S. EPA Method TO-15 | SOP for Determination of VOCs in Air Collected in Specially-Prepared Canisters and Analyzed by GC/MS; Revision 5, December 20, 2010 | Definitive | Total VOCs | Gas chromatograph/mass spectrometer | Pace Analytical Pace Analytical | N |
| U.S. EPA SW-846 Method 8260B | SOP for GC/MS Determination of VOCs and Volatile Petroleum Hydrocarbons; Revision 6, May 14, 2009 | Definitive | Total VOCs | Gas chromatograph/mass spectrometer | Pace Analytical | N |
| U.S. EPA SW-846 Method 1311/8260B | SOP for the TCLP for VOCs; Revision 7, April 12, 2012 | Definitive | TCLP VOCs | Gas chromatograph/mass spectrometer | Pace Analytical | N |
| U.S. EPA SW-846 Method 3550 | SOP for Preparation of Non-Aqueous Samples Using Sonication | Definitive | Total VOCs | Balance, glassware | Pace Analytical | N |

Notes:

GC- Gas Chromatography
LSOP – Laboratory Standard Operating Procedure
MS - Mass Spectrometry
QAPP – Quality Assurance Project Plan
SOP – Standard Operating Procedure
TCLP – Toxicity Characteristic Leaching Procedure
VOC – Volatile organic compounds

QAPP Worksheet #24
Analytical Instrument Calibration Table

| Instrument | Calibration Procedure | Frequency of Calibration | Acceptance Criteria | Corrective Action (CA) | Person Responsible for CA | SOP Reference¹ |
|-------------------|-----------------------------------|---------------------------------|----------------------------|-------------------------------|----------------------------------|--|
| GC/MS | See referenced SOPs in Appendix C | | | | Pace Analytical Chemist | U.S. EPA Method TO-15, U.S. EPA SW-846 Method 8260B, and U.S. EPA SW-846 Method 1311/8260B |

Notes:

¹Specify the appropriate reference letter or number from the Analytical SOP References table (Worksheet #23).

GC/MS – Gas Chromatography/ Mass Spectrometry

QAPP – Quality Assurance Project Plan

SOP – Standard Operating Procedure

QAPP Worksheet #25
Analytical Instrument and Equipment Maintenance, Testing, and Inspection Table

| Instrument/ Equipment | Maintenance Activity | Testing Activity | Inspection Activity | Frequency | Acceptance Criteria | Corrective Action | Responsible Person | SOP Reference¹ |
|----------------------------------|----------------------------------|-----------------------------|--------------------------------|------------------|--------------------------------|------------------------------|-------------------------------|--|
| GC/MS | See referenced SOP in Appendix C | | | | | | Pace Analytical Chemist | U.S. EPA Method TO-15, U.S. EPA SW-846 Method 8260B, and U.S. EPA SW-846 Method 1311/8260 B |

Notes:

¹Specify the appropriate reference letter or number from the Analytical SOP References table (Worksheet #23).

GC/MS – Gas Chromatography/ Mass Spectrometry

QAPP – Quality Assurance Project Plan

SOP – Standard Operating Procedure

QAPP Worksheet #26 Sample Handling System

| |
|--|
| SAMPLE COLLECTION, PACKAGING, AND SHIPMENT |
| <ul style="list-style-type: none"> Sample Collection (Personnel/Organization): WESTON START Site Lead |
| <ul style="list-style-type: none"> Sample Packaging (Personnel/Organization): WESTON START Site Lead or ERRS designated personnel |
| <ul style="list-style-type: none"> Coordination of Shipment (Personnel/Organization): ERRS Laboratory Procurement Coordinator or WESTON START Site Lead |
| <ul style="list-style-type: none"> Type of Shipment/Carrier: Federal Express, delivery, or courier pick-up |
| SAMPLE RECEIPT AND ANALYSIS |
| <ul style="list-style-type: none"> Sample Receipt (Personnel/Organization): Laboratory Sample Login (TBD) |
| <ul style="list-style-type: none"> Sample Custody and Storage (Personnel/Organization): Laboratory Sample Receipt (TBD) |
| <ul style="list-style-type: none"> Sample Preparation (Personnel/Organization): Laboratory Personnel (TBD) |
| <ul style="list-style-type: none"> Sample Determinative Analysis (Personnel/Organization): Laboratory Personnel (TBD) |
| SAMPLE ARCHIVING |
| <ul style="list-style-type: none"> Field Sample Storage (No. of days from sample collection): All samples will be sent to the laboratory. The laboratory shall retain the samples in accordance with their laboratory SOPs. |
| <ul style="list-style-type: none"> Sample Extract/Digestate Storage (No. of days from extraction/digestion): Six months |
| <ul style="list-style-type: none"> Biological Sample Storage (No. of days from sample collection): Not Applicable |
| SAMPLE DISPOSAL |
| <ul style="list-style-type: none"> Personnel/Organization: Pace Analytical |
| <ul style="list-style-type: none"> Number of Days from Analysis: In accordance with the laboratory SOPs. |

Notes:

ERRS – Emergency and Rapid Response Services

SOPs – Standard Operating Procedures

START – Superfund Technical Assessment and Response Team

TBD – To Be Determined

WESTON – Weston Solutions, Inc.

QAPP Worksheet #27

Sample Custody Requirements

Chain-of-Custody Procedures: A COC record will be maintained from the time the sample is collected until its delivery to the laboratory. To maintain a record of sample collection, transfer between personnel, shipment, and receipt by the laboratory, a COC record will be filled out for each sample at each sampling location. Each individual in possession of the samples must sign and date the sample COC document. Each time the samples are transferred, the signatures of the persons relinquishing and receiving the samples, as well as the date and time, will be documented. A copy of the COC is retained by the site leader for the site file. When samples (or groups of samples) are not under direct control of the individual responsible for them, they must be stored in a locked container sealed with a custody seal. The COC record will be considered completed upon receipt at the laboratory. The COC record should include (at minimum) the following:

- Type (s) of analysis(es) to be performed
- Sample ID number
- Sample information
- Sample station location
- Sample date
- Name(s) and signature(s) of sampler(s)
- Signature(s) of any individual(s) with control over samples

A separate COC form must accompany each cooler in each shipment. Within the laboratory, the person responsible for sample receipt must sign and date the COC form; verify that custody seals are intact on shipping containers; compare samples received against those listed on the COC form; examine all samples for possible shipping damage, leakage, and improper sample preservation; note on the COC record or laboratory receiving documentation that specific samples were damaged; notify sampling personnel as soon as possible so that appropriate samples may be resampled; verify that sample holding times have not been exceeded; maintain laboratory COC documentation; and place the samples in appropriate laboratory storage. If requested, the laboratory may submit internal COC documentation with the data package. Final sample disposition is completed according to laboratory license requirements.

Sample Identification Procedures: All samples for laboratory analysis, including QC samples, will be given a unique sample number. The sample numbers will be recorded in the field logbook, the COC paperwork, and the shipment documents. The sample number highlights the suspected contaminated area and location, and will be used for documentation purposes in field logbooks, as well as for presentation of the analytical data in memoranda and reports.

The sample numbering system for the preliminary soil samples will be composed of the components below:

KP-SBXX-(X-X)GGG-MMDDYY

Where:

- “KP” indicates that the sample is from the Kiser Plating Removal Action Site
- “SB” indicates the sample is collected from the a soil boring
- “XX” indicates the soil boring number
- “X-X” indicates the depth of the sample
- “GGG” indicates the grid location sampled
- “MMDDYY” indicates the date sampled

The sample numbering system for the preliminary soil equipment rinsate samples will be composed of the components below:

KP-ERXX-MMDDYY

Where:

- “KP” indicates that the sample is from the Kiser Plating Removal Action Site
- “ER” indicates the sample is collected from equipment rinsate
- “XX” indicates the equipment rinsate number
- “MMDDYY” indicates the date sampled

The sample numbering system for indoor air samples will be composed of the components below:

KP-SG-123Main-MMDDYY

Where:

- “KP” indicates that the sample is from the Kiser Plating Removal Action Site
- “SG” indicates the sample matrix is soil gas
- “123Main” indicates the address where the soil gas sample was collected from
- “MMDDYY” indicates the date sampled

KP-SS-123Main-MMDDYY

Where:

- “KP” indicates that the sample is from the Kiser Plating Removal Action Site
- “SS” indicates the sample matrix is subslab vapor
- “123Main” indicates the address where the soil gas sample was collected from

- “MMDDYY” indicates the date sampled

KP-IA-123Main- MMDDYY

Where:

- “KP” indicates that the sample is from the Kiser Plating Removal Action Site
- “IA” indicates the sample matrix is indoor air
- “123Main” indicates the address where the soil gas sample was collected from
- “MMDDYY” indicates the date sampled

KP-AA-123Main- MMDDYY

Where:

- “KP” indicates that the sample is from the Kiser Plating Removal Action Site
- “AA” indicates the sample matrix is ambient air
- “123Main” indicates the address where the soil gas sample was collected from
- “MMDDYY” indicates the date sampled

The sample numbering system for confirmation soil samples will be composed of the components below:

KP-CS-GGG-L-MMDDYY

Where:

- “KP” indicates that the sample is from the Kiser Plating Removal Action Site
- “CS” indicates the sample is a confirmation soil sample
- “GGG” indicates the grid location sampled
- “L” indicates the sample location; W – wall or B – bottom of excavation
- “MMDDYY” indicates the date sampled

An example of the sample identifications for the Site is as follows:

- **KP-SBAA-3-4:** Soil sample collected from soil boring grid AA from 0 to 2 feet below ground surface.
- **KP-AA-061913:** Kiser Plating Removal Action Site soil gas sample collected from location identified by AA on June 19, 2013
- **KP-SS-630Clay-102213:** Kiser Plating Removal Action Site sub-slab vapor sample collected from 630 Clay on October 22, 2013
- **KP-IA-150044th-102213:** Kiser Plating Removal Action Site indoor air sample collected from 1500 44th on October 22, 2013

- **KP-AA-150044th-102213:** Kiser Plating Removal Action Site ambient air sample collected outside 1500 44th on October 22, 2013
- **KP-CS-AA-B-102213:** Confirmation soil sample collected from the bottom of the Grid F02 excavation at the Kiser Plating Removal Action Site on October 22, 2013

Notes:

COC – Chain-of-Custody

ID - Identification

MS/MSD – Matrix spike/matrix spike duplicate

QC – Quality Control

U.S. EPA – United States Environmental Protection

Agency

QAPP Worksheet #28

QC Samples Table

| | |
|---|----------------------|
| Matrix | Vapor / Soil |
| Analytical Group | Total VOCs |
| Concentration Level | High/Medium/Low |
| Analytical Method/ SOP Reference | TO-15 / SW-846 8260B |
| Sampler's Name: | Keith Hughes |
| Field Sampling Organization: | WESTON START |
| Analytical Organization: | Pace Analytical |
| Sampling SOP | See Worksheet #17 |
| No. of Sample Locations: | 859 |

| QC Sample | Frequency / Number | Method/SOP QC Acceptance Limits | Corrective Action | Person(s) Responsible for Corrective Action | Data Quality Indicator (DQI) | Measurement Performance Criteria |
|---|---------------------------|--|--|--|---|---|
| Laboratory Duplicate (LCS duplicate) | 1/10 | RPD \leq 20% | Flag associated data as estimated | Chemist | Laboratory Precision | See EPA Method TO-15 and U.S. 846 8260B in Appendix C |
| Method Blank | 1/20 | No target analyte concentrations above reporting limit | Flag data at less than 10 times the blank concentratio n as not detected | Chemist | Laboratory Contamination | See EPA Method TO-15 and U.S. 846 8260B in Appendix C |

| | | | | | | |
|--------|------|---|-----------------------------------|---------|---|---|
| LCS | 1/20 | % Recovery in accordance with laboratory limits | Flag associated data as estimated | Chemist | Laboratory Accuracy | See EPA Method TO-15 and U.S. 846 8260B in Appendix C |
| MS/MSD | 1/20 | % Recovery in accordance with laboratory limits | Flag associated data as estimated | Chemist | Matrix Interference/ Laboratory Accuracy | See EPA Method TO-15 and U.S. 846 8260B in Appendix C |

Notes:

DQI – Data Quality Indicator

ERRS – Emergency and Rapid Response Services

QAPP – Quality Assurance Project Plan

QC – Quality Control

LCS/LCSD – Laboratory Control Sample/Laboratory Control Sample Duplicate

MS/MSD – Matrix Spike/Matrix Spike Duplicate

RL – Reporting Limit

RPD – Relative Percent Difference

SOP – Standard Operation Procedure

WESTON – Weston Solutions, Inc.

QAPP Worksheet #29
Project Documents and Records Table

| Sample Collection Documents and Records | On-Site Analysis Documents and Records | Off-Site Analysis Documents and Records | Data Assessment Documents and Records | Other |
|--|---|---|--|------------------------------|
| Logbook(s) | Logbook(s) | Sample Receipt, Custody, and Tracking Records | Data Validation Reports | Investigation Summary Report |
| COC Forms | Final Analytical Data Summary Report | Preliminary Analytical Data Reports | Corrective Action Reports | |
| Photos | | Final Analytical Data Summary Reports | | |
| Field Diagrams | | Laboratory Electronic Data Deliverables | | |
| GPS Coordinates | | Sample Preparation Logs | | |
| Vapor Intrusion Sample Log | | Corrective Action Reports | | |
| | | Equipment Maintenance, Testing, and Inspection Logs | | |
| | | Quality Control Sample Summary Forms | | |
| | | Sample Disposal Records | | |

Notes:
COC – Chain-of-Custody
GPS – Global Positioning Satellite
QAPP – Quality Assurance Project Plan

**QAPP Worksheet #30
Analytical Services Table**

| Matrix | Analytical Group | Concentration Level | Sample Locations/ ID Numbers¹ | Analytical SOP | Data Package Turnaround Time | Laboratory / Organization (Name and Address, Contact Person and Telephone Number) | Backup Laboratory / Organization (Name and Address, Contact Person and Telephone Number) |
|---------------|-------------------------|----------------------------|---|------------------------------------|-------------------------------------|---|---|
| Soil | Total VOCs | High/Medium/Low | On-site subsurface soil / KP-SBAA-(X-X) through KP-SBHC--(X-X) Side walls and bottom of the on-site excavation / KP-CS-GGG-L-MMDDYY through KP-CS-GGG-L-MMDDYY | U.S. EPA SW-846 Method 8260B | 1 week | Pace Analytical Services 7726 Moller Rd, Indianapolis, IN 46268 (317) 875-5894 | TBD |
| Soil | TCLP VOCs | High/Medium/Low | On-site subsurface soil / KP-SB01-(X-X)-GGG-MMDDYY through TC-SBXX--(X-X)-GGG-MMDDYY Side walls and bottom of the on-site excavation / KP-CS-GGG-L-MMDDYY through KP-CS-GGG-L-MMDDYY | U.S. EPA SW-846 Method 1131/8260 B | 1 week | Pace Analytical Services 7726 Moller Rd, Indianapolis, IN 46268 (317) 875-5894 | TBD |

| | | | | | | | |
|-------|------------|---------------------|--|------------------------|--------|--|-----|
| Vapor | Total VOCs | High/Medium/ Low | Soil gas, subslab vapor, indoor air; KP-SG- 123Main-MMDDYY through KP-SG-999Clay- MMDDYY, KP-SS- 123Main-MMDDYY through KP-SS-999Clay- MMDDYY, KP-IA- 123Main-MMDDYY through KP-IA-999Clay- MMDDYY, KP-AA- 123Main-MMDDYY through KP-AA- 999Clay-MMDDYY | EPA Method TO-15 | 1 week | Pace Analytical Services 1700 SE Elm St, Minneapolis, MN 55414 (612) 607-1700 | TBD |
|-------|------------|---------------------|--|------------------------|--------|--|-----|

Notes:

¹See Worksheet #27 for a description of sampled numbers to be used

ID – Identification

QAPP – Quality Assurance Project Plan

SOP – Standard Operating Procedure

TBD – To Be Determined

TCLP – Toxicity Characteristic Leaching Procedure

VOC – Volatile Organic Compound

QAPP Worksheet #31
Planned Project Assessments Table

| Assessment Type | Frequency | Internal or External | Organization Performing Assessment | Person(s) Responsible for Performing Assessment (Title and Organizational Affiliation) | Person(s) Responsible for Responding to Assessment Findings (Title and Organizational Affiliation) | Person(s) Responsible for Identifying and Implementing CA (Title and Organizational Affiliation) | Person(s) Responsible for Monitoring Effectiveness of CA (Title and Organizational Affiliation) |
|--|------------------|-----------------------------|---|---|---|---|--|
| Not Applicable - A field audit is not planned for this assessment. | | | | | | | |

Notes:

CA – Corrective Action

QAPP – Quality Assurance Project Plan

QAPP Worksheet #32
Assessment Findings and Response Actions

| Assessment Type | Nature of Deficiencies Documentation | Individual(s) Notified of Findings (Name, Title, Organization) | Timeframe of Notification | Nature of Corrective Action Response Documentation | Individual(s) Receiving Corrective Action Response (Name, Title, Org.) | Timeframe for Response |
|--|---|---|----------------------------------|---|---|-------------------------------|
| Not Applicable - A field audit is not planned for this assessment. | | | | | | |

Notes:
QAPP – Quality Assurance Project Plan

QAPP Worksheet #33
QA Management Reports Table

| Type of Report | Frequency (daily, weekly monthly, quarterly, annually, etc.) | Projected Delivery Date(s) | Person(s) Responsible for Report Preparation (Title and Organizational Affiliation) | Report Recipient(s) (Title and Organizational Affiliation) |
|-----------------------|--|---|--|---|
| Data Compliance Check | To be prepared following receipt of an analytical data package | Two weeks following receipt of final data package from laboratory | Richie Byrd, RM. ERRS will perform a compliance check of the data. | Shelly Lam, OSC, U.S. EPA Region V |
| Final Project Report | To be prepared upon receipt of the analytical data validation report | Six months following receipt of all data validation reports | Dan Liebau, PM, WESTON START | Shelly Lam OSC, U.S. EPA Region V |
| Monthly Report | Every month for the prior month activities | Monthly | Dan Leibau, PM, WESTON START | Shelly Lam, OSC, U.S. EPA Region V |

Notes:

ERRS – Emergency and Rapid Response Services
OSC – On-Scene Coordinator
PM – Project Manager
QA – Quality Assurance
QAPP – Quality Assurance Project Plan
TBD – To Be Determined
U.S. EPA – United States Environmental Protection Agency
WESTON – Weston Solutions, Inc.

QAPP Worksheet #34
Sampling and Analysis Verification (Step I) Process Table

| Verification Input | Description | Internal/ External | Responsible for Verification (Name, Organization) |
|---------------------------|---|---------------------------|---|
| COC Forms | The site leader will submit COC forms to the ERRS SMC within 2 hours following all sample shipments to the laboratory. The PM and SMC will review the COC forms for completeness to ensure that the proper analyses are being performed. | Internal | Richie Byrd, ERRS RM, SMC Dan Liebau, PM, WESTON START |
| Logbook | The PM will review the logbook for accuracy and completeness following field sampling activities. | Internal | Dan Liebau, PM, WESTON START |
| Laboratory Data | All laboratory data will be verified by the QA officer of the laboratory performing the sample analyses. The laboratory data will be evaluated in accordance with the procedures described in Worksheet #35 and Worksheet #36. The ERRS RM will perform a compliance check of all data received from the laboratory. | External Internal | QA Officer, Laboratory Richie Byrd, ERRS RM, SMC |

Notes:

COC – Chain-of-Custody
ERRS – Emergency and Rapid Response Services
PM – Project Manager
QA – Quality Assurance
QAPP – Quality Assurance Project Plan
RM – Response Manager
START – Superfund Technical Assessment and Response Team
WESTON – Weston Solutions, Inc

QAPP Worksheet #35
Sampling and Analysis Validation (Steps IIa and IIb) Process Table

| Step IIa/IIb | Validation Input | Description | Responsible for Validation (Name, Organization) |
|---------------------|--|---|--|
| IIa | SOPs and logbook | The PM and site leader will ensure that all SOPs were followed in the field. | Dan Liebau, PM and Keith Hughes, Site Leader, WESTON START |
| IIb | Preliminary Data and Final Analytical Data Package | The ERRS SMC will review the preliminary data and final analytical data package to ensure that all analyses requested were received and to ensure that required project quantitation limits were met. | Richie Byrd, ERRS ER, SMC |
| IIb | Final Analytical Data Package | The data validator will perform data validation of the final analytical data package to ensure that all QC requirements specified in the QAPP were met. ERRS will perform a compliance check of all validated data. | TBD |

Notes:

ERRS – Emergency and Rapid Response Services
PM – Project Manager
QAPP – Quality Assurance Project Plan
QC – Quality Control
SMC – Sample Management Coordinator
SOP – Standard Operating Procedure
START – Superfund Technical Assessment and Response Team
TBD – To Be Determined
WESTON – Weston Solutions, Inc

QAPP Worksheet #36
Sampling and Analysis Validation (Steps IIa and IIb) Summary Table

| Step IIa/IIb | Matrix | Analytical Group | Concentration Level | Validation Criteria | Data Validator (title and organizational affiliation) |
|---------------------|---------------|-------------------------|----------------------------|----------------------------|--|
| IIb | Vapor/Soil | Total VOCs | High/Medium/Low | TBD | ERRS SMC |

Notes:

ERRS – Emergency and Rapid Response Services

QAPP – Quality Assurance Project Plan

TBD – To Be Determined

VOC – Volatile organic compound

QAPP Worksheet #37 Data Usability Assessment

Summarize the usability assessment process and all procedures, including interim steps and any statistics, equations, and computer algorithms that will be used: Data, whether generated in the field or by the laboratory, are tabulated and reviewed for precision, accuracy, representativeness, and completeness by the site leader for field data or by the data validator for laboratory data from a fixed laboratory. The review of these data quality indicators (DQI) will compare the DQI with the data quality objects (DQO) detailed in the project-specific QAPP and in the analytical methods used.

Questions about data, as observed during the data review process, are resolved by contacting the respective site personnel and laboratories for resolution. All communications are documented including the resolution to the observed deficiencies. Hard copies of all original data and deliverables are kept in the Technical Direction Document (TDD) file.

When the data do not meet the project DQOs, ERRS will investigate the root cause to the deficiency. Reasons may include laboratory operation, failure of laboratory reporting limits to meet Site action limits, or poor correlation between field screening and laboratory results. In these situations, ERRS will discuss corrective actions with the OSC. These actions may include:

- Resampling for all or some of the parameters
- Preparing a technical memorandum to the site file, detailing limitations to the data
- Validating the data at a higher tier level to better qualify the results
- Preparing a technical memorandum determining the bias of field results

Describe the evaluative procedures used to assess overall measurement error associated with the project: The following specific items will be assessed in the manner described below:

Precision – Results of all laboratory duplicates and field duplicates will be presented in the laboratory data validation report. For each duplicate pair, the relative percent difference (RPD) will be calculated for each analyte with results greater than or equal to the quantitation limit. The RPDs will be checked against the measurement performance criteria presented on Worksheet #12. The RPDs exceeding criteria will be identified on the tables in the final report with appropriate qualifiers. A discussion will follow summarizing the results of the laboratory precision. Any conclusions about the precision of the analyses will be drawn and any

limitations on the use of the data will be described in the final report.

Accuracy/Bias Contamination – Results for all laboratory method blanks and instrument blanks will be presented in the laboratory data validation report. The results for each analyte will be checked against the measurement performance criteria presented on Worksheet #12. Results for analytes that exceed criteria will be identified on the tables in the final report with appropriate qualifiers. A discussion will follow summarizing the results of the laboratory accuracy/bias. Any conclusions about the accuracy/bias of the analyses based on contamination will be drawn and any limitations on the use of the data will be described.

Overall Accuracy/Bias – The results for the continuing calibration standards will be presented in the laboratory case narrative. These results will be compared to the requirements listed on Worksheet #12. A discussion will follow summarizing overall accuracy/bias. Any conclusions about the overall accuracy/bias of the analyses will be drawn and any limitations on the use of the data will be described.

Sensitivity – All sample results for monitoring well samples will be presented in tabular format. The sample results for each analyte will be checked against the method detection limits. Results for analytes that do not meet the contract required quantitation limits will be discussed. Any conclusions about the sensitivity of the analyses will be drawn and any limitations on the use of the data will be described.

Representativeness – Representativeness will be maintained by the site leader who will ensure that all sampling personnel are adhering to the sampling procedures dictated in the field sampling plan. In addition, the project manager will be in close contact with the field team leader to ensure that proper sampling techniques are being followed. Any conclusions about the representativeness of the sampling will be drawn and any limitations on the use of the data will be described.

Completeness – A completeness check will be done on all samples collected in the field and data generated by the laboratory. Completeness criteria are presented on Worksheet #12. Completeness will be calculated as follows. For each sample collected, completeness will be calculated as the number of samples collected and number of analyses performed, divided by the total number of planned sample collection points and analyses. A discussion will follow summarizing the calculation of data completeness. Any conclusions about the completeness of the data for each analyte will be drawn and any limitations on the use of the data will be described.

Reconciliation – Each of the project quality objectives presented on Worksheet #12 will be examined to determine if the objective was met. Each analysis will first be evaluated in terms of the major impacts observed from the data validation, DQIs, and measurement performance criteria assessments. Based on the results of these assessments, the quality of the data will be determined. Based on the quality determined, the usability of the data for each analysis will be determined. Based on the usability of the data from all analyses for an objective, it will be determined if the project quality objective was met. The final report will include a summary of all the points that went into the reconciliation of each objective. As part of the reconciliation of each objective, conclusions will be drawn and any limitations on the usability of any of the data will be described.

Identify the personnel responsible for performing the usability assessment: The site leader will determine the usability of field data. The ERRS or START Chemist will validate the data and the ERRS or WESTON Sample Management Coordinator will do a compliance check of the data to determine the usability of analytical data. The Project Manager, Sarah Meyer, will be responsible for the overall usability to meet project objectives.

Describe the documentation that will be generated during usability assessment and how usability assessment results will be presented so that they identify trends, relationships (correlations), and anomalies: A data validation report will be prepared. Overall usability of data to meet project objectives will be described in the final report to be prepared by the Project Manager.

Notes:

DQI – Data Quality Indicator

DQO – Data Quality Objective

ERRS – Emergency and Rapid Response Services

OSC – On-Scene Coordinator

QAPP – Quality Assurance Project Plan

RPD – Relative Percent Difference

SMC – Sample Management Coordinator

START – Superfund technical Assessment and Response Team

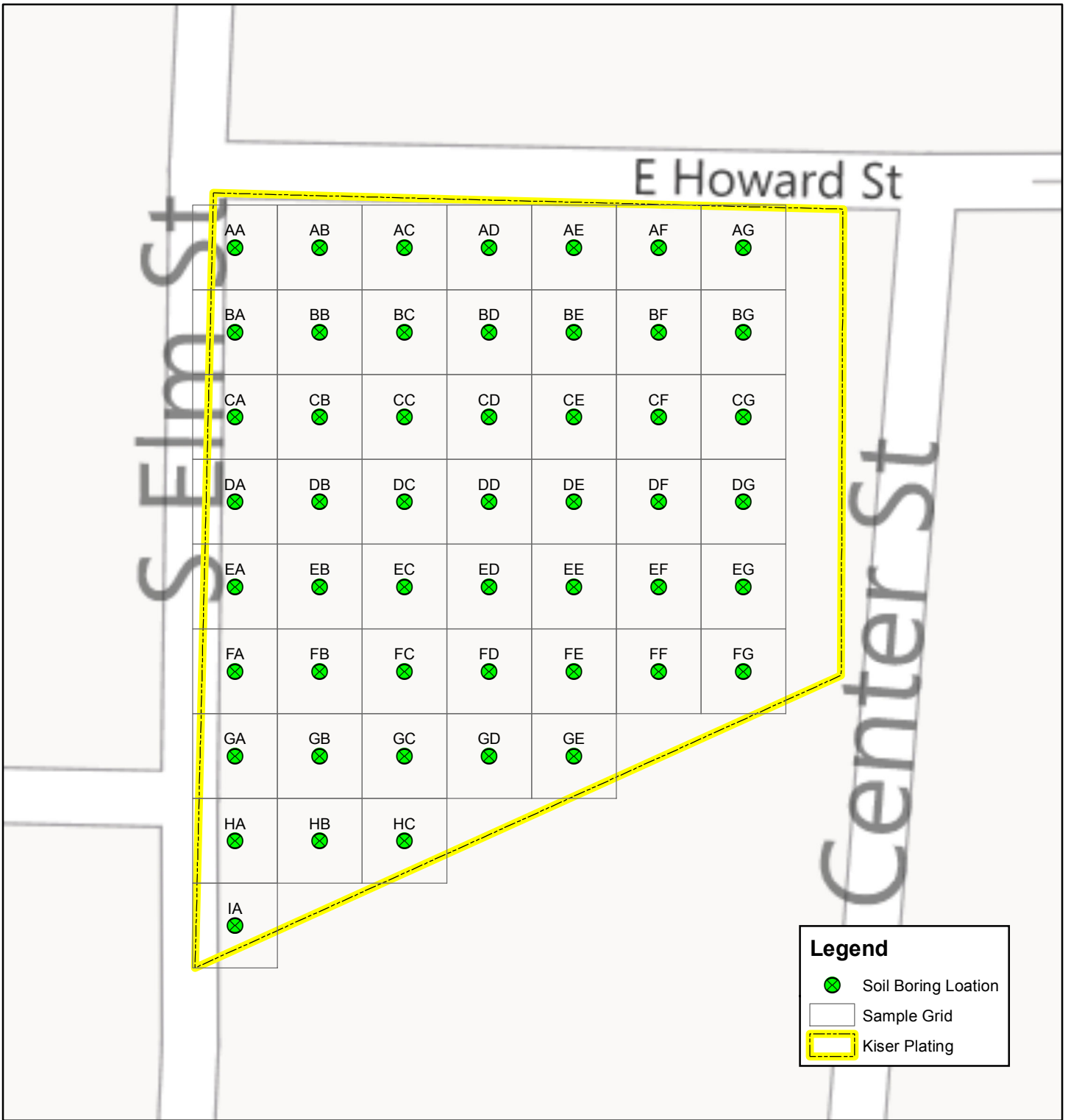
TDD – Technical Direction Document

U.S. EPA – United States Environmental Protection Agency

WESTON – Weston Solutions, Inc.

APPENDIX A

FIGURES

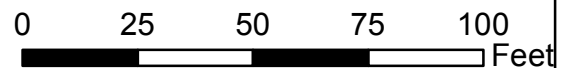


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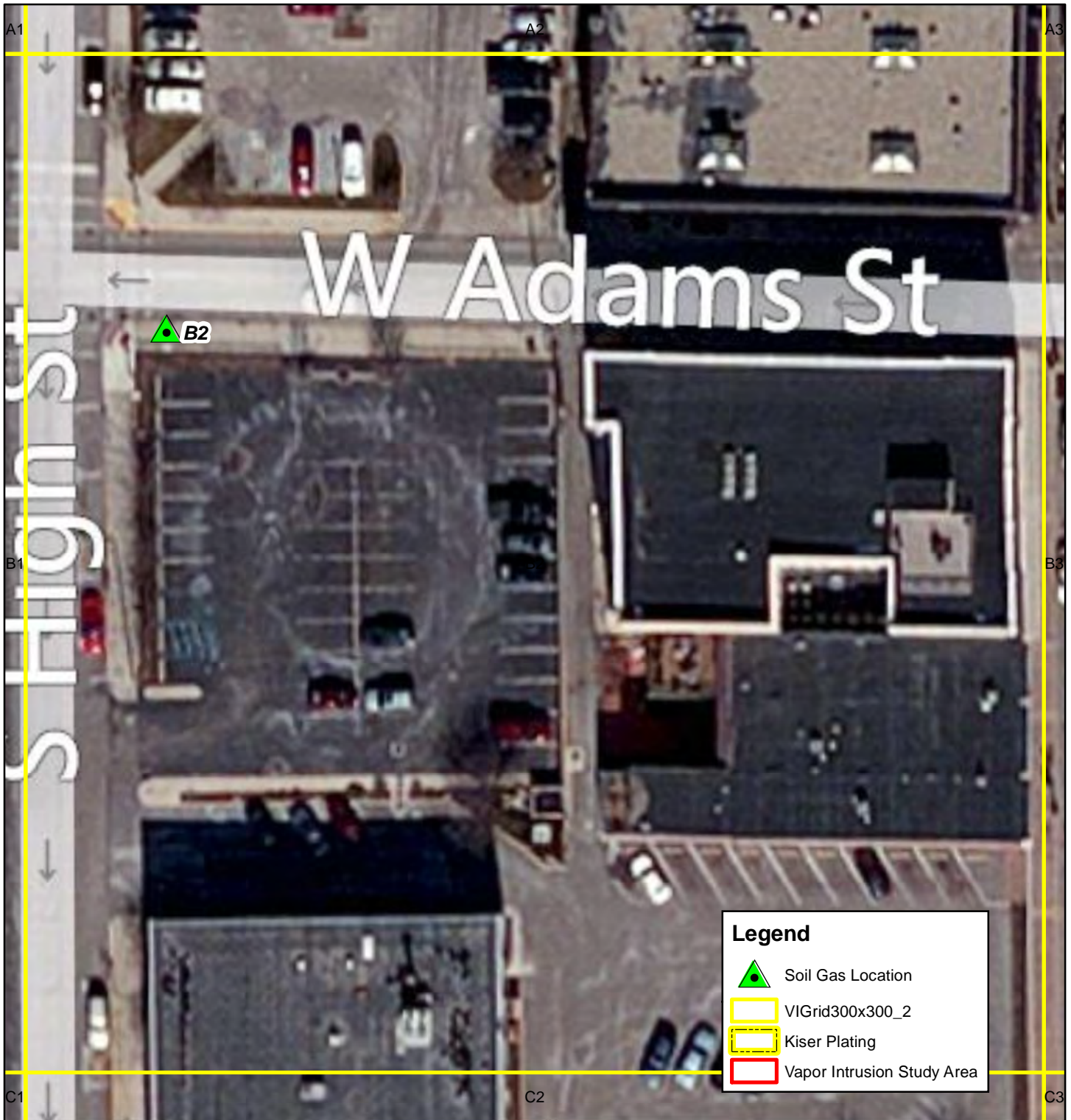
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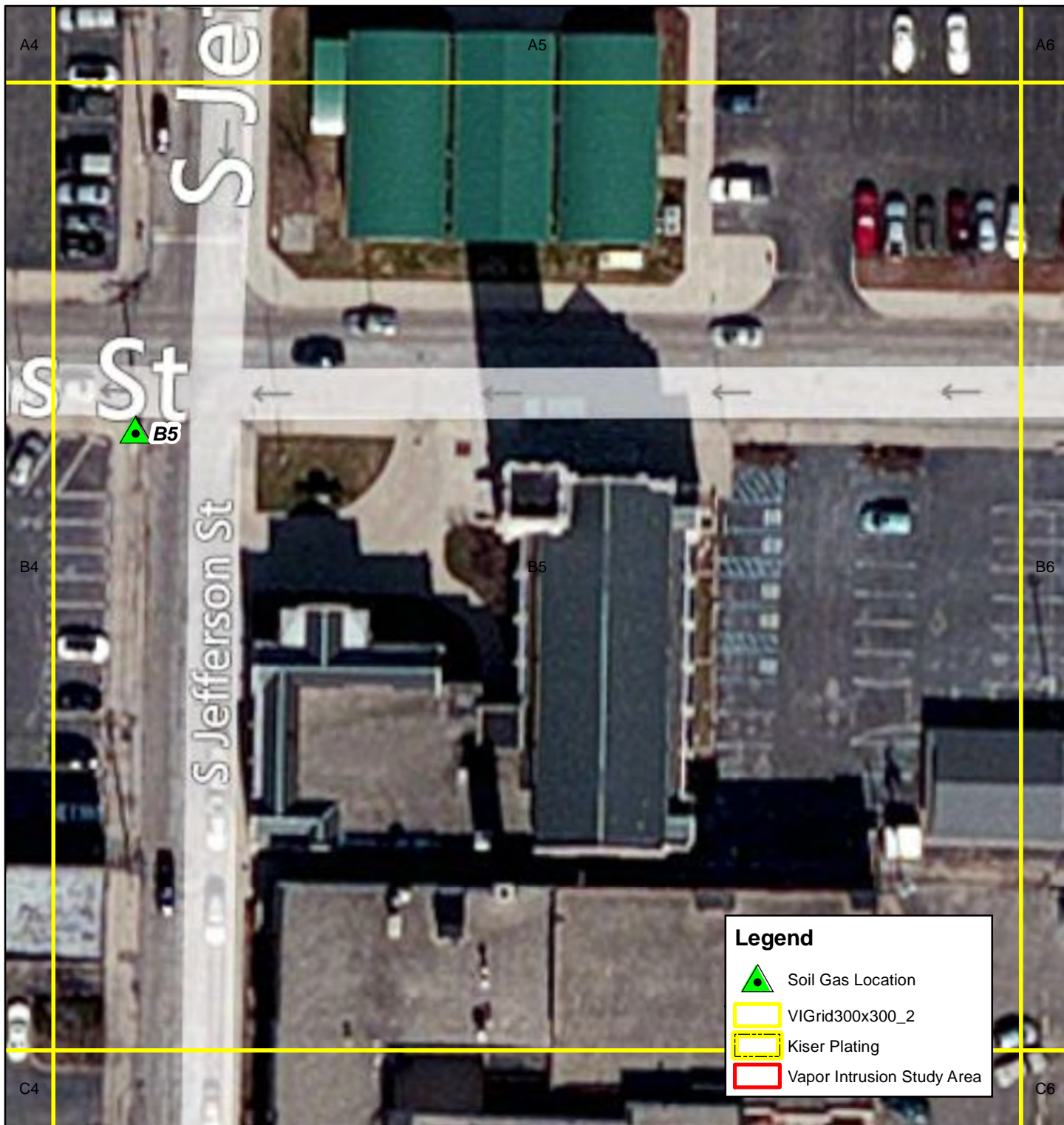
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Legend

-  Soil Gas Location
-  VIGrid300x300_2
-  Kiser Plating
-  Vapor Intrusion Study Area

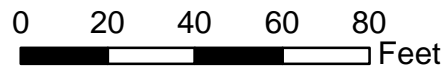


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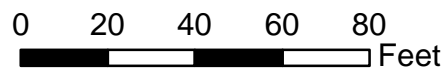


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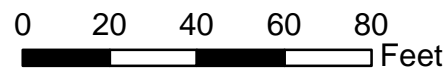


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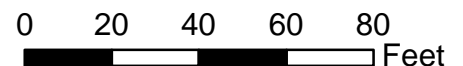


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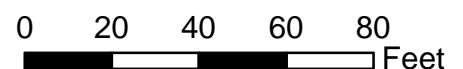


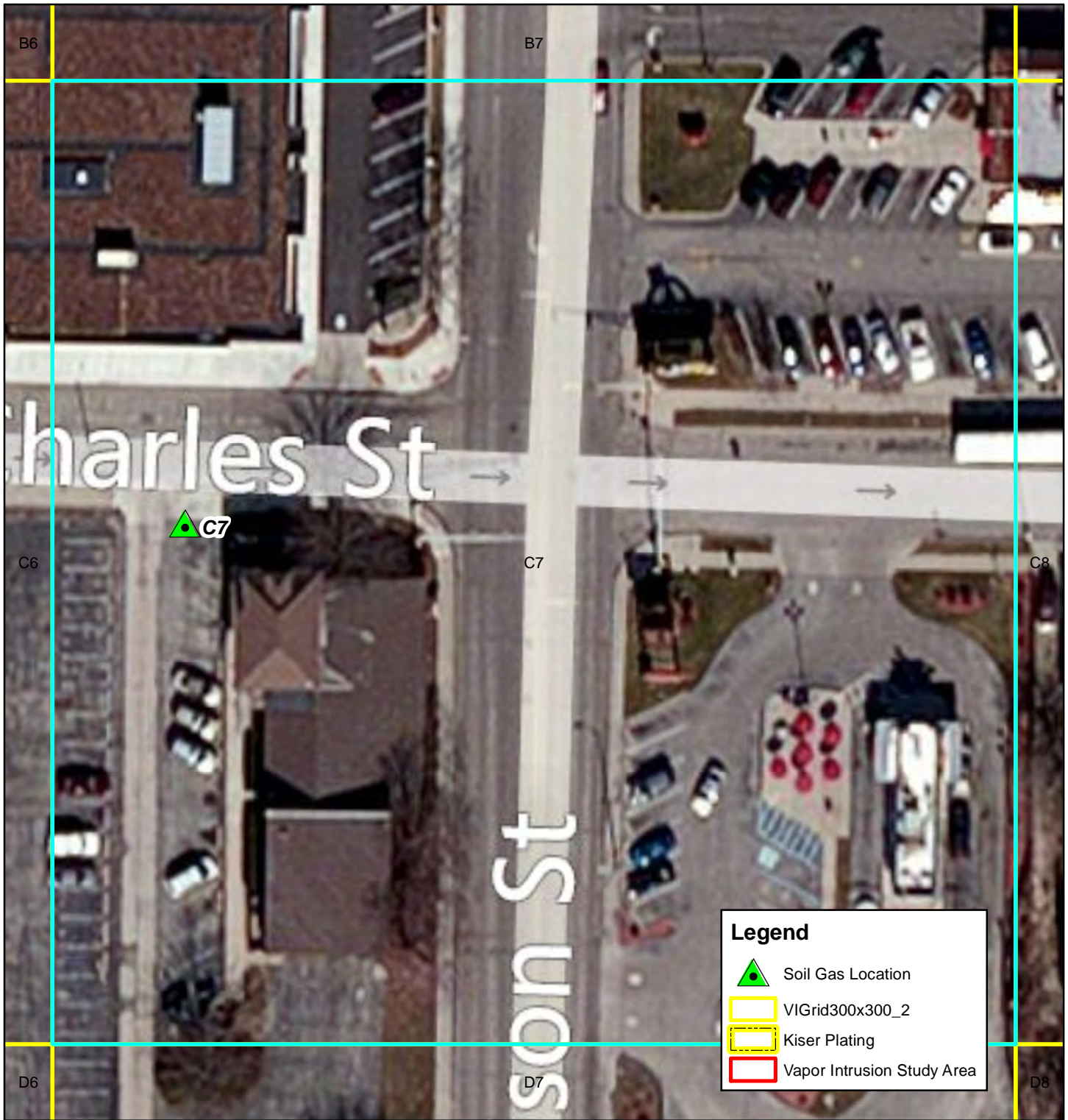
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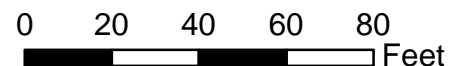


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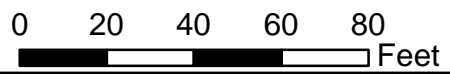


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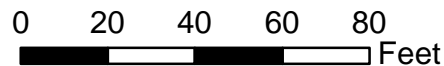


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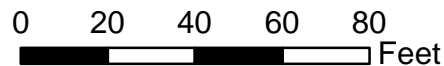


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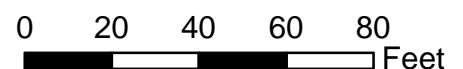


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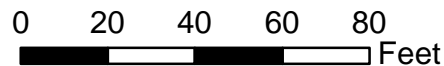


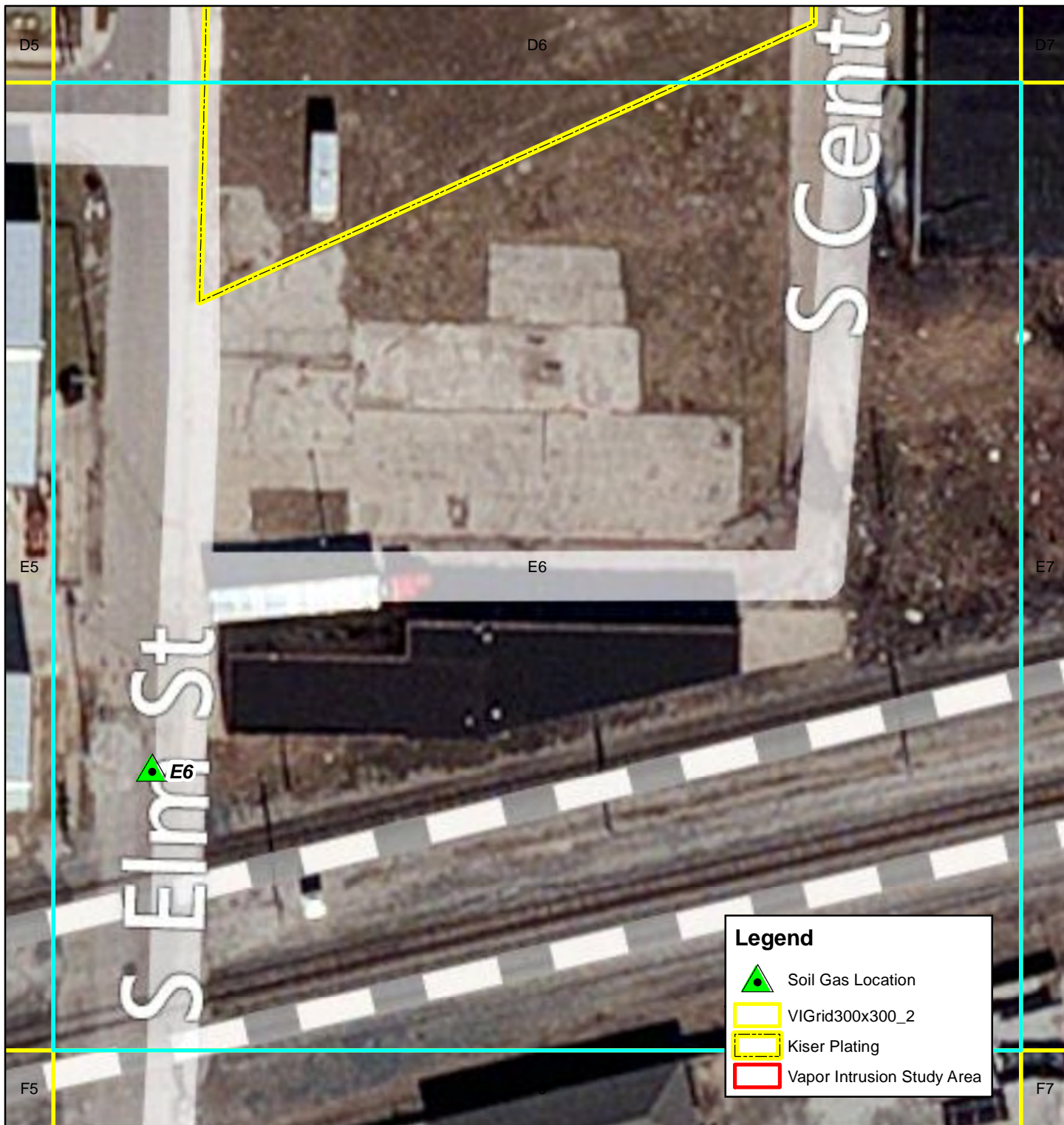
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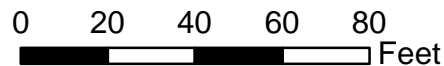


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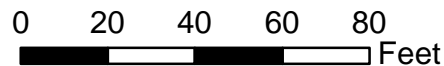


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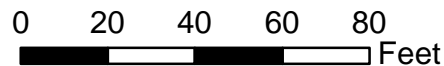


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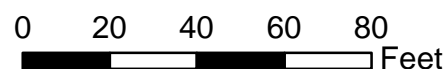


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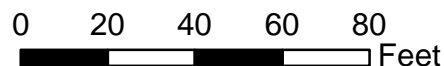


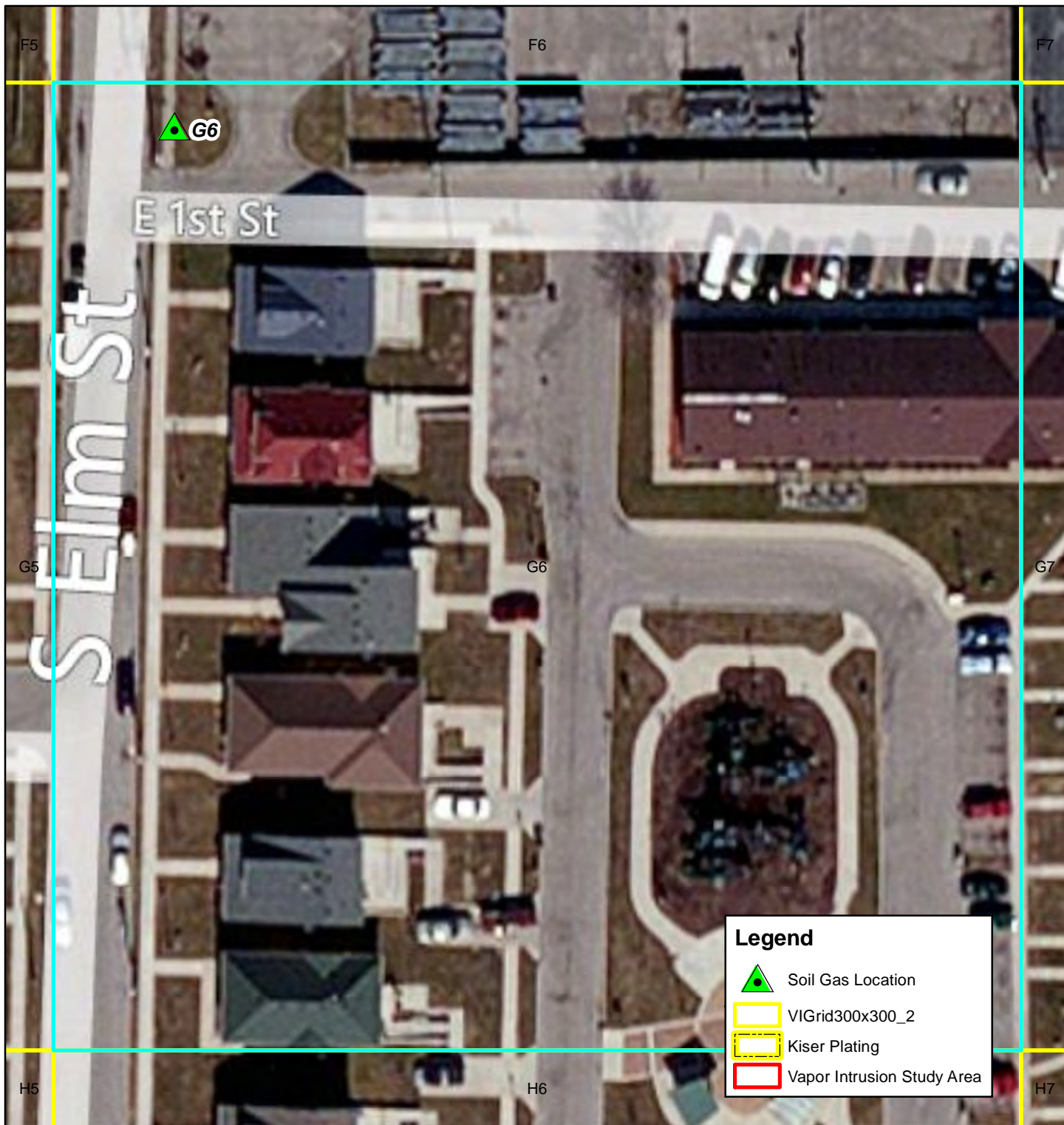
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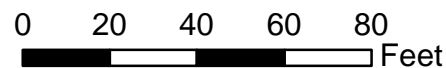


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KISER PLATING
401 E. HOWARD STREET
MUNCIE, DELAWARE COUNTY, INDIANA**



This map presents land cover imagery for the world and detailed topographic maps for the United States. The map includes the National Park Service (NPS) Natural Earth physical map at 1:24km per pixel for the world at small scales, i-cubed eTOPO 1:250,000-scale maps for the contiguous United States at medium scales, and National Geographic TOPO! 1:100,000 and 1:24,000-scale maps (1:250,000 and 1:63,000 in Alaska) for the United States at large scales. The TOPO! maps are seamless, scanned images of United States Geological Survey (USGS) paper topographic maps.

1:528





Legend

- Soil Gas Location
- VI Grid 300x300_2
- Kiser Plating
- Vapor Intrusion Study Area

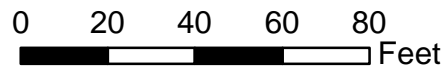


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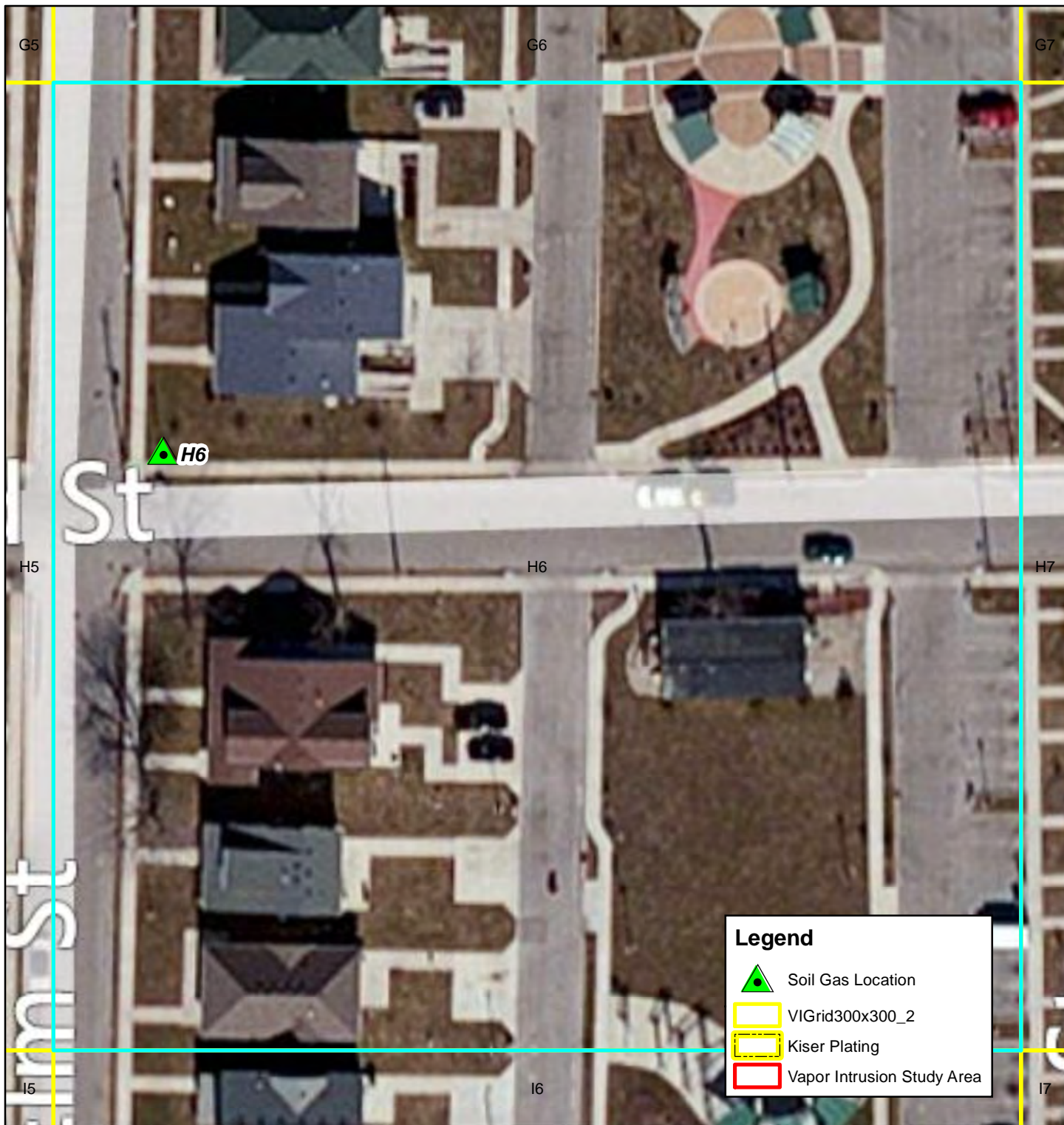
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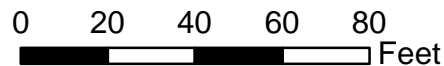


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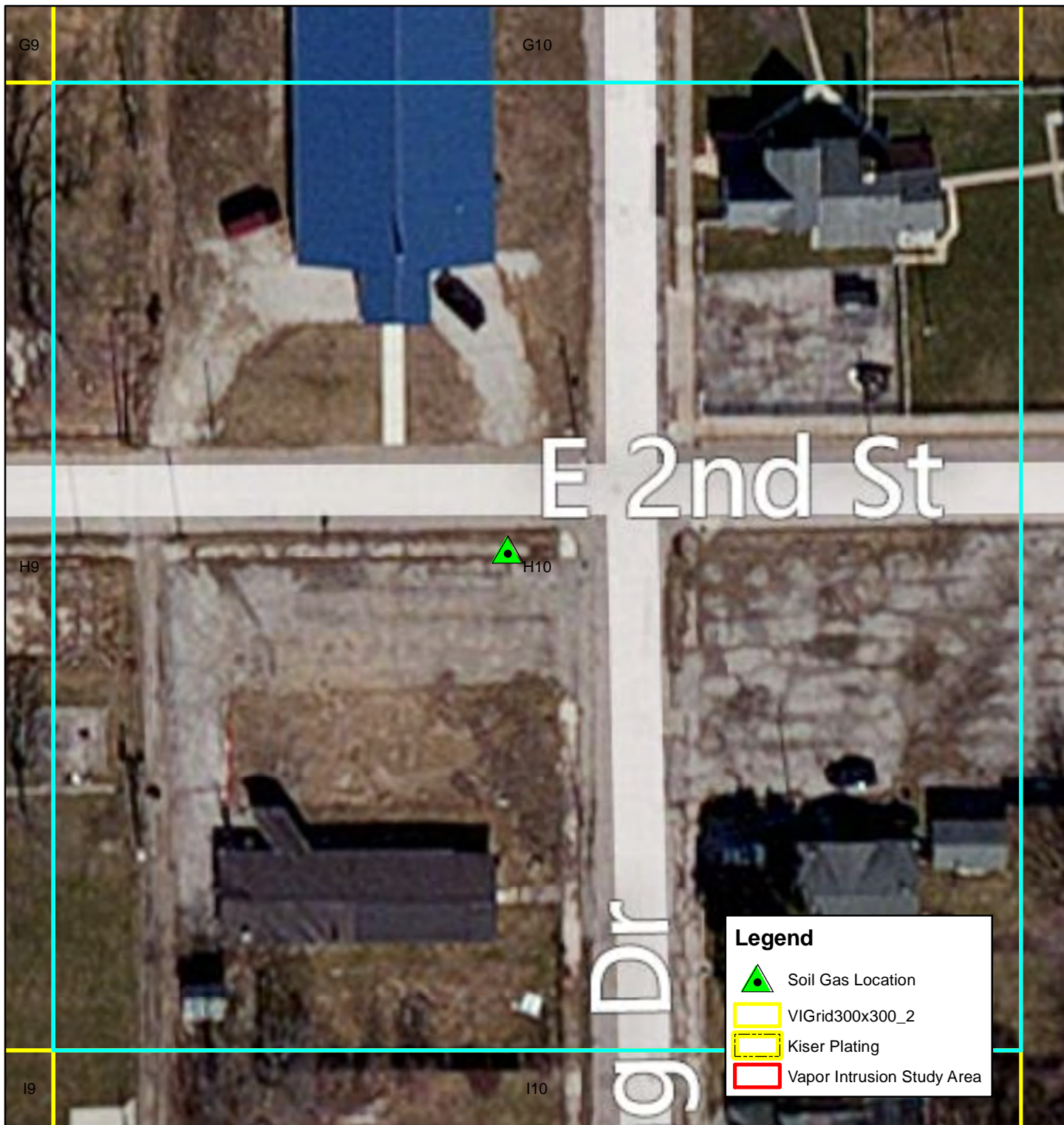
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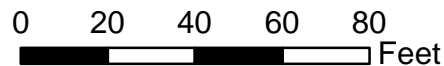


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Legend

- Soil Gas Location
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- Kiser Plating
- Vapor Intrusion Study Area

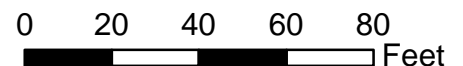


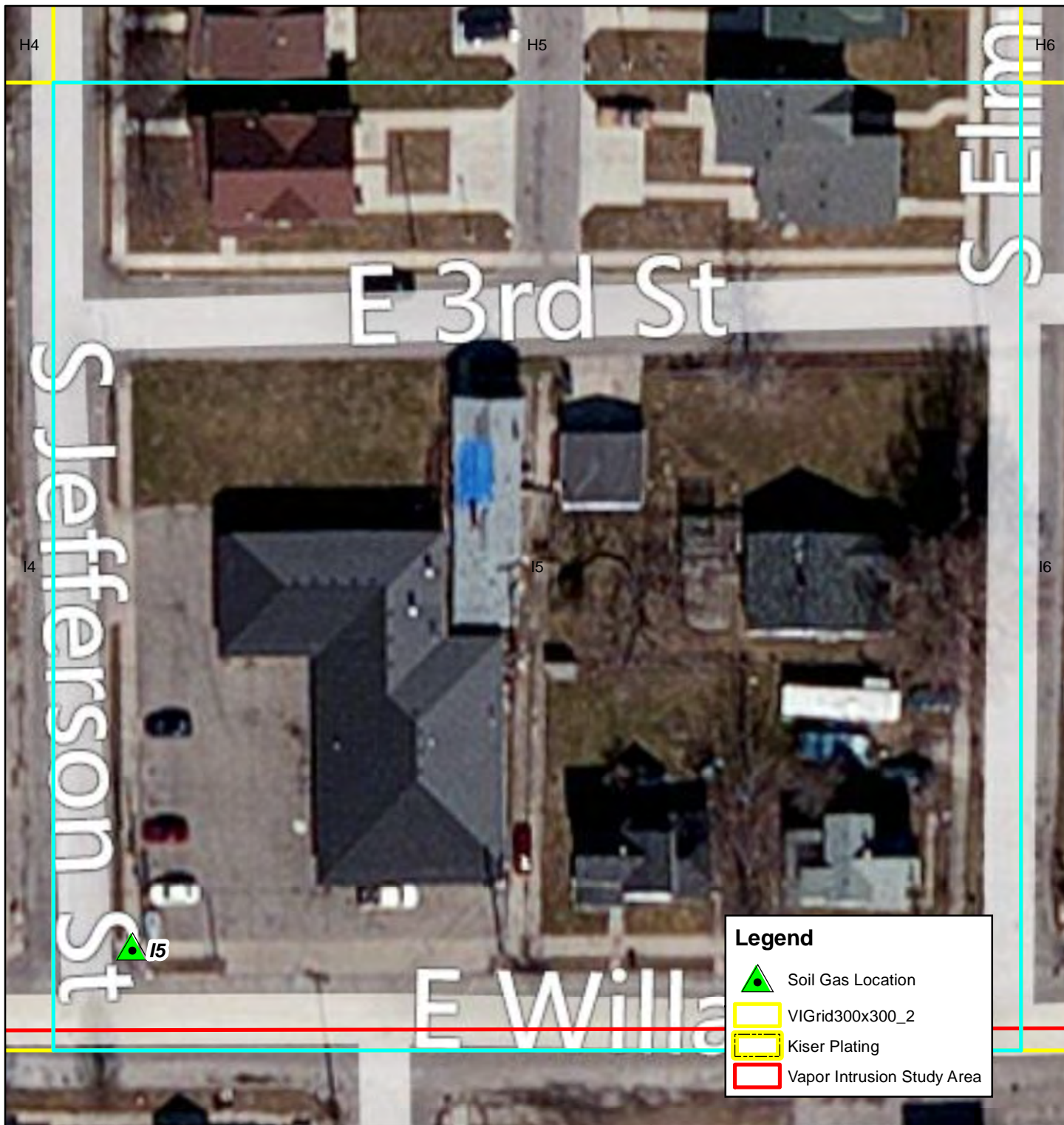
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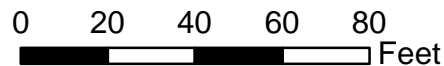


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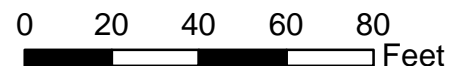


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APPENDIX B
FIELD SAMPLING SOPs

SUPERFUND TECHNICAL ASSESSMENT RESPONSE TEAM
STANDARD OPERATING PROCEDURE

SOP 103
CHAIN-OF-CUSTODY DOCUMENTATION

1.0 INTRODUCTION

The purpose of this Standard Operating Procedure (SOP) is to provide Roy F. Weston, Inc. (WESTON®), Superfund Technical Assessment Response Team (START) members with a step-by-step guide for chain-of-custody documentation.

2.0 SCOPE

This SOP describes the minimum requirements for sample chain-of-custody procedures. These procedures permit traceability from the time of sample collection to generation of the analytical data report.

These procedures are intended to document sample possession from the time of sample collection to sample disposal (i.e., sample shipment, sample storage, sample analysis).

3.0 GENERAL PROTOCOL

A chain-of-custody record will be maintained from the time of sample collection until final disposition. There are presently three chain-of-custody forms used by EPA Region III. These forms are the standard EPA Region III Chain-of-custody Record, the Organic Traffic Report/Chain-of-custody Record (EPA Form No. 9110-1, Revision 5-91) and the Inorganic Traffic Report/Chain-of-custody Record (EPA Form No. 9110-2, Revision 3-93).

The traffic report chain-of-custody forms are to be used only for EPA Contract Laboratory Program (CLP) sampling events. The EPA Region III chain-of-custody forms should be used for all START subcontracted laboratories, the EPA Region III laboratory and all Delivery of Analytical Services (DAS) sampling events.

For the case of the chain-of-custody forms and the traffic report chain-of-custody forms, every transfer of custody will be noted and signed. The distribution of the chain-of-custody forms will be done in accordance with the distribution list at the bottom of each form. The chain-of-custody record shall contain, at a minimum, the following information:

X CLP case/DAS number or START Analytical Project Number

- X Project code
- X Project identification number
- X Sample number
 - X Sample type and description
 - X Sample location
 - X Time and date of sample collection (military time)
 - X Requested analyses
 - X Sample information (e.g., no. of bottles, preservatives, etc.)
 - X Names and signatures of samplers
 - X Signatures of individuals who have had sample custody
 - X The name of the carrier and the airbill number, if the sample is shipped
 - X EPA Sample Tag Numbers

4.0

PROTOCOL FOR NON-CLP SHIPMENTS

All non-CLP sampling events will use the EPA Region III chain-of-custody record (see Attachment 1). All pertinent data must be recorded legibly in black or blue ink. Mistakes can only be corrected by drawing a single line through the mistake and then initialing and dating the correction.

Listed below are the information needed in each section of the chain-of-custody record.

- X The PROJ. NO. section of the form will always contain the site Project Control System (PCS) number.
- X The PROJECT NAME section of the form will contain the Site name only if the samples are being analyzed by the EPA Region III Laboratory, otherwise it will contain the START Analytical Technical Directive Document (TDD) number or the DAS project number.
- X The SAMPLERS (*Signature*) section should contain the printed name and signature of samplers.
- X The STA. NO. section should contain the sample number assigned by the samplers.
- X The TIME section should be in military time.
- X The STATION LOCATION section should give a description of the sample location (note: never put resident's names or full addresses in this section).
- X The diagonal lines on the top of the form should contain the analyses to be performed for each sample. An X should be placed in the block beneath the

analyses to be performed for each sample.

- X The REMARKS section should contain information such as if a sample is to be used for laboratory QC, sample preservatives, sample tag numbers, and in the case of a DAS sampling event the DAS sample number.
- X The airbill number and shipper should be identified in the REMARKS section at the bottom of the form.

Every time the samples are relinquished to a different individual the current sample custodian is required to relinquish the custody to the new individual. Attachments 2, 3, and 4 contain examples of a sampling event going to the Region III Laboratory, a START subcontracted laboratory and a DAS laboratory, respectively.

5.0 PROTOCOL FOR CLP SHIPMENTS

The EPA Organic Traffic Report/Chain-of-custody Record (EPA Form No. 9110-1, Revision 5-91) and the Inorganic Traffic Report/Chain-of-custody Record (Form No. 9110-2, Revision 3-93) are to be used for CLP sampling events. All pertinent data must be recorded legibly in black or blue ink. Mistakes can only be corrected by drawing a single line through the mistake and then initialing and dating the correction.

Listed below are the information needed in each section of the traffic report chain-of-custody record/.

- X The SAS No. section is the CLP case number.
- X Section 1, Project Code, is the site PCS number.
- X The Account Code is the Superfund account code (if known).
- X The Regional Information and NON-Superfund Program sections are to be filled in if information is available.
- X Fill in the Site Name, City and State sections.
- X If known, fill in the Site Spill ID.
- X The Region number section will be “3”.
- X The Sampling Co. section will be Weston.
- X The Sampler section will contain the primary sampler’s name printed and the Signature section will contain their signature.

- X Select the most applicable purpose in the Purpose Section. START sampling events do not always fit these choices.
- X Section 4 should be self explanatory.
- X Section 5 should contain the CLP lab address and contact.
- X The CLP Sample Number section should contain the CLP sample number assigned to your sample. CLP inorganic numbers begin with M and contain a combination of 5-6 numbers/letters. CLP organic sample numbers begin with C and contain a combination of 5-6 numbers/letters.
- X The Matrix section should contain a number chosen from Section 6 (please note that field blanks, trip blanks and rinsate blanks should be “4”).
- X Section D, Preservatives, should be chosen from Section 7 (note “ice only” should be chosen if no chemical preservative is added to the sample).
- X Section F should contain the EPA tag number; if more than one tag is used (one tag per bottle) for the sample, then use consecutive tag numbers and record the entire first tag number, hyphen, and the last three digits of the final tag number.
- X Section I will contain the corresponding inorganic or organic sample number if applicable (if none put a dash).
- X Section K will contain field QC identifiers (note: use only “B” for blanks and the corresponding CLP sample number for field duplicates). Laboratory QC samples are not to be noted in this section.
- X Put the CLP sample number of the sample that you collected extra volume for laboratory QC in the section labeled “Samples to be Used for Laboratory QC” on the bottom of the form.
- X Please note in the Shipment for Case Complete section if the shipment is complete and note the page number if multiple traffic reports are needed for your sampling event.
- X Please complete the Additional Samplers Signature section if there was more than one sampler for your event.
- X The current EPA chain-of-custody seals in this region do not have a number so this section can be left empty.

- X As in the case of the regular chain-of-custody record, every time the samples are relinquished to a different individual the current sample custodian is required to relinquish the custody to the new individual.

Attachments 5 and 6 contain examples of a properly filled out Organic Traffic Report Chain-of-custody Record and a Inorganic Traffic Report Chain-of-custody Record, respectively.

6.0 SAMPLE LABELS, SAMPLE TAGS AND CUSTODY SEALS

In addition to chain-of-custody forms, sample labels, sample tags and custody seals are needed to ensure sample custody has been maintained. Sample labels and sample tags should contain the same information contained on the chain-of-custody record to ensure the proper analyses are being performed on the samples. Custody seals ensure that the samples have not been tampered with during sample shipment. Below is a description of the information needed for the labels, tags and custody seals.

6.1 Sample Labels

The following information will be recorded on the sample labels affixed to each container:

- X Project identification number
- X Sample number
- X Time and date of sample collection
- X Sample type (composite/grab)
- X Sample location
- X Analyses requested
- X Preservatives used

6.2 Sample Tags

In addition to a sample label, one sample tag, in accordance with EPA Tag (GPO): 1994-379-334, will be affixed to each sample container. The sample tags bear serial numbers. The following information, at a minimum, will be recorded on the sample tag:

- X CLP case/DAS number
- X Project code
- X Sample number
- X Time and date of sample collection
- X Sample type (composite/grab)
- X Sample location
- X Analyses requested
- X Preservatives used
- X Signatures of samplers

| | |
|---|------------------------------------|
| X | Lot number of the sample container |
| X | Remarks |

6.3 Custody Seals

Custody seals confirm that samples have not been tampered with. The individual who has custody of the samples will sign, date, and affix the seals to the cooler or shipping box which contains the samples so that it cannot be opened without breaking the seal. A wide clear tape will be placed over the seals to ensure that the seals are not accidentally broken during transportation.

7.0 REFERENCES

U.S. Environmental Protection Agency (EPA). 1998. *Sample Submission Guidelines*. Fifth edition. EPA Region III Office of Analytical Services and Quality Assurance. Annapolis, MD. February.

American Standards for Testing and Materials (ASTM). 1993. *Standard Practices for Sampling Chain-of-custody Procedures*. Designation D 4840-88 (Reapproved 1993). Philadelphia, PA. May.

Attachments: 6

ATTACHMENT 1

CHAIN-OF-CUSTODY RECORD

ATTACHMENT 2

**EXAMPLE OF A SAMPLING EVENT GOING TO THE
REGION III LABORATORY**

ATTACHMENT 3

EXAMPLE OF A SAMPLING EVENT GOING TO A START SUBCONTRACTED LABORATORY

ATTACHMENT 4

**EXAMPLE OF A SAMPLING EVENT GOING TO A
DAS LABORATORY**

ATTACHMENT 5

ORGANIC TRAFFIC REPORT & CHAIN-OF-CUSTODY RECORD

ATTACHMENT 6

INORGANIC TRAFFIC REPORT & CHAIN-OF-CUSTODY RECORD



SUMMA CANISTER SAMPLING

SOP#: 1704
DATE: 07/27/95
REV. #: 0.1

1.0 SCOPE AND APPLICATION

The purpose of this standard operating procedure (SOP) is to describe a procedure for sampling of volatile organic compounds (VOCs) in ambient air. The method is based on samples collected as whole air samples in Summa passivated stainless steel canisters. The VOCs are subsequently separated by gas chromatography (GC) and measured by mass-selective detector or multidetector techniques. This method presents procedures for sampling into canisters at final pressures both above and below atmospheric pressure (respectively referred to as pressurized and subatmospheric pressure sampling).

This method is applicable to specific VOCs that have been tested and determined to be stable when stored in pressurized and subatmospheric pressure canisters. The organic compounds that have been successfully collected in pressurized canisters by this method are listed in the Volatile Organic Compound Data Sheet (Appendix A). These compounds have been measured at the parts per billion by volume (ppbv) level.

These are standard (i.e., typically applicable) operating procedures which may be varied or changed as required, dependent on site conditions, equipment limitations or limitations imposed by the procedure or other procedure limitations. In all instances, the ultimate procedures employed should be documented and associated with the final report.

Mention of trade names or commercial products does not constitute U.S. EPA endorsement or recommendation for use.

2.0 METHOD SUMMARY

Both subatmospheric pressure and pressurized sampling modes use an initially evacuated canister. Both modes may also use a mass flow controller/vacuum pump arrangement to regulate flow. With the above configuration, a sample of ambient air

is drawn through a sampling train comprised of components that regulate the rate and duration of sampling into a pre-evacuated Summa passivated canister. Alternatively, subatmospheric pressure sampling may be performed using a fixed orifice, capillary, or adjustable micrometering valve in lieu of the mass flow controller/vacuum pump arrangement for taking grab samples or short duration time-integrated samples. Usually, the alternative types of flow controllers are appropriate only in situations where screening samples are taken to assess for future sampling activities.

3.0 SAMPLE PRESERVATION, CONTAINERS, HANDLING, AND STORAGE

After the air sample is collected, the canister valve is closed, an identification tag is attached to the canister, and the canister is transported to a laboratory for analysis. Upon receipt at the laboratory, the canister tag data is recorded. Sample holding times and expiration should be determined prior to initiating field activities.

4.0 INTERFERENCES AND POTENTIAL PROBLEMS

Contamination may occur in the sampling system if canisters are not properly cleaned before use. Additionally, all other sampling equipment (e.g., pump and flow controllers) should be thoroughly cleaned.

5.0 EQUIPMENT/APPARATUS

The following equipment/apparatus (Figure 1, Appendix B) is required:

5.1 Subatmospheric Pressure Sampling Equipment

1. VOC canister sampler - whole air sampler capable of filling an initially evacuated canister by action of the flow controlled pump from vacuum to near atmospheric pressure. (Andersen Samplers Inc., Model 87-100 or equivalent).
2. Sampling inlet line - stainless steel tubing to connect the sampler to the sample inlet.
3. Sample canister - leak-free stainless steel pressure vessels of desired volume with valve and Summa passivated interior surfaces (Scientific Instrumentation Specialist, Inc., ID 83843, Andersen Samplers, Inc., or equivalent).
4. Particulate matter filter - 2- μ m sintered stainless steel in-line filter (Nupro Co., Model SS-2F-K4-2, or equivalent).
5. Chromatographic grade stainless steel tubing and fittings - for interconnections (Alltech Associates, Cat. #8125, or equivalent). All materials in contact with sample, analyte, and support gases should be chromatographic grade stainless steel.
6. Fixed orifice, capillary, or adjustable micrometering valve - used in lieu of the electronic flow controller/vacuum pump for grab samples or short duration time-integrated samples.

5.2 Pressurized Sampling Equipment

1. VOC canister sampler - whole air sampler capable of filling an initially evacuated canister by action of the flow controlled pump from vacuum to near atmospheric pressure. (Andersen Samplers Inc., Model 87-100).
2. Sampling inlet line - stainless steel tubing to connect the sampler to the sample inlet.
3. Sample canister - leak-free stainless steel pressure vessels of desired volume with valve and Summa passivated interior

surfaces (Scientific Instrumentation Specialist, Inc., ID 83843, Andersen Samplers, Inc., or equivalent).

4. Particulate matter filter - 2- μ m sintered stainless steel in-line filter (Nupro Co., Model SS-2F-K4-2, or equivalent).
5. Chromatographic grade stainless steel tubing and fittings - for interconnections (Alltech Associates, Cat. #8125, or equivalent). All materials in contact with sample, analyte, and support gases should be chromatographic grade stainless steel.

6.0 REAGENTS

This section is not applicable to this SOP.

7.0 PROCEDURE

7.1 Subatmospheric Pressure Sampling

7.1.1 Sampling Using a Fixed Orifice, Capillary, or Adjustable Micrometering Valve

1. Prior to sample collection, the appropriate information is completed on the Canister Sampling Field Data Sheet (Appendix C).
2. A canister, which is evacuated to 0.05 mm Hg and fitted with a flow restricting device, is opened to the atmosphere containing the VOCs to be sampled.
3. The pressure differential causes the sample to flow into the canister.
4. This technique may be used to collect grab samples (duration of 10 to 30 seconds) or time-integrated samples (duration of 12 to 24 hours). The sampling duration depends on the degree to which the flow is restricted.
5. A critical orifice flow restrictor will have a decrease in the flow rate as the pressure approaches atmospheric.
6. Upon sample completion at the location, the appropriate information is recorded on the

Canister Sampling Field Data Sheet.

7.1.2 Sampling Using a Mass Flow Controller/Vacuum Pump Arrangement (Andersen Sampler Model 87-100)

1. Prior to sample collection the appropriate information is completed on the Canister Sampling Field Data Sheet (Appendix C).
2. A canister, which is evacuated to 0.05 mm Hg and connected in line with the sampler, is opened to the atmosphere containing the VOCs to be sampled.
3. A whole air sample is drawn into the system through a stainless steel inlet tube by a direct drive blower motor assembly.
4. A small portion of this whole air sample is pulled from the inlet tube by a specially modified inert vacuum pump in conjunction with a mass flow controller.
5. The initially evacuated canister is filled by action of the flow controlled pump to near atmospheric pressure.
6. A digital time-program is used to pre-select sample duration and start and stop times.
7. Upon sample completion at the location, the appropriate information is recorded on the Canister Sampling Field Data Sheet.

7.2 Pressurized Sampling

7.2.1 Sampling Using a Mass Flow Controller/Vacuum Pump Arrangement (Anderson Sampler Model 87-100)

1. Prior to sample commencement at the location, the appropriate information is completed on the Canister Sampling Field Data Sheet.
2. A canister, which is evacuated to 0.05 mm Hg and connected in line with the sampler, is opened to the atmosphere containing the

VOCs to be sampled.

3. A whole air sample is drawn into the system through a stainless steel inlet tube by a direct drive blower motor assembly.
4. A small portion of this whole air sample is pulled from the inlet tube by a specially modified inert vacuum pump in conjunction with a mass flow controller.
5. The initially evacuated canister is filled by action of the flow controlled pump to a positive pressure not to exceed 25 psig.
6. A digital time-programmer is used to pre-select sample duration and start and stop times.
7. Upon sample completion at the location, the appropriate information is recorded on the Canister Sampling Field Data Sheet.

8.0 CALCULATIONS

1. A flow control device is chosen to maintain a constant flow into the canister over the desired sample period. This flow rate is determined so the canister is filled to about 88.1 kPa for subatmospheric pressure sampling or to about one atmosphere above ambient pressure for pressurized sampling over the desired sample period. The flow rate can be calculated by:

$$F = \frac{(P)(V)}{(T)(60)}$$

where:

| | | |
|---|---|---|
| F | = | flow rate (cm ³ /min) |
| P | = | final canister pressure, atmospheres absolute |
| V | = | volume of the canister (cm ³) |
| T | = | sample period (hours) |

For example, if a 6-L canister is to be filled to 202 kPa (two atmospheres) absolute pressure in 24 hours, the flow rate can be calculated by:

$$F = \frac{(2)(6000)}{(24)(60)} \cdot 8.3 \text{ cm}^3/\text{min}$$

2. If the canister pressure is increased, a dilution factor (DF) is calculated and recorded on the sampling data sheet.

$$DF = \frac{Y_a}{X_a}$$

where:

X_a = canister pressure (kPa, psia) absolute before dilution.
 Y_a = canister pressure (kPa, psia) absolute after dilution.

After sample analysis, detected VOC concentrations are multiplied by the dilution factor to determine concentration in the sampled air.

9.0 QUALITY ASSURANCE/QUALITY CONTROL

The following general quality assurance procedures apply:

1. All data must be documented on standard chain of custody records, field data sheets, or site logbooks.
2. All instrumentation must be operated in accordance with operating instructions as supplied by the manufacturer, unless otherwise specified in the work plan. Equipment checkout and calibration activities must occur prior to sampling/operation, and they must be documented.

10.0 DATA VALIDATION

This section is not applicable to this SOP.

11.0 HEALTH AND SAFETY

When working with potentially hazardous materials, follow U.S. EPA, OSHA, and corporate health and safety practices. Specifically, pressurizing of Summa canisters should be performed in a well ventilated room, or preferably under a fume hood. Care must be taken not to exceed 40 psi in the canisters. Canisters are under pressure, albeit only 20-30 psi, and should not be dented or punctured. They should be stored in a cool dry place and always be placed in their plastic shipping boxes during transport and storage.

12.0 REFERENCES

1. Ralph M. Riggan, Technical Assistance Document for Sampling and Analysis of Toxic Organic Compounds in Ambient Air, EPA-600/4-83-027 U. S. Environmental Protection Agency, Research Triangle Park, NC, 1983.
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APPENDIX A

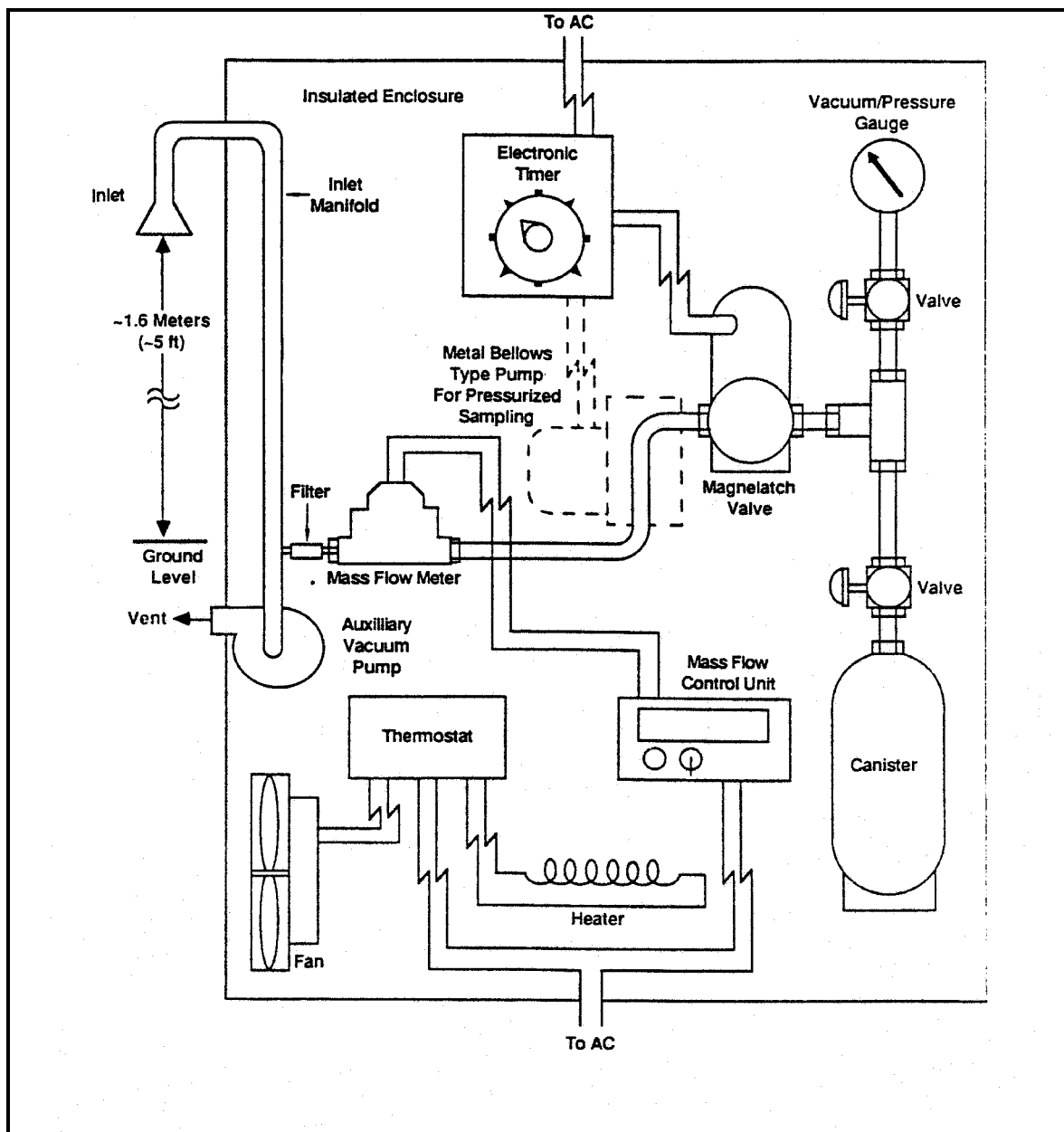
Volatile Organic Compound Data Sheet

TABLE 1. VOLATILE ORGANIC COMPOUND DATA SHEET

| COMPOUND (SYNONYM) | FORMULA | MOLECULAR WEIGHT | BOILING POINT (°C) | MELTING POINT (°C) | CAS NUMBER |
|--|---|------------------|--------------------|--------------------|------------|
| Freon 12 (Dichlorodifluoromethane) | Cl ₂ CF ₂ | 120.91 | -29.8 | -158.0 | 74-87-3 |
| Methyl chloride (Chloromethane) | CH ₃ Cl | 50.49 | -24.2 | -97.1 | |
| Freon 114 (1,2-Dichloro-1,1,2,2-tetrafluoroethane) | ClCF ₂ CClF ₂ | 170.93 | 4.1 | -94.0 | |
| Vinyl chloride (Chloroethylene) | CH ₂ =CHCl | 62.50 | -13.4 | -1538.0 | 75-01-4 |
| Methyl bromide (Bromomethane) | CH ₃ Br | 94.94 | 3.6 | -93.6 | 74-83-9 |
| Ethyl chloride (Chloroethane) | CH ₃ CH ₂ Cl | 64.52 | 12.3 | -136.4 | 75-00-3 |
| Freon 11 (Trichlorofluoromethane) | CCl ₃ F | 137.38 | 23.7 | -111.0 | 75-35-4 |
| Vinylidene chloride (1,1-Dichloroethene) | C ₂ H ₂ Cl ₂ | 96.95 | 31.7 | -122.5 | |
| Dichloromethane (Methylene chloride) | CH ₂ Cl ₂ | 84.94 | 39.8 | -95.1 | |
| Freon 113 (1,1,2-Trichloro-1,2,2-trifluoroethane) | CF ₂ ClCCl ₂ F | 187.38 | 47.7 | -36.4 | 75-09-2 |
| 1,1-Dichloroethane (Ethylidene chloride) | CH ₃ CHCl ₂ | 98.96 | 57.3 | -97.0 | 74-34-3 |
| cis-1,2-Dichloroethylene | CHCl=CHCl | 96.94 | 60.3 | -80.5 | |
| Chloroform (Trichloromethane) | CHCl ₃ | 119.38 | 61.7 | -63.5 | |
| 1,2-Dichloroethane (Ethylene dichloride) | ClCH ₂ CH ₂ Cl | 98.96 | 83.5 | -35.3 | 67-66-3 |
| Methyl chloroform (1,1,1-Trichloroethane) | CH ₃ CCl ₃ | 133.41 | 74.1 | -30.4 | 107-06-2 |
| Benzene (Cyclohexatriene) | C ₆ H ₆ | 78.12 | 80.1 | 5.5 | 71-55-6 |
| Carbon tetrachloride (Tetrachloromethane) | CCl ₄ | 153.82 | 76.5 | -23.0 | 71-43-2 |
| 1,2-Dichloropropane (Propylene dichloride) | CH ₃ CHClCH ₂ Cl | 112.99 | 96.4 | -100.4 | 56-23-5 |
| Trichloroethylene (Trichloroethene) | ClCH=CCl ₂ | 131.29 | 87 | -73.0 | 78-87-5 |
| cis-1,3-Dichloropropene (cis-1,3-dichloropropylene) | CH ₃ CCl=CHCl | 110.97 | 76 | | 79-01-6 |
| trans-1,3-Dichloropropene (cis-1,3-Dichloropropylene) | ClCH ₂ CH=CHCl | 110.97 | 112.0 | | |
| 1,1,2-Trichloroethane (Vinyl trichloride) | CH ₂ ClCHCl ₂ | 133.41 | 113.8 | -36.5 | 79-00-5 |
| Toluene (Methyl benzene) | C ₆ H ₅ CH ₃ | 92.15 | 110.6 | -95.0 | 108-88-3 |
| 1,2-Dibromoethane (Ethylene dibromide) | BrCH ₂ CH ₂ Br | 187.88 | 131.3 | 9.8 | 106-93-4 |
| Tetrachloroethylene (Perchloroethylene) | Cl ₂ C=CCl ₂ | 165.83 | 121.1 | -19.0 | 127-18-4 |
| Chlorobenzene (Phenyl chloride) | C ₆ H ₅ Cl | 112.56 | 132.0 | -45.6 | 108-90-7 |
| Ethylbenzene | C ₆ H ₅ C ₂ H ₅ | 106.17 | 136.2 | -96.0 | 100-41-4 |
| m-Xylene (1,3-Dimethylbenzene) | 1,3-(CH ₃) ₂ C ₆ H ₄ | 106.17 | 139.1 | -47.9 | |
| p-Xylene (1,4-Dimethylxylene) | 1,4-(CH ₃) ₂ C ₆ H ₄ | 106.17 | 138.3 | 13.3 | |
| Styrene (Vinyl benzene) | C ₆ H ₅ CH=CH ₂ | 104.16 | 145.2 | -30.6 | 100-42-5 |
| 1,1,2,2-Tetrachloroethane | CHCl ₂ CHCl ₂ | 167.85 | 146.2 | -36.0 | 79-34-5 |
| o-Xylene (1,2-Dimethylbenzene) | 1,2-(CH ₃) ₂ C ₆ H ₄ | 106.17 | 144.4 | -25.2 | |
| 1,3,5-Trimethylbenzene (Mesitylene) | 1,3,5-(CH ₃) ₃ C ₆ H ₃ | 120.20 | 164.7 | -44.7 | 108-67-8 |
| 1,2,4-Trimethylbenzene (Pseudocumene) | 1,2,4-(CH ₃) ₃ C ₆ H ₃ | 120.20 | 169.3 | -43.8 | 95-63-6 |
| m-Dichlorobenzene (1,3-Dichlorobenzene) | 1,3-Cl ₂ C ₆ H ₄ | 147.01 | 173.0 | -24.7 | 541-73-1 |
| Benzyl chloride (α-Chlorotoluene) | C ₆ H ₅ CH ₂ Cl | 126.59 | 179.3 | -39.0 | 100-44-7 |
| o-Dichlorobenzene (1,2-Dichlorobenzene) | 1,2-Cl ₂ C ₆ H ₄ | 147.01 | 180.5 | -17.0 | 95-50-1 |
| p-Dichlorobenzene (1,4-Dichlorobenzene) | 1,4-Cl ₂ C ₆ H ₄ | 147.01 | 174.0 | 53.1 | 106-46-7 |
| 1,2,4-Trichlorobenzene | 1,2,4-Cl ₃ C ₆ H ₃ | 181.45 | 213.5 | 17.0 | 120-82-1 |
| Hexachlorobutadiene (1,1,2,3,4,4-Hexachloro-1,3-butadiene) | | | | | |

APPENDIX B

FIGURE 1. Subatmospheric/Pressurized Sampling Equipment



APPENDIX C

Canister Sampling Field Data Sheet

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SUMMA AIR SAMPLING WORK SHEET

Site: _____
 Samplers: _____
 Date: _____

Site#: _____
 Work Assignment Manager: _____
 Project Leader: _____

| | | | | | |
|----------------------------|--------|--------|--------|--------|--------|
| Sample # | | | | | |
| Location | | | | | |
| SUMMA ID | | | | | |
| Orifice Used | | | | | |
| Analysis/Method | | | | | |
| Time (Start) | | | | | |
| Time (Stop) | | | | | |
| Total Time | | | | | |
| SUMMA WENT TO AMBIENT | YES/NO | YES/NO | YES/NO | YES/NO | YES/NO |
| Pressure Gauge | | | | | |
| Pressure Gauge | | | | | |
| Flow Rate (Pre) | | | | | |
| Flow Rate (Post) | | | | | |
| Flow Rate (Average) | | | | | |
| MET Station On-site? Y / N | | | | | |
| General Comments: | | | | | |



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 - 4.1 PID Measurements*
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SUPERCEDES: SOP #2042; Revision 0; 6/1/96; U.S. EPA Contract 68-C4-0022.



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SOIL GAS SAMPLING

1.0 SCOPE AND APPLICATION

Soil gas monitoring provides a quick means of detecting volatile organic compounds (VOCs) in the soil subsurface. Using this method, underground VOC contamination can be identified, and the source, extent, and movement of pollutants can be traced.

This standard operating procedure (SOP) outlines the methods used for the installation of soil gas wells; the collection of soil gas using Tedlar® bags, sorbent tubes, and/or Summa canisters; and measurement of organic vapor levels in the soil gas using a Photo Ionization Detector (PID), Flame Ionization Detector (FID) and/or other air monitoring devices.

These are standard (i.e., typically applicable) operating procedures which may be varied or changed as required, dependent on site conditions, equipment limitations or limitations imposed by the procedure. In all instances, the ultimate procedures employed should be documented and associated with the final report.

Mention of trade names or commercial products does not constitute United States Environmental Protection Agency (U.S. EPA) endorsement or recommendation for use.

2.0 METHOD SUMMARY

A 4-inch (") diameter hole is driven into the ground using manual (i.e., slam bar) or power driven mechanical (i.e., Geoprobe) methods. Soil gas can be sampled at specific depths by controlled penetration and/or the use of a longer bar or bar attachments. A 1/4" outer diameter (O.D.) stainless steel probe is inserted into the hole. The hole is sealed around the top of the probe using clean modeling clay. The gas contained in the interstitial spaces of the soil is pulled through the probe using an air sampling pump. The sample may be stored in Tedlar® bags, drawn through sorbent cartridges, or analyzed directly using a field portable instrument such as a PID. An air sampling pump is not used for Summa® canister sampling of soil gas; sampling is achieved by soil gas equilibration with the evacuated Summa® canister.

Power driven mechanical devices may be used to make holes when conditions make the use of manual devices unfeasible (i.e., frozen ground, very dense clays, pavement, etc.). Commercially available soil gas sampling probes (hollow, 1/2" O.D. steel probes) can be driven to the desired depth using a power hammer (e.g., demolition hammer or Geoprobe™). Soil gas samples can be drawn through the probe itself, or through Teflon tubing inserted through the probe and attached to the probe point. Samples are collected and analyzed as described below.

Other field air monitoring devices, such as the Combustible Gas Indicator (CGI) and the Organic Vapor Analyzer (OVA), can also be used, depending on specific site conditions. Measurement of soil temperature using a temperature probe may also be desirable. Bagged samples may be analyzed in a field laboratory using portable gas chromatography (GC) instrumentation, or shipped to a laboratory using an overnight service.

3.0 SAMPLE PRESERVATION, CONTAINERS, HANDLING, AND STORAGE

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3.1 Tedlar® Bags

Soil gas samples are generally collected in 1.0-liter (L) Tedlar® bags. Bagged samples should be stored in the dark (i.e., in opaque containers) and protected from mechanical damage during transit to the laboratory. Further, bagged samples should be maintained at ambient temperature by placing them in coolers and out of direct sunlight. Samples should be analyzed as soon as possible, preferably within 24 to 48 hours following sample collection. Refer to ERT/REAC SOP# 2102, *Tedlar® Bag Sampling*, for additional information.

3.2 Sorbent Tubes

Soil gas can be drawn directly onto sorbent tubes (i.e., Tenax tubes) and analyzed by Gas Chromatography/Mass Spectrometer (GC/MS) methodologies. Bagged samples can also be drawn onto tubes. If sorbent tubes are to be used, special care must be taken to avoid contamination. Refer to ERT/REAC SOP# 2104, *Tenax/CMS Tube Sampling*, for additional information. Samples should be refrigerated at 4 °C during storage and analyzed within 30 days of collection. Samples taken on multi-sorbent tubes should be analyzed as soon as possible after sampling.

3.3 Summa® Canisters

The Summa® canisters used for soil gas sampling have a 6-L sample capacity and are certified clean by GC/MS analysis before being used in the field. After sampling is completed, they are stored and shipped in travel cases. Most volatile organic compounds (VOCs) can be recovered from canisters with minimal loss up to thirty days. Refer to ERT/REAC SOP# 1704, *Summa Canister Sampling*, for additional information.

4.0 INTERFERENCES AND POTENTIAL PROBLEMS

4.1 PID Measurements

A number of factors specific to soil gas can affect the response of a PID (e.g., HNu® PI 101). High humidity can cause lamp fogging and decreased sensitivity. This can occur when soil moisture levels are high, or when a soil gas probe is in the saturated zone. High concentrations of methane can cause a downscale deflection of the meter. High and low temperature, electrical fields, FM radio transmission, and naturally occurring compounds, such as terpene hydrocarbons in wooded areas, will affect instrument response. Refer to ERT/REAC SOP# 2114, *Photoionization Detector (PID) HNu®* for additional information.

4.2 FID Measurements

A number of factors specific to soil gas can affect the response of an FID (e.g., OVA Model 128). High humidity can cause the FID to flame out or not ignite at all. This can be significant when soil moisture levels are high, or when a soil gas probe is in the saturated zone. The FID can only read organic based

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compounds (they must contain carbon in the molecular structure). The FID also responds poorly to hydrocarbons and halogenated hydrocarbons (such as gasoline, propane fuel). High and low temperature, electrical fields and FM radio transmission will also affect instrument response. Consult the instrument manual for additional information.

4.3 Factors Affecting the Concentrations of Organic Compounds in Soil Gas

Concentrations of organic compounds in soil gas can be affected by the physical and chemical characteristics of the soil and by soil moisture. Organic molecules can be tightly adsorbed to the surface of chemically active soil particles, such as clays, thus reducing the concentration in the soil interstitial spaces. Similarly, some organic compounds can be dissolved in the soil water or associated with soil organic components (i.e., humic acids).

Soil porosity and permeability will affect the movement of soil gas and the recharge rate of the soil gas well. The movement of organic vapors through fine textured soil may be very slow, thus limiting the sample volume available and the use of this technique. Existing information and soil surveys prepared by the Soil Conservation Service should be consulted prior to planning and designing a soil gas survey.

The presence of a high, or perched water table, or of an impermeable underlying layer (such as a clay lens or layer of buried slag) may interfere with the movement and sampling of the soil gas. Knowledge of site geology is useful in such situations, and can prevent inaccurate sampling.

4.4 Soil Probe Clogging

A common problem with the soil gas sampling is clogging of the probe. A clogged probe can be identified by using an in-line vacuum gauge or by listening for the sound of the pump laboring. This problem can usually be eliminated by using a wire cable to clear the probe (see Section 7.1.3.).

4.5 Underground Utilities

Prior to selecting sample locations, an underground utility search must be completed. The local utility companies can be contacted and requested to mark the locations of their underground lines. Each sample location should also be screened with a metal detector or magnetometer to verify that no underground metallic or ferro-magnetic pipes or drums are present.

5.0 EQUIPMENT/APPARATUS

5.1 Slam Bar Method

- C Slam bar
- C Soil gas probes: stainless steel tubing, 1/4" O.D., 5-foot (ft) length
- C Flexible wire or cable

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- C "Quick Connect" fittings
- C Modeling clay.
- C Vacuum box
- C Pumps, capable of drawing approximately 3.0 L/min
- C ¼" Teflon® tubing, 2-ft to 3-ft lengths
- C ¼" Tygon tubing
- C Tedlar® bags, 1.0-L
- C Sample documentation (soil gas sample labels, field data sheets, logbook, etc.)
- C PID/FID, or other field air monitoring devices
- C Cooler(s)
- C Metal detector or magnetometer
- C Portable GC instrument
- C Summa® canisters (plus shipping cases)
- C Large dark plastic bags

5.2 Power Hammer Method

- C Power (Demolition) hammer
- C ½" O.D. steel probes, extensions, and points
- C Dedicated aluminum sampling points
- C ¼" Teflon® tubing, 2-ft to 3-ft lengths
- C "Quick Connect" fittings
- C Modeling clay.
- C Vacuum box
- C Pumps, capable of drawing approximately 3.0 L/min
- C ¼" Tygon tubing
- C Tedlar® bags, 1.0-L
- C Sample documentation (soil gas sample labels, field data sheets, logbook, etc.)
- C PID/FID or other field air monitoring devices
- C Cooler(s)
- C Metal detector or magnetometer
- C Portable GC instrument
- C Summa® canisters (plus shipping cases)
- C Generator w/extension cords.
- C High lift jack assembly
- Large dark plastic bags

5.3 Direct-Push (Geoprobe™) Method

- Tubing; polyethylene, Teflon®, or stainless steel
- Gas sampling cap
- Probe rods
- Tubing adaptor(s)

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- Expendable point holder, threaded
- Expendable drive point(s)
- O-rings for expendable point holder
- O-rings for adaptor
- O-rings for probe rods
- O-rings for gas sampling cap
- Vacuum pumps
- Tape
- Tedlar® bags, 1.0-L
- Summa® canisters (plus shipping cases)
- Sample documentation (soil gas labels, field data sheets, logbook, etc.)
- Metal detector or magnetometer
- Cooler(s)
- Large dark plastic bags
- Portable GC instrument

6.0 REAGENTS

- C Calibration and spike gases
- C Deionized, organic-free water
- C Methanol, High Performance Liquid Chromatography (HPLC) grade
- C Ultra-zero grade compressed air
- C Propane torch

7.0 PROCEDURES

7.1 Soil Gas Probe Installation

7.1.1 Slam Bar Method

1. A hole slightly deeper than the desired sampling depth is made. For sampling up to 5 feet, a 5-ft single piston slam bar is used. For deeper depths, a piston slam bar with threaded 4-ft-long extensions is used.
2. The tip of the rod is placed on the ground and the piston of the slam bar is used to drive the rod to the desired depth. The number of blows required to reach the desired depth is recorded.
3. After the hole is made, the slam bar is carefully withdrawn to prevent the collapse of the walls.
4. The soil gas probe is carefully inserted into the hole. To prevent plugging of the probe, a decontaminated metal wire or cable, slightly longer than the probe and with

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an O.D. slightly less than the inner diameter (I.D.) of the rod, is inserted in the probe rod; 1- to 2-inches of wire should protrude from the end of the probe. The probe is inserted to full depth of the hole, then pulled up three to six inches. The probe is cleared by moving the cable up and down several times.

5. The top of the sample hole is sealed at the surface to prevent infiltration of ambient air. A golf-ball size lump of clean modeling clay is kneaded until it becomes soft. The clay is carefully molded around the probe at the soil surface to seal the space between the probe and the hole.
6. If semi-permanent soil gas installations are required, the probe remains in the hole, which may be sealed by backfilling with clean sand, soil, or bentonite.

7.1.2 Power Hammer Method

1. A power hammer may be used to make holes when the soil is very hard, frozen or fine textured (clay), or when soil gas from beneath pavement or concrete is collected.
2. A power hammer is used to drive the probe to the desired depth (up to 12 feet may be attained with extensions). Threaded extensions are added until the desired depth is needed.
3. After the hole is made, the threaded rod is carefully withdrawn. This should be done in such a manner to prevent collapse of the walls. If necessary, a jack assembly may be used to retrieve the rods.
4. The soil gas probe is installed in the hole as described in Section 7.1.1, Steps 4 and 5.
5. If semi-permanent soil gas installations are required, the probe remains in the hole, which may be sealed by backfilling with clean sand, soil, or bentonite.

7.1.3 Direct-Push Method

1. Direct-push sampling technology refers to soil gas samplers that are inserted into the ground without the use of slam bars, demolition hammers, or drilling rigs. The U.S. EPA/ERT utilizes a Direct-Push unit mounted on an all-terrain track mounted vehicle, and direct push tools. These tools are able to collect samples at depths greater than 50 feet, depending on soil conditions.
2. Sampling probes, consisting of 3-foot sections of flush-threaded, 1¼-inch hardened steel alloy steel rod tipped by an expendable steel point, are driven into the ground

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to the target depth. The probe tools are withdrawn to release the expendable tip and allow soil gas to flow into the tool's tubing.

3. To ensure a representative soil gas sample, a discrete volume of gas is purged to rid the tubing of atmospheric air and allow the subsurface soil gas to enter the probe tubing. The volume of gas removed is determined by the volume of tubing employed in the probe. (Unlike groundwater sampling, purging of a soil gas probe is designed to remove only the ambient air within the tubing.)
4. After allowing the system to return to atmospheric pressure, an aliquot of soil gas is withdrawn from the probe. Duplicate samples are collected as necessary and required.
5. If semi-permanent soil gas installations are required, the probe remains in the hole, which may be sealed by backfilling with clean sand, soil, or bentonite.

7.2 Screening with Field Instruments

1. It is recommended that any appropriate SOPs and the manufacturers' manuals be consulted for the correct use and calibration of all instrumentation. Pumps should be calibrated prior to use in the field.
2. An amount of air, equivalent to the volume of the soil gas well must be calculated prior to sampling. Connect a vacuum pump to the sample probe using a section of Teflon® tubing. The pump is turned on and adjusted to a flow rate of 3.0 L/minute. The calculated volume of air is evacuated from the hole by pulling a vacuum through the probe for the specified length of time. Longer time is required for sample wells of greater depths.
3. After evacuation, a monitoring instrument (i.e. HNu® or OVA) is connected to the probe using a Teflon connector. Upon stabilization, the reading is recorded on soil gas data sheets.
4. Readings may be above or below the range set on the field instruments. The range may be reset, or the response recorded as a greater than or less than figure. The recharge rate of the well with soil gas must be considered when resampling at a different range setting.

7.3 Tedlar® Bag Sampling

1. Follow step 1 of section 7.2 to evacuate well volume. If air monitoring instrument screening was performed prior to sample collection, evacuation is not necessary.
2. Use the vacuum box and sampling train (Figure 1) to collect the sample. The sampling train is designed to minimize the introduction or loss of contaminants due to adsorption and other factors. All parts used are either Teflon® or stainless steel, and a vacuum is drawn indirectly

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to avoid contamination from sample pumps.

3. Place the Tedlar® bag inside the vacuum box, attach it to the sampling port and open the valve. The sample probe is attached to the sampling port via Teflon® tubing and a "Quick Connect" fitting.
4. Draw a vacuum around the outside of the bag, using a pump connected to the vacuum box evacuation port, via Tygon tubing and a "Quick Connect" fitting. The negative pressure inside the box causes the bag to inflate, drawing the sample into the bag.
5. Break the vacuum by removing the Tygon line from the pump. Remove the bagged sample from the box and close the valve. Record the date, time, sample location ID, and the PID/FID instrument reading(s) on sample bag label and on data sheets or in logbooks.

Bags should not be labeled directly with a marker or pen (particularly those containing volatile solvents) or should adhesive labels be affixed directly to the bags. Inks and adhesive may diffuse through the bag material and contaminate the sample. Labels should be tied to the metal eyelets provided on the bags.

Chain of custody sheets must accompany all samples.

7.4 Sorbent Tube Sampling

Samples collected in Tedlar® bags may be adsorbed onto sorbent tubes for further analysis by GC/MS.

7.4.1 Additional Apparatus

- Syringe, with a luer-lock tip, capable of drawing a soil gas or air sample from a Tedlar® bag onto a sorbent tube. The syringe capacity is dependent upon the volume of sample being drawn onto the tube.
- Adapters, for fitting the sorbent tube between the Tedlar® bag and the sampling syringe. The adapter attaching the Tedlar® bag to the sorbent tube consists of a reducing union (1/4" to 1/16" O.D. - Swagelok cat. # SS-400-6-ILV or equivalent) and a length of 1/4" O.D. Teflon® tubing, which replaces the nut on the 1/16" (Tedlar® bag) side. A 1/4" I.D. Teflon® or silicone O-ring replaces the ferrules in the nut on the 1/4" (sorbent tube) side of the union.

The adapter, attaching the sampling syringe to the sorbent tube, consists of a reducing union (1/4" to 1/16" O.D. - Swagelok Cat. # SS-400-6-ILV or equivalent) and a 1/4" I.D. Teflon® or silicone O-ring, which replaces the ferrules in the nut on the 1/4" (sorbent tube) side and the needle of a luer-lock syringe inserted into the 1/16"

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side (held in place with a 1/16" ferrule). The luer-lock end of the needle can be attached to the sampling syringe. It is useful to have a luer-lock on/off valve situated between the syringe and the needle.

- Two-stage glass sampling cartridge (1/4" O.D. x 1" I.D. x 5") contained in a flame-sealed tube containing two sorbent sections retained by glass wool:
 - Teflon-capped culture tubes or stainless steel tube containers for sorbent tube storage and shipping. These containers should be conditioned by baking at 120° C for at least two hours. The culture tubes should contain a glass wool plug to prevent sorbent tube breakage during transport. Reconditioning of the containers should occur between uses or after extended periods of disuse (i.e., two weeks or more).
 - Nylon gloves or lint-free cloth. (Hewlett Packard Part # 8650-0030 or equivalent.)
- 7.4.2 Sample Collection
- Handle sorbent tubes with care, using nylon gloves (or other lint-free material) to avoid contamination.
 - Immediately before sampling, break one end of the sealed tube and remove the sorbent cartridge.
 - Connect the valve on the Tedlar® bag to the sorbent tube adapter. If using a Tenax/CMS sorbent tube, connect the sorbent tube to the sorbent tube adapter with the Tenax (white granular) side of the tube facing the Tedlar® bag. Connect the sampling syringe assembly to the carbon molecular sieve [CMS (black)] side of the sorbent tube. Fittings on the adapters should be finger-tight. Open the valve on the Tedlar® bag. Open the on/off valve of the sampling syringe. Depending on work plan stipulations, at least 10% of the soil gas samples analyzed by field screening methods must be submitted for confirmation GC/MS analysis (according to a modified TO-17 method for sorbent tubes). Each soil gas sample must be absorbed on replicate sorbent tubes. The volume adsorbed on a sorbent tube is dependent on the total concentration of the compounds measured by field screening methods as follows:

| <u>Total Concentration (ppm)</u> | <u>Sample Volume (mL)</u> |
|----------------------------------|---------------------------|
| >10 | Use Serial Dilution |
| 10 | 10-50 |
| 5 | 20-100 |
| 1 | 100-250 |

- After sampling, remove the tube from the sampling train with gloves or a clean



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cloth. DO NOT LABEL OR WRITE ON THE SORBENT TUBE.

- Place the sorbent tube in a conditioned stainless steel tube holder or culture tube. Culture tube caps should be sealed with Teflon tape.
- Each sample tube container (not tube) must be labeled with the site name, sample number, date sampled, and volume sampled. Verify that all sample containers are properly labeled.
- Chain of custody sheets must accompany all samples to the laboratory.

7.5 Summa® Canister Sampling

1. Follow Section 7.2, step 1, to evacuate well volume. If PID/FID readings were taken prior to taking a sample, evacuation is not necessary.
2. Attach a certified clean, evacuated 6-L Summa® canister via the ¼" Teflon tubing.
3. Open valve on Summa® canister. The soil gas sample is drawn into the canister by pressure equilibration. The approximate sampling time for a 6-L canister is 20 minutes.
4. Sample number, sample location, date collected and work assignment number must be recorded on a chain of custody form and on a blank tag attached to the canister.
5. Chain of custody sheets must accompany all samples to the laboratory.

8.0 CALCULATIONS

8.1 Field Screening Instruments

Instrument readings are usually read directly from the meter. In some cases, the background level at the soil gas location may be subtracted:

Final Reading = Sample Reading - Background Reading

8.2 Field Portable GC Analysis

Calculations used to determine concentrations of individual components by field portable GC analysis are beyond the scope of this SOP and are covered ERT/REAC SOP #2109, *Photovac GC Analysis for Soil, Water and Air/Soil Gas*.

9.0 QUALITY ASSURANCE/QUALITY CONTROL

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9.1 Sample Sorbent Tubes

Before field use, a quality assurance (QA) check must be performed on each batch of sorbent tubes by thermal desorption/cryogenic trapping GC/MS. These tubes are prepared and cleaned in accordance with EPA Method EMSL/RTP-SOP-EMD-013 by the vendor. Prior to purchasing a lot of tubes from a vendor, ten tubes from the lot are sent to the REAC laboratory where the tubes are tested for cleanliness, precision and reproducibility.

Sample tubes should be stored out of ultraviolet (UV) light (i.e., sunlight) and kept on ice until analysis. Samples should be collected in duplicate, whenever possible.

9.2 Sample Probe Contamination

Sample probe contamination is checked between each sample by drawing ambient air through the probe using a vacuum pump (e.g., Gilian pump) and checking the response of the FID/PID. If readings are higher than background, replacement or decontamination is necessary.

Sample probes may be decontaminated simply by drawing ambient air through the probe until the HNu® reading is at background. Contamination can also be removed by decontaminating with methanol and deionized water, then air drying. For persistent contamination, use of a portable propane torch may be needed. Using a pair of pliers to hold the probe, run the torch up and down the length of the sample probe for approximately 1-2 minutes. Let the probe cool before handling. When using this method, make sure to wear gloves to prevent burns. Having more than one probe per sample team will reduce lag times between sample stations while probes are decontaminated.

9.3 Sample Train Contamination

The Teflon® line forming the sample train from the probe to the Tedlar® bag should be changed on a daily basis. If visible contamination (soil or water) is drawn into the sampling train, it must be changed immediately. When sampling in highly contaminated areas, the sampling train should be purged with ambient air, via a vacuum pump (e.g., Gilian pump), for approximately 30 seconds between each sample. After purging, the sampling train can be checked using an FID or PID, or other field monitoring device, to establish the cleanliness of the Teflon® line.

9.4 FID/PID Calibration

The FID and PID must be calibrated at least once a day using the appropriate calibration gases.

9.5 Trip Blanks

A trip blank detects any sample contamination during shipping and storage. With the exception of Summa® canisters, the trip blank is prepared and added to the site samples after sampling has been completed and prior to shipment.

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9.5.1 Tedlar® Bags

Each cooler containing Tedlar® bag samples must contain one Tedlar® bag of ultra-zero grade air, acting as a trip blank, when samples are shipped to an outside laboratory. A chain of custody record must accompany each cooler of samples and should include the blank that is dedicated to that group of samples.

9.5.2 Sorbent Tubes

At least one trip blank per cooler must be submitted with the sorbent tube samples. The ends of the sorbent tube are broken but no air is drawn through the tube.

9.5.3 Summa® Canisters

Canister trip blanks are evacuated containers that are shipped to and from the site with the canisters used for air sampling.

9.6 Field Blanks

A field blank detects sample contamination during the handling and shipping process. The field blank must be associated with an actual sampling event.

9.6.1 Tedlar® Bags

For each day of sampling, a Tedlar® bag is filled with ultra-zero air at the beginning of the day. The field blank is handled in the same manner as the samples.

9.6.2 Sorbent Tubes

For each day of sampling, a field blank must be submitted for sorbent tubes. The ends of the sorbent tube are broken at the beginning of the day but no air is drawn through the tube.

9.7 Trip Standards

If Tedlar® bags are used for sampling, each cooler containing samples should contain a Tedlar® bag of standard gas to calibrate the analytical instruments (Photovac GC, etc.). This trip standard will be used to determine any changes in concentrations of the target compounds during the course of the sampling day (e.g., migration through the sample bag, degradation, or adsorption). A fresh trip standard must be provided and placed in each cooler pending additional sample collection. A chain of custody record must accompany each cooler of samples and should include the trip standard that is dedicated to that group of samples.

9.8 Lot Blanks

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9.8.1 Tedlar® Bags

Prior to use, one bag is removed from each lot of Tedlar® bags to be used for sampling and checked for possible contamination as follows: Fill the test bag with ultra-zero grade air; withdraw a sample from the bag and analyze using a field portable GC or any other applicable field instrument used for sample analysis. This procedure will ensure sample container cleanliness prior to the start of the sampling effort.

9.8.2 Summa® Canister Check

From each lot of four cleaned Summa® canisters, one is used for a GC/MS certification check. If the canister passes certification, it is re-evacuated and all four canisters from that lot are available for sampling. If the chosen canister is contaminated, the entire lot of four Summa® canisters must be re-cleaned, and a single canister is re-analyzed by GC/MS for certification.

9.8.3 Sorbent Tubes

Provide a minimum of one sorbent tube per sampling event. Do not break the ends of the tube.

9.9 Options

9.9.1 Duplicate Samples

A minimum of 5% of all samples should be collected in duplicate (i.e., if a total of 100 samples are to be collected, five samples should be collected in duplicate). In choosing which samples to duplicate, the following criteria applies: if, after filling the first Tedlar® bag and evacuating the well for 15 seconds, the second HNu® reading (or other field monitoring device being used) matches or is close to (within 20%) the first reading, a duplicate sample may be taken.

9.9.2 Spikes

A Tedlar® bag spike and sorbent tube spike may be desirable in situations where high concentrations of contaminants other than the target compounds are found to exist (landfills, etc.). The additional level of QA/QC attained by this practice can be useful in determining the effects of interferences caused by these non-target compounds. Summa canisters containing samples are not spiked.

10.0 DATA VALIDATION

10.1 Blanks



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For each target compound, the concentration found in the sample must be greater than three times the level (for that compound) found in the appropriate blank (lot, field, trip) that accompanied that sample, to be considered valid.

11.0 HEALTH AND SAFETY

Because the sample is being drawn from underground, and no contamination is introduced into the breathing zone, soil gas sampling usually occurs in Level D. Nevertheless, ambient air should be constantly monitored using the HNu® P101 to obtain background and breathing zone readings during the sampling procedure. As long as the levels in ambient air do not rise above background, no upgrade of the level of protection is needed.

When conducting soil gas sampling, appropriate personal protective equipment [PPE (leather gloves, steel-toed shoes, Tyvek® safety suit)] should be worn, and proper slam bar techniques should be implemented. Also, an underground utility search must be performed prior to sampling (See Section 4.5).

12.0 REFERENCES

Gilian Instrument Corp. 1983. *Instruction Manual for Hi Flow Sampler: HFS113, HFS 113 T, HFS 113U, HFS 113 UT.*

HNu® Systems, Inc. 1975. *Instruction Manual for Model PI 101 Photoionization Analyzer.*

New Jersey Department of Environmental Protection. 1992. *Field Sampling Procedures Manual.*

U.S. Environmental Protection Agency. 1984. *Characterization of Hazardous Waste Sites - A Methods Manual: Volume II. Available Sampling Methods.* 2nd ed. EPA-600/4-84-076.

U.S. Environmental Protection Agency. 1995. *Superfund Program Representative Sampling Guidance. Volume 2: Air (Short-Term Monitoring).* EPA 540-R-95/140. Interim Final.

13.0 APPENDICES

- A - Figures
- B - HNu® Field Procedure



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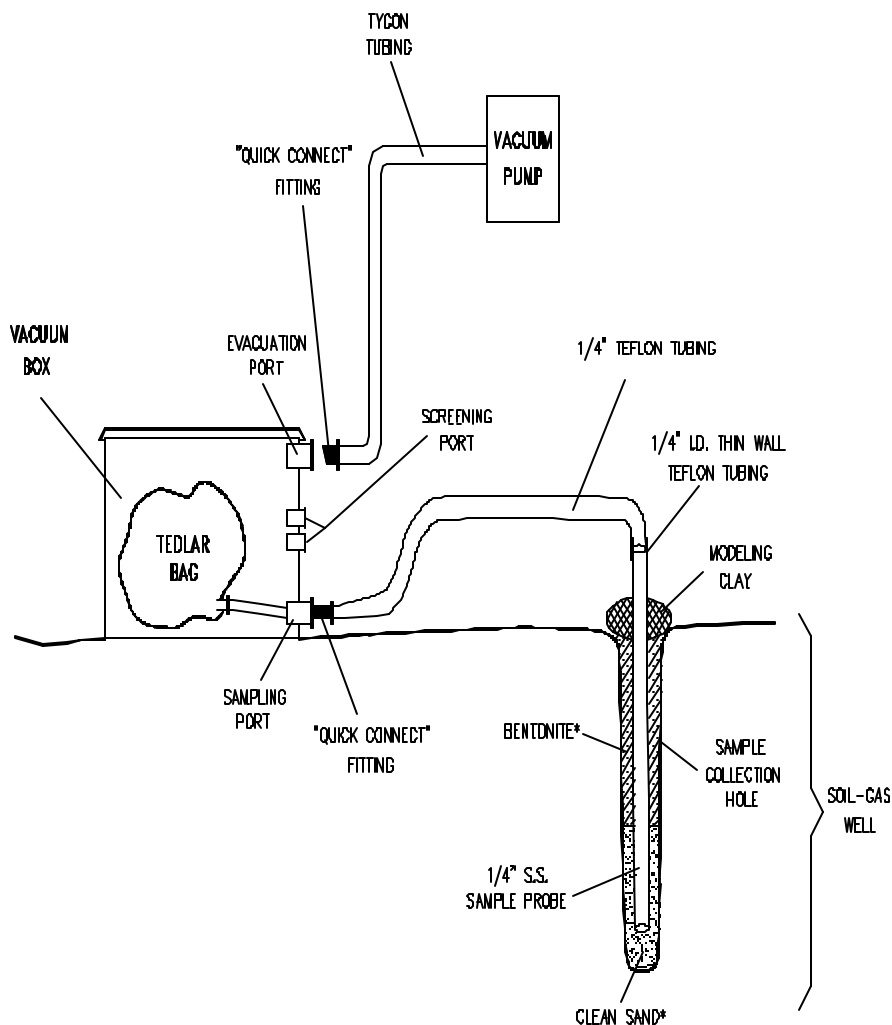
APPENDIX A
Figure
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FIGURE 1. Sampling Train Schematic



* USED ONLY FOR SEMIPERMANENT INSTALLATIONS



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APPENDIX B
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HNu® Field Procedure

The following sections detail the procedures that are to be followed when using the HNu® in the field.

Startup Procedure

- a. Before attaching the probe, check the function switch on the control panel to ensure that it is in the off position. Attach the probe by plugging it into the interface on the top of the readout module. Use care in aligning the prongs in the probe cord with the plug in; don't force the probe cord.
- b. Turn the function switch to the battery check position. The needle on the meter should read within or above the green battery area on the scale. If not, recharge the battery. If the red indicator light comes on, the battery needs recharging.
- c. Turn the function switch to any range setting. Look into the end of the probe for no more than two to three seconds to see if the lamp is on. If it is on, it will give a purple glow. Do not stare into the probe any longer than three seconds. Long term exposure to UV light can damage eyes. Also, listen for the hum of the fan motor.
- d. To ZERO the instrument, turn the function switch to the standby position and rotate the zero adjustment until the meter reads zero. A calibration gas is not needed for this instrument. If the span adjustment setting is changed after the zero is set, the zero should be rechecked and adjusted, if necessary. Wait 15 to 20 seconds to ensure that the zero reading is stable. If necessary, readjust the instrument to zero.

Operational Check

- a. Follow the start-up procedure.
- b. With the instrument set on the 0-20 ppm range, hold a solvent-based magic marker near the probe tip. If the meter deflects upscale, the instrument is working.

Field Calibration Procedure

- a. Follow the start-up procedure and the operational check.
- b. Set the function switch to the range setting for the concentration of the calibration gas.
- c. Attach a regulator to a disposable cylinder of isobutylene gas. Connect the regulator to the probe of the HNu® with a piece of clean Tygon tubing. Turn on the regulator valve.
- d. After fifteen seconds, adjust the span dial until the meter reading equals the concentration of the calibration gas used. Be careful to unlock the span dial before adjusting it. If the span has to be set below 3.0, calibrate the instrument internally or return to equipment maintenance for repair.



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- e. Record in the field logbook: the instrument ID no. (EPA decal or serial number if the instrument is a rental); the initial and final span settings; the date and time; concentration and type of calibration gas used; and the name of the person who calibrated the instrument.

Operation

- a. Follow the start-up procedure, operational check, and calibration check.
- b. Set the function switch to the appropriate range. If the concentration of gases or vapors is unknown, set the function switch to the 0-20 ppm range. Adjust it if necessary.
- c. While taking care not to permit the HNu® to be exposed to excessive moisture, dirt, or contamination, monitor the work activity as specified in the site specific Health and Safety Plan.
- d. When the activity is completed or at the end of the day, carefully clean the outside of the HNu® with a damp disposable towel to remove any visible dirt. Return the HNu® to a secure area and place on charge.
- e. With the exception of the probe's inlet and exhaust, the HNu® can be wrapped in clear plastic to prevent it from becoming contaminated and to prevent water from getting inside in the event of precipitation.

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1.0 SCOPE AND APPLICATION

This standard operating procedure (SOP) outlines the procedure used for the construction and installation of permanent sub-slab soil gas wells. The wells are used to sample the gas contained in the interstitial spaces beneath the concrete floor slab of dwellings and other structures.

Soil gas monitoring provides a quick means of detecting volatile organic compounds (VOCs) in the soil subsurface. Using this method, underground VOC contamination can be identified and the source, extent and movement of pollutants can be traced.

2.0 METHOD SUMMARY

Using an electric Hammer Drill or Rotary Hammer, an inner or pilot hole is drilled into the concrete slab to a depth of approximately 2" with the $\frac{3}{8}$ " diameter drill bit. Using the pilot hole as the center, an outer hole is drilled to an approximate depth of 1 $\frac{3}{8}$ " using the 1" diameter drill bit. The 1" diameter drill bit is then replaced with the $\frac{3}{8}$ " drill bit. The pilot hole is drilled through the slab and several inches into the sub-slab material. Once drilling is completed, a stainless steel probe is assembled and inserted into the pre-drilled hole. The probe is mounted flush with the surrounding slab so it will not interfere with pedestrian or vehicular traffic and cemented into place. A length of Teflon[®] tubing is attached to the probe assembly and to a sample container or system.

3.0 SAMPLE PRESERVATION, CONTAINERS, HANDLING AND STORAGE

3.1 SUMMA[®] Canister Sampling

After the sub-slab soil gas sample is collected, the canister valve is closed, an identification tag is attached to the canister and the canister is transported to a laboratory under chain of custody for analysis. Upon receipt at the laboratory, the data documented on the canister tag is recorded. Sample holding times are compound dependent, but most VOCs can be recovered from the canister under normal conditions near the original concentration for up to 30 days. Refer to REAC SOP #1704, *SUMMA Canister Sampling* for more details.

3.2 Tedlar[®] Bag Sampling

Tedlar[®] bags most commonly used for sampling have a 1-liter volume capacity. After sampling, the Tedlar[®] bags are stored in either a clean cooler or an opaque plastic bag at ambient temperature to prevent photodegradation. It is essential that sample analysis be undertaken within 24 to 48 hours following sample collection since VOCs may escape or become altered. Refer to REAC SOP #2102, *Tedlar[®] Bag Sampling* for more details.

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4.0 INTERFERENCES AND POTENTIAL PROBLEMS

The thickness of a concrete slab may vary from structure to structure. A structure may also have a single slab where the thickness varies. A slab may contain steel reinforcement (REBAR). Drill bits of various sizes and cutting ability will be required to penetrate slabs of varying thicknesses or those that are steel-reinforced.

5.0 EQUIPMENT/APPARATUS

- Hammer Drill or Rotary Hammer
- Alternating current (AC) extension cord
- AC generator, if AC power is not available on site
- Hammer or Rotary Hammer drill bit, 3/8" diameter
- Hammer or Rotary Hammer drill bit, 1" diameter
- Portable vacuum cleaner
- 1 - 3/4" open end wrench or 1-medium adjustable wrench
- 2 - 9/16" open end wrenches or 2-small adjustable wrenches
- Hex head wrench, 1/4"
- Tubing cutter
- Disposable cups, 5 ounce (oz)
- Disposable mixing device (i.e., popsicle stick, tongue depressor, etc.)
- Swagelok® SS-400-7-4 Female Connector, 1/4" National Pipe Thread (NPT) to 1/4" Swagelok® connector
- Swagelok® SS-400-1-4 Male Connector, 1/4"NPT to 1/4" Swagelok® connector
- 1/4" NPT flush mount hex socket plug, Teflon®-coated
- 1/4" outer diameter (OD) stainless steel tubing, pre-cleaned, instrument grade
- 1/4" OD Teflon® tubing
- Teflon® thread tape
- 1/8" OD stainless steel rod, 12" to 24" length
- Swagelok Tee, optional (SS-400-3-4TMT or SS-400-3-4TTM)

6.0 REAGENTS

- Tap water, for mixing anchoring cement
- Anchoring cement
- Modeling clay

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7.0 PROCEDURES

7.1 Probe Assembly and Installation

1. Drill a $\frac{3}{8}$ " diameter inner or pilot hole to a depth of 2" (Figure 1, Appendix A).
2. Using the $\frac{3}{8}$ " pilot hole as your center, drill a 1" diameter outer hole to a depth of $1\frac{3}{8}$ ". Vacuum out any cuttings from the hole (Figure 2, Appendix A).
3. Continue drilling the $\frac{3}{8}$ " inner or pilot hole through the slab and a few inches into the sub-slab material (Figure 3, Appendix A). Vacuum out any cuttings from the outer hole.
4. Determine the length of stainless steel tubing required to reach from the bottom of the outer hole, through the slab and into the open cavity below the slab. To avoid obstruction of the probe tube, ensure that it does not contact the sub-slab material. Using a tube cutter, cut the tubing to the desired length.
5. Attach the measured length (typically 12") of $\frac{1}{4}$ " OD stainless tubing to the female connector (SS-400-7-4) with the Swagelok® nut. Tighten the nut.
6. Insert the $\frac{1}{4}$ " hex socket plug into the female connector. Tighten the plug. **Do not over tighten.** If excessive force is required to remove the plug during the sample set up phase, the probe may break loose from the anchoring cement.
7. Place a small amount of modeling clay around the stainless steel tubing adjacent to the Swagelok® nut, which connects the stainless steel tubing to the female connector. Use a sufficient amount of modeling clay so that the completed probe, when placed in the outer hole, will create a seal between the outer hole and the inner hole. The clay seal will prevent any anchoring cement from flowing into the inner hole during the final step of probe installation.
8. Place the completed probe into the outer hole. The probe tubing should not contact the sub-slab material and the top of the female connector should be flush with the surface of the slab and centered in the outer hole (Figure 4, Appendix A). If the top of the completed probe is not flush with the surface of the slab, due to the outer hole depth being greater than $1\frac{3}{8}$ ", additional modeling clay may be placed around the stainless steel tubing adjacent to the Swagelok® nut, which connects the stainless steel tubing to the female connector. Use a sufficient amount of clay to raise the probe until it is flush with the surface of the slab while ensuring that a portion of the clay will still contact and seal the inner hole.

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9. Mix a small amount of the anchoring cement. Fill the space between the probe and the outside of the outer hole. Allow the cement to cure according to manufacturers instructions before sampling.

7.2 Sampling Set-Up

1. Wrap one layer of Teflon® thread tape onto the NPT end of the male connector (SS-400-1-4). Refer to Figure 5, Appendix A.
2. Remove the ¼" hex socket plug from the female connector (SS-400-7-4). Refer to Section 7.3 if the probe breaks loose from the anchoring cement during this step.
3. To ensure that the well has not been blocked by the collapse of the inner hole below the end of the stainless steel tubing, a stainless steel rod, ⅛" diameter, may be passed through the female connector and the stainless steel tubing. The rod should pass freely to a depth greater than the length of the stainless steel tubing, indicating an open space or loosely packed soil below the end of the stainless steel tubing. Either condition should allow a soil gas sample to be collected.

If the well appears blocked, the stainless steel rod may be used as a ramrod in an attempt to open the well. If the well cannot be opened, the probe should be reinstalled or a new probe installed in an alternate location.

4. Screw and tighten the male connector (SS-400-1-4) into the female connector (SS-400-7-4). **Do not over tighten.** This may cause the probe to break loose from the anchoring cement during this step or when the male connector is removed upon completion of the sampling event. Refer to Section 7.3 if the probe breaks loose from the anchoring cement during this step.
5. If a collocated sub-slab sample or split sample is desired, a stainless steel Swagelok Tee (SS-400-3-4TMT or SS-400-3-4TTM) may be used in place of the Swagelok male connector (SS-400-1-4).
6. Attach a length of ¼" OD Teflon® tubing to the male connector with a Swagelok® nut. The Teflon® tubing is then connected to the sampling container or system to be used for sample collection.
7. After sample collection remove the male connector from the probe and reinstall the hex socket plug. **Do not over tighten** the hex socket plug. If excessive force is required to remove the plug during the next sampling event the probe may break loose from the

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anchoring cement. Refer to Section 7.3 if the probe breaks loose from the anchoring cement during this step.

7.3 Repairing a Loose Probe

1. If the probe breaks loose from the anchoring cement while removing or installing the hex head plug or the male connector (SS-400-1-4), lift the probe slightly above the surface of the concrete slab.
2. Hold the female connector (SS-400-7-4) with the $\frac{3}{4}$ " open end wrench.
3. Complete the step being taken during which the probe broke loose, following the instructions contained in this SOP (i.e., **Do not over tighten** the hex socket plug or male connector).
4. Push the probe back down into place and reapply the anchoring cement.
5. Modeling clay may be used as a temporary patch to effect a seal around the probe until the anchoring cement can be reapplied.

8.0 CALCULATIONS

This section is not applicable to this SOP.

9.0 QUALITY ASSURANCE/QUALITY CONTROL

An additional collocated soil gas well is installed with the frequency of 10 percent (%) or as specified in the site-specific Quality Assurance Project Plan (QAPP). The following general Quality Assurance (QA) procedures apply:

1. A rough sketch of the area is drawn where the ports are installed with the major areas noted on the sketch. This information may be transferred to graphing software for incorporation into the final deliverable.
2. A global positioning system (GPS) unit may be used to document coordinates outside of a structure as a reference point.
3. Equipment used for the installation of sampling ports should be cleaned by heating, inspected and tested prior to deployment.



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10.0 DATA VALIDATION

This section is not applicable to this SOP.

11.0 HEALTH AND SAFETY

When working with potentially hazardous materials, follow Environmental Protection Agency (EPA), Occupational Safety and Health Administration (OSHA) and Lockheed Martin corporate health and safety procedures. All site activities should be documented in the site-specific health and safety plan (HASP).

12.0 REFERENCES

This section is not applicable to this SOP.

13.0 APPENDICES

A - Figures



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CONSTRUCTION AND INSTALLATION OF PERMANENT SUB-SLAB SOIL GAS WELLS

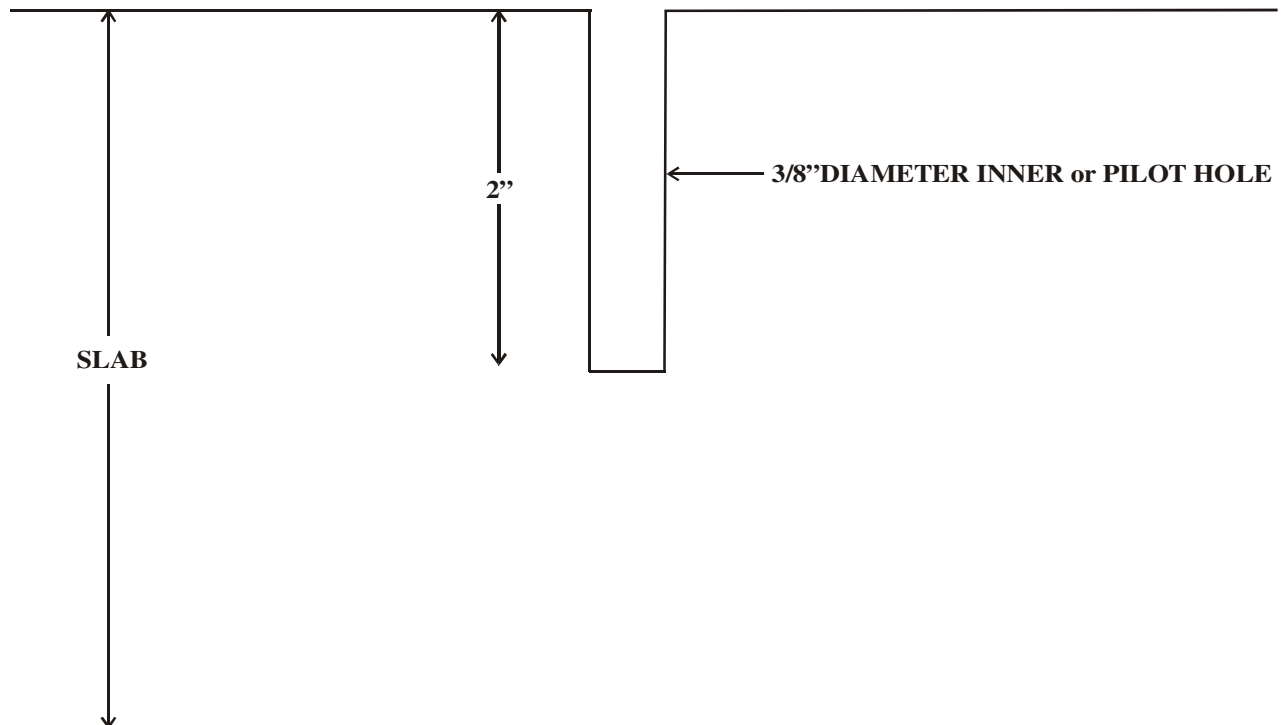
APPENDIX A
Soil Gas Installation Figures
SOP #2082
March 2007

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CONSTRUCTION AND INSTALLATION OF PERMANENT SUB-SLAB SOIL GAS WELLS

FIGURE 1
INNER or PILOT HOLE

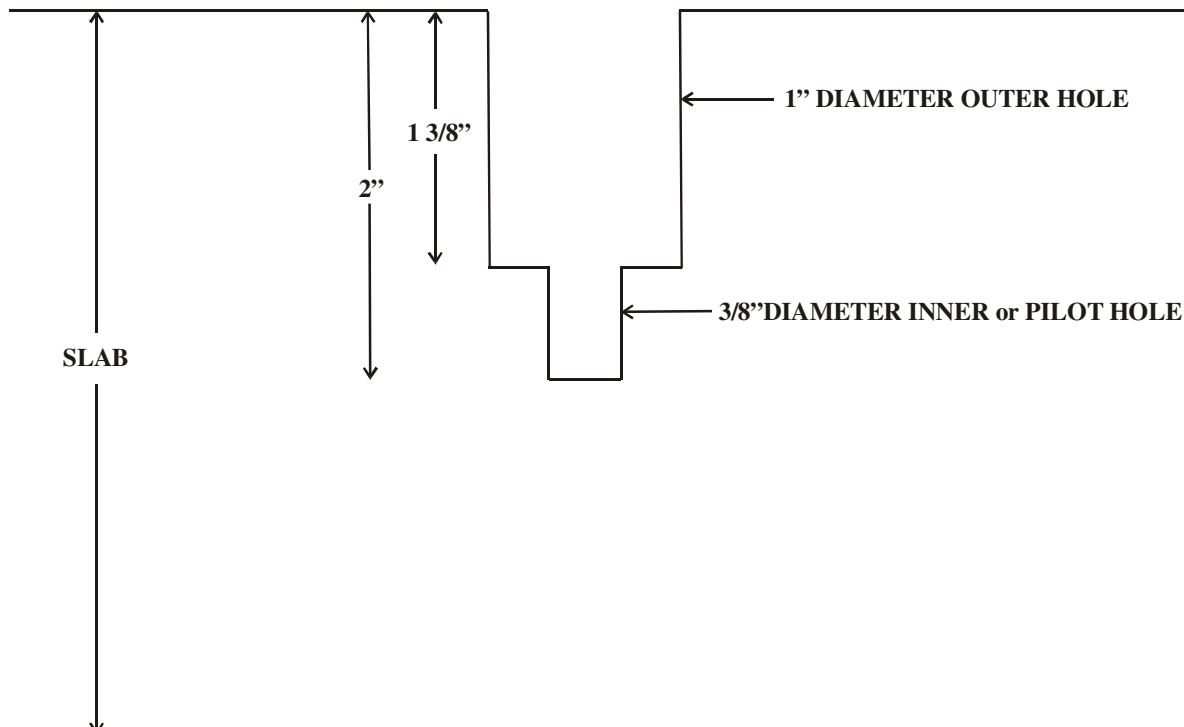


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FIGURE 2
OUTER HOLE



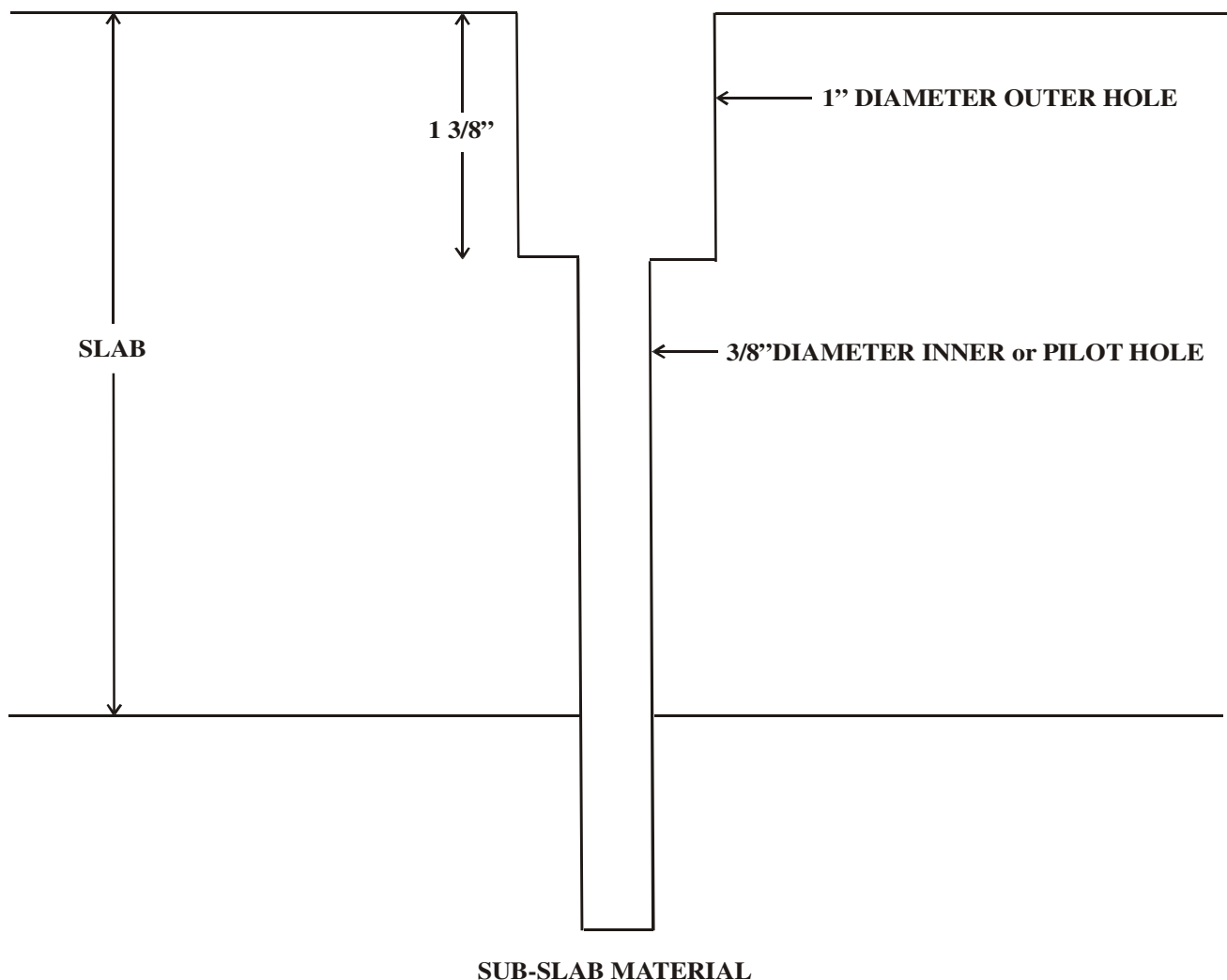
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CONSTRUCTION AND INSTALLATION OF PERMANENT SUB-SLAB SOIL GAS WELLS

FIGURE 3

COMPLETED HOLE PRIOR to PROBE INSTALLATION

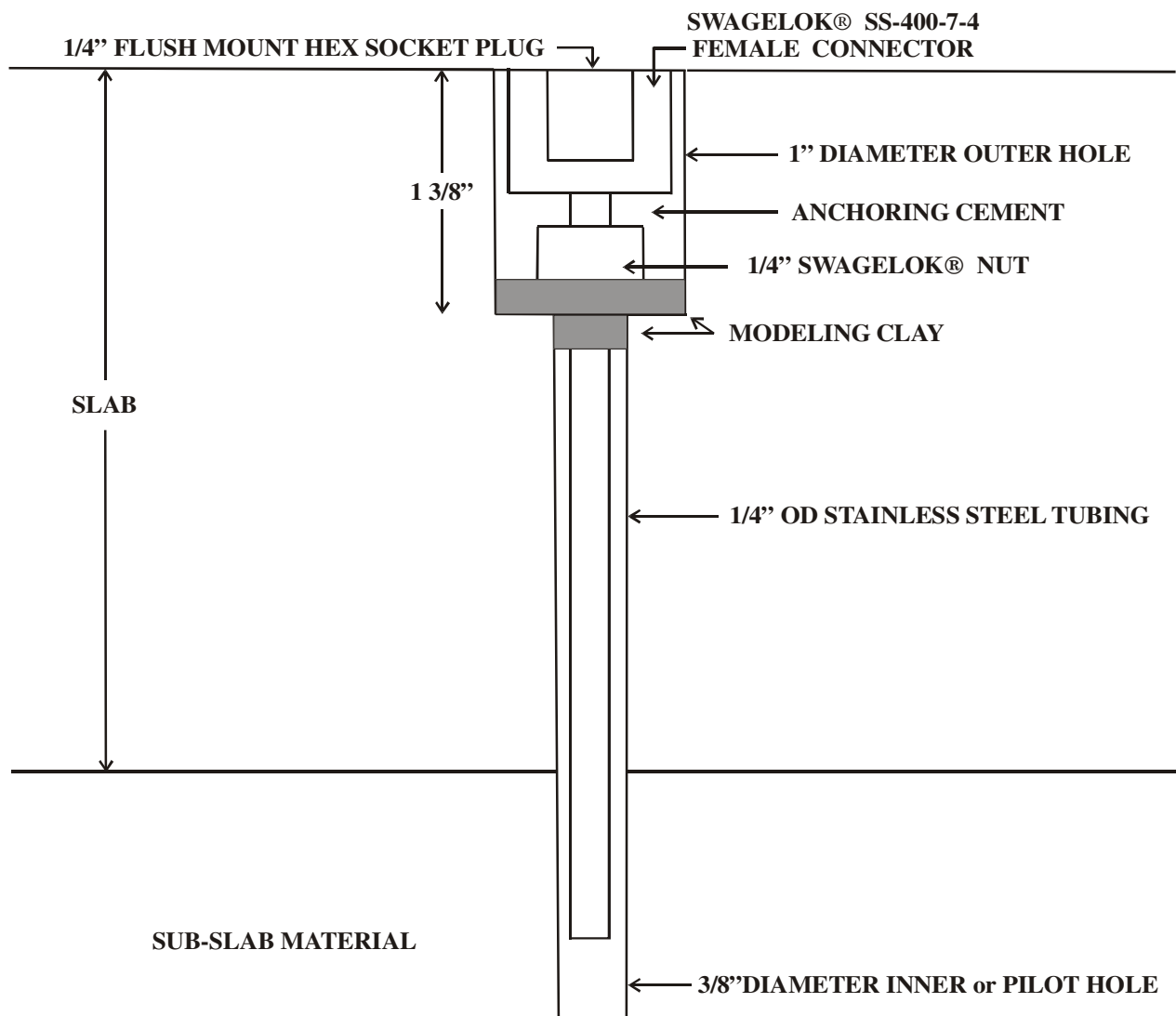


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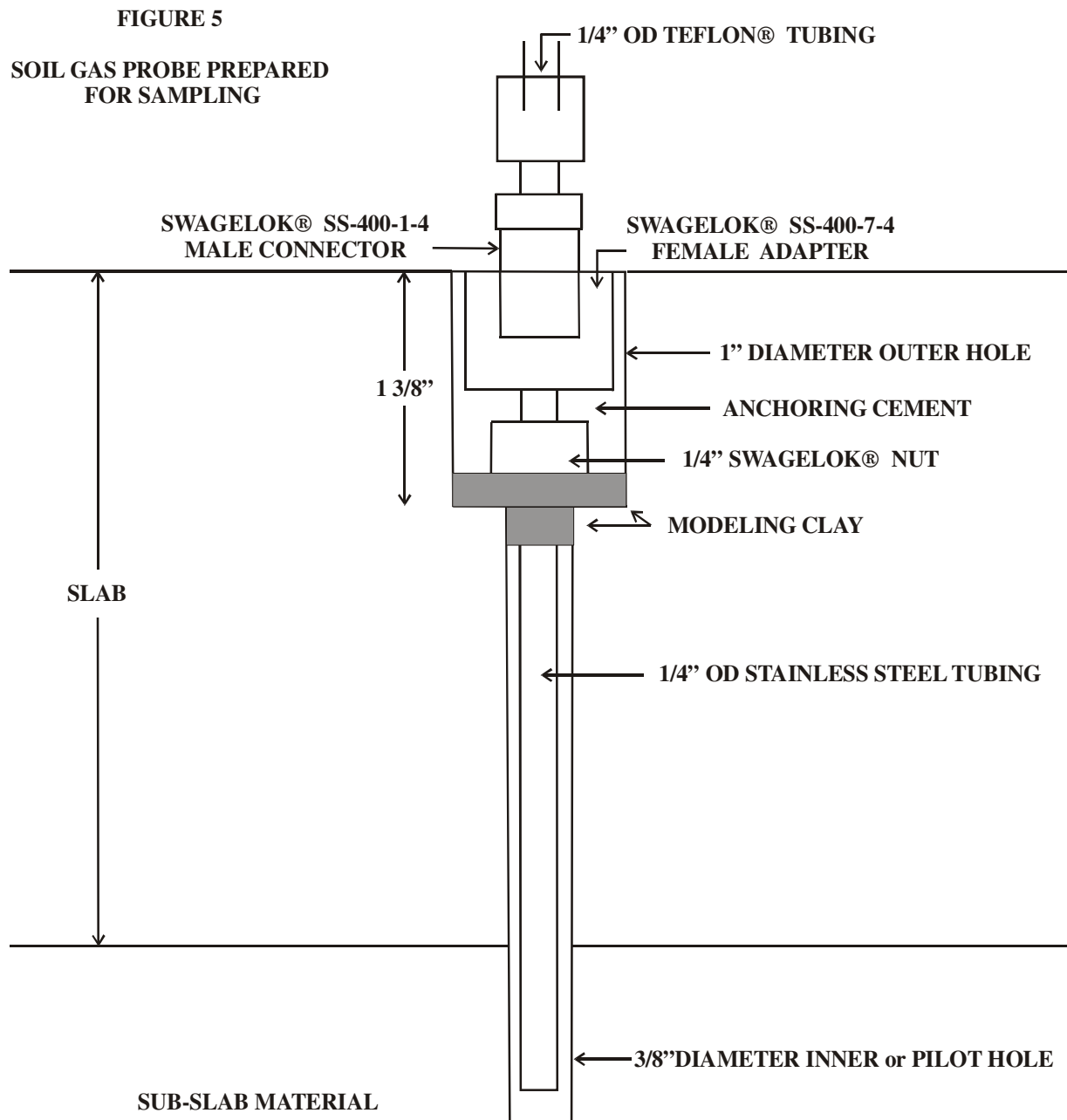
FIGURE 4
SOIL GAS PROBE INSTALLED



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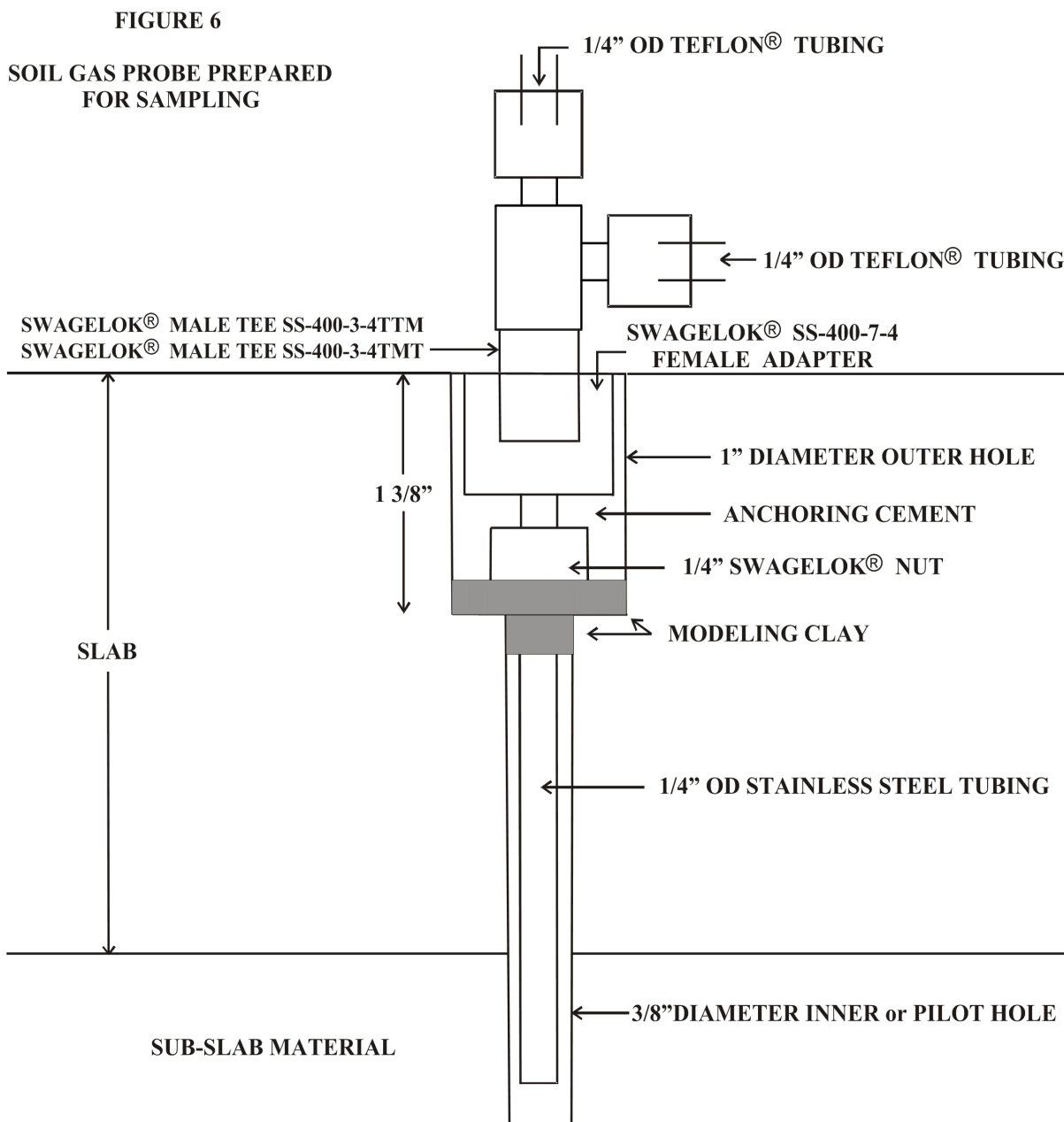
CONSTRUCTION AND INSTALLATION OF PERMANENT SUB-SLAB SOIL GAS WELLS



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SUPERFUND TECHNICAL ASSESSMENT RESPONSE TEAM
STANDARD OPERATING PROCEDURE

SOP 302
SURFACE SOIL SAMPLING

1.0 INTRODUCTION

The purpose of this Standard Operating Procedure (SOP) is to provide Roy F. Weston, Inc. (WESTON®), Superfund Technical Assessment Response Team (START) members with a step-by-step guide for collecting representative surface soil samples using scoops and bucket augers.

2.0 MATERIALS REQUIRED

Below is a list of the materials needed for surface soil sampling events. Both dedicated and reusable sampling equipment are required.

- Personal protective equipment (as specified in the Health and Safety Plan)
- Sampling plan
- Maps/sketches
- Compass
- Tape measure (up to 300 ft)
- Survey flags/stakes
- Aluminum homogenization pans
- Sample jars
- Logbook
- Sample labels/tags
- Chain-of-custody forms and custody seals
- Field data sheets
- Coolers
- Ice
- Decontamination equipment (brushes, buckets, garden sprayer, phosphate-free soap, water, etc.)
- Ziploc® bags
- Plastic sheeting
- Paper towels
- Ball-point pen
- Permanent marker
- Grease pencil
- Marking spray paint
- Digital camera or a camera with film
- Air monitoring equipment [Micro FID, Multi RAE 5 Gas detector, etc.]
- Plastic sample scoops, if applicable
- Bucket auger, if applicable
- Thin-walled tube sampler, if applicable

- Plastic garbage bag
- Scissors

3.0 SAFETY PRECAUTIONS

Due to unknown constituents of the soil media, the exposure potential for personnel exists and must be of primary concern. Before any soil sampling is performed, a Health and Safety Plan (HASP) must be approved by the Regional Safety Officer.

1. Follow the HASP safety schedule.
2. Determine the appropriate levels of protection to be worn by personnel.
3. Conduct air monitoring in the breathing zones and screen the sample location holes once they are selected.
4. Ensure that equipment is properly decontaminated and in working condition prior to the mobilizing to the site.
5. Coordinate efforts and staffing with the client or agency with which you are working.

4.0 SAMPLING PROCEDURES

1. Perform a general site reconnaissance to verify actual site conditions consistent with the HASP.
2. Identify and mark all sampling locations using sample flags or stakes as specified in the sampling plan. *All sample locations should be measured, documented, and mapped in reference to a permanent marker, i.e. specified utility pole, benchmark, property marker, etc.*
3. Mark the pertinent site information in a site logbook and on field data sheets. *When large amounts of samples are collected, field data sheets allow for easy organization in addition to logbook entries.*
4. Make sure all sampling equipment is properly decontaminated prior to sampling.
5. Wear clean, disposable surgical gloves for each sampling location.
6. Begin sampling by cutting or pulling back debris with a stainless steel or dedicated plastic scoop.
7. Cover the sample location area with plastic sheeting if the soil has a high probability of contamination.
8. Continue cutting to the required depth. Generally, surface sampling is considered 0-3 inches below the surface. It is recommended sample holes be kept the same size diameter (suggested 6 inches) even when using scoops to keep samples relative to each other. Sample collection will focus on soil particles, not plant and tree roots, stones, rocks, concrete and other materials intermixed in the soil matrix.
9. If a grab sample is to be collected, transfer the sample volume directly into the sample container using a sampling device. *Check the preferred sampling apparatus list for various analytical parameters.* A grab sample pertains to a discreet depth or area in a given matrix.
10. Transfer the sample volume to a homogenization container if the sample is a composite sample or a pseudo-grab sample. A composite sample is a mixture of different depths, areas, and/or strata. Composite samples are not recommended for

the collection of VOC samples because mixing causes volatile compounds to evaporate.

11. There are several homogenization techniques. The “quartering technique” requires the total volume of samples be divided into fourths inside the aluminum pan. Each quarter is then mixed individually, then the quarters are combined. This technique is repeated until a thorough mixing has occurred. The second method is the “bakers technique”, which simply entails mixing the soil volume with hands covered by surgical gloves or sampling scoops. The “shake and bake technique” allows the cleanest mixing. This technique requires emptying the sample volume into a Ziploc® bag, sealing the bag, and then shaking the bag until the sample volume is thoroughly mixed. Note the qualities (color, texture, etc.) of the homogenized sample.
12. Place the sample in the designated sample container after the sample has been homogenized.
13. Label the sample container. Sample labels and tags are to be filled out with a permanent marker (*ball point pen ink bleeds when wet*). Use a grease pencil to fill out labels and tags for samples to be analyzed for VOCs. Additionally, it is recommended that the bottom of the sample jar be marked with the time of collection, the sample location, and the sampler’s initials, in case the labels are rendered illegible.
14. Place the sample jar into an appropriate sized Ziploc® bag.
15. Place the sample on ice, if applicable. Generally, soil samples do not require any preservative; however, unless told otherwise, it is always good practice to put samples on ice.
16. Decontaminate the sampling apparatus using the proper procedure (see Section 6.0 Decontamination of Sampling Equipment).
17. Complete the chain-of-custody form in a clear and concise manner.
18. Repeat steps 1-17 for each sample location.

5.0 SAMPLING DEVICES

Three common sampling devices used by START personnel include the sample scoop, the auger, and the thin-walled sampler/corer. The sample scoop includes both dedicated disposable plastic scoops and stainless steel scoops. Augers include bucket augers and hand augers. The thin-walled sampler/corer is the least used device of the three.

5.1 Scoops

Scoops make sampling quick and easy. Any time rough terrain is encountered, scoops are the ideal device. Generally disposable scoops are used because no wet decontamination is required. Never reuse dedicated scoops and always make sure proper decontamination has been performed for non-disposable sample scoops.

5.2 Bucket and Hand Augers

Augers are manually driven stainless steel sampling devices. The hand auger is a smaller version of the bucket auger. Augers tend to fluff sample volumes. Because of their design, augers are recommended for composite sampling. Augers are not recommended for VOC sampling because volatiles will be driven off.

5.2.1 Auger Sampling Procedures

1. Decontaminate augers before collecting first sample.
2. Cut a 12-inch hole in the plastic sheeting around sample location using scissors.
3. Discard debris and other surface material.
4. Place the auger perpendicular to the ground and twist the “T” handle in a clockwise rotation until the desired depth is achieved. To determine the depth of the sample measure the actual removed core or the depth of the newly bore hole.
5. Retrieve the specified sample volume. Any additional sample volume can be returned to the sample hole.
6. Place the sample volume into a homogenization pan and mix thoroughly.
7. Place the sample in the designated sample container. Note: Only VOA containers are to be packed tightly.
8. Label the sample container. Sample labels and tags are to be filled out with a permanent marker (*ball point pen ink bleeds when wet*). Use a grease pencil to fill out labels and tags for samples to be analyzed for VOCs. Additionally, it is recommended that the bottom of the sample jar be marked with the time of collection, the sample location, and the sampler’s initials, in case the labels are rendered illegible.
9. Place the sample jar into an appropriate sized Ziploc® bag.
10. Place the sample on ice, if applicable. Generally, soil samples do not require any preservative; however, unless told otherwise, it is always good practice to put samples on ice.
11. Decontaminate the auger using the proper procedure (see Section 6.0 Decontamination of Sampling Equipment).
12. Complete the chain-of-custody form in a clear and concise manner.
13. Repeat steps 1-12 for each sample location.

Note: A major drawback for auger sampling is that roots, stones and other materials will not allow for good penetration. Different sample locations may have to be selected to collect samples.

5.3 Thin-Walled Sampler/Corer

The thin-walled sampler/corer is the least used of the common sampling devices. It works similar to an auger; however, it has a much smaller diameter and the core is visible from the side of the sampler barrel. This device is even more prone to refusal than the bucket auger. This device works well in moist soils with small grain sizes.

5.3.1 Corer Sampling Procedures

1. Decontaminate the augers before collecting the first sample.
2. Cut a 12-inch hole into plastic sheeting around sample location.
3. Discard debris and other surface material.
4. Place the thin-walled sampler perpendicular to the ground and twist the “T” handle in a clockwise rotation until desired depth is achieved.
5. Retrieve the specified sample volume. Any additional sample volume can be returned to the sample hole.
6. Place the sample volume into a homogenization pan and mix thoroughly.
7. Place the sample in the designated sample container.
8. Label the sample container. Sample labels and tags are to be filled out with a permanent marker only (*ball point pen ink bleeds when wet*). Use a grease pencil to fill out labels and tags for samples to be analyzed for VOCs. Additionally, it is recommended that the bottom of the sample jar be marked with the time of collection, the sample location, and the sampler’s initials, in case the labels are rendered illegible.
9. Place the sample jar in an appropriate sized Ziploc® bag.
10. Place the sample on ice, if applicable. Generally, soil samples do not require any preservative; however, unless told otherwise, it is always good practice to put samples on ice.
11. Decontaminate the auger using the proper procedure (see Section 6.0 Decontamination of Sampling Equipment).
12. Complete the chain-of-custody form.
13. Repeat steps 1-12 for each sample location.

6.0 DECONTAMINATION OF SAMPLING EQUIPMENT

This procedure is arguably the most important step in sound sample collection. Poor decontamination will result in cross-contamination and inaccurate sample results. The adequacy of the decontamination is generally tested by daily rinsate blanks. The following procedures pertain to the three sampling devices noted in this SOP.

1. Determine an area to be used as a decontamination station and lay plastic sheeting down.
2. Fill and pressurize a garden sprayer with distilled water. Fill one decontamination bucket with distilled water and Alconox®. Fill and pressurize another garden sprayer (if available) with de-ionized water for the final rinse.
3. Brush off soil residue from the sampling device with a dry brush.
4. Quickly spray the sampling device with the garden sprayer to loosen the soil before placing the sampling device into the soapy water.

5. Put the sampling device into soapy water bucket. Remove soil residue with a long-handled brush, toilet brush or cleaning device. Spray off soap residue with distilled water.
6. Place the sampling device into another bucket and spray the sampling device thoroughly again with distilled water.
7. Final rinse the sampling device with de-ionized water. If solvents or weak acids are used for the final rinse, see START SOP No. 406, Investigative Derived Waste.
8. If stainless steel scoops are used, use multiple scoops so that decontamination does not have to be after every hole.
9. Repeat steps 1-7.
10. Contact the OSC to determine if decontaminated water may be dumped on site. Be sure to address this issue before the sampling event occurs. All PPE and other refuse generated can be disposed as solid industrial waste.

7.0 REFERENCES

- EPA. 1991. *Compendium of Emergency Response Team (ERT) Soil Sampling and Surface Geophysics Procedures*. Office of Solid Waste and Emergency Response, Washington, DC. EPA/540/P-91/006.
- EPA 1991. *Removal Program Representative Sampling Guidance*. Volume 1 - Soil. Office of Solid Waste and Emergency Response, Washington, DC. 9630.4-10 P892-963408.
- WESTON® (Roy F. Weston, Inc.) 1993. *Standard Practices Manual for Soil Sampling With a Spade, Scoop and Stainless Surface Soil Sampler Auger and Tube Sampler*. West Chester, PA.

Attachment: 1

ATTACHMENT 1

SOIL SAMPLING DATA SHEET

Sample Number(s): _____
Date: _____
Time: _____

Soil Sampling Data Sheet

Site Name: _____ Sampler: _____

Sample Depth: _____ Surface (0-0.5 ft) _____ Shallow (0.5-5.0 ft)

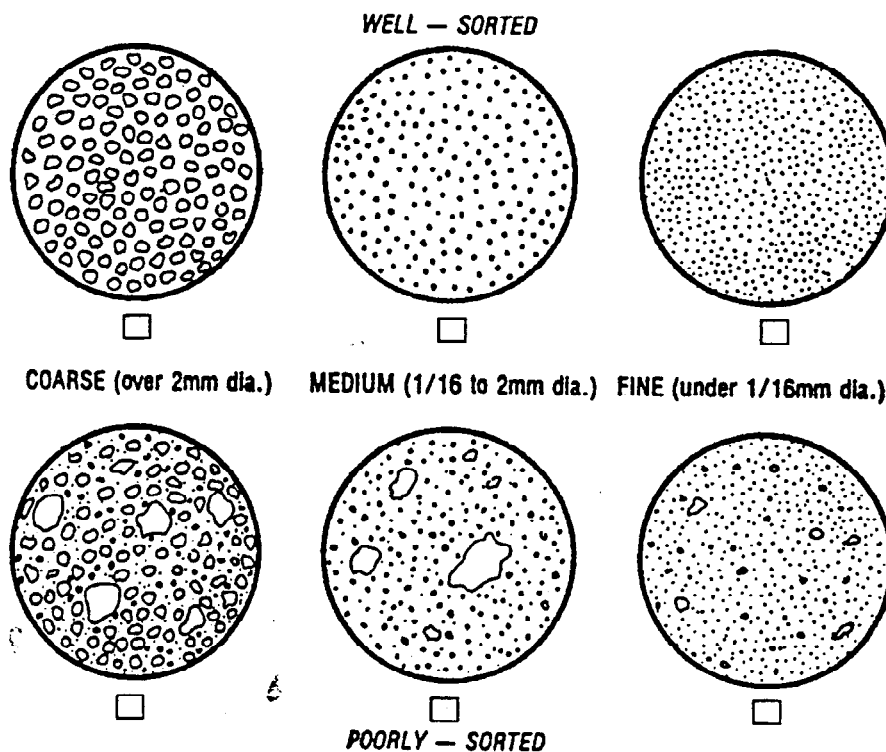
Sample Method(Circle One): _____ Scoop (2,3,4,5,6,7,8,A,C,+) _____ Hand Auger(2,3,4,5,6,7,B,+,-)
_____ Slide-Hammer (1,2,3,4,5,6,7,8,A,B,+,-) _____ Open Tube (A,+,-)
_____ Split/Solid Tube (1,2,3,4,5,6,7,8,A,B,-) _____ Thin-Wall Tube(8,A,-)

Preferred Methods

| | | | |
|--------------------|------------------|--------------------------|-------------|
| 1 - Volatiles | 5 - PCBs | A - Grab | + - Surface |
| 2 - Semi-Volatiles | 6 - TPH | B - Composite (Vertical) | - - Shallow |
| 3 - Primary Metals | 7 - Rad | C - composite (Areal) | |
| 4 - Pesticides | 8 - Geotechnical | | |

Soil Description (Munsell): Chart _____ Value _____ Hue _____

Grain Size and Distribution:



SUPERFUND TECHNICAL ASSESSMENT RESPONSE TEAM
STANDARD OPERATING PROCEDURE

SOP 304
SUBSURFACE SOIL SAMPLING

1.0 INTRODUCTION

The purpose of this Standard Operating Procedure (SOP) is to provide Roy F. Weston, Inc. (WESTON®), Superfund Technical Assessment Response Team (START) members with a step-by-step guide for collecting representative subsurface soil samples.

2.0 MATERIALS REQUIRED

Equipment requests should be completed and issued to the equipment room at least one week in advance of site operations. *Pre-assemble all sampling equipment that requires assembly prior to departure* (especially split-spoon apparatus).

Equipment for subsurface sampling is as follows:

- Personal protective equipment (as specified in the Health and Safety Plan)
- Sampling plan
- Maps/sketches
- Compass
- Tape measure (up to 300 ft)
- Survey stakes/flags
- Aluminum homogenization trays
- Sample jars
- Logbook
- Sample labels/tags
- Chain of custody forms and custody seals
- Field data sheets
- Coolers
- Ice
- Decontamination supplies (brushes, baskets, garden sprayer, phosphate-free soap, de-ionized water, etc.)
- Ziploc® bags
- Plastic sheeting
- Paper towels
- Ball-point pen
- Permanent marker
- Grease pencil
- Camera with film
- Plastic sample scoops
- Stainless steel trowels
- Plastic garbage bags
- Scissors

- Methanol/hexane/nitric acid
- Screw auger
- Bucket auger
- Post-hole auger
- T-handle
- Extension rods
- Split spoon halves (2 sets) with shoes (2)
- Plastic sample baskets
- Adapter
- Drive head
- Bit(s)
- Extension (optional)
- Slam bar (manual sampling only)
- Slam hammer (manual sampling only)
- Extraction head
- Gas can
- Generator oil
- Generator
- Jackhammer (rental)
- Heavy duty lift-jack
- Wheel barrow
- Ground fault interrupter (GFI)
- 24-inch pipe wrenches (2)
- Manual sledge hammer
- Rubber mallet
- Spray-lubricant (ingredients must not interfere with analysis parameters)
- Shovel/spade
- Roofing nails
- Tool box (vice grips, adjustable wrenches, pliers, channel locks, etc.)
- Back support belt(s)

3.0 SAFETY PRECAUTIONS

Due to unknown constituents of the soil media, the exposure potential for personnel exists and must be of primary concern. Before any soil sampling is performed, a Health and Safety Plan (HASP) must be approved by the Regional Safety Officer.

1. Follow the HASP safety schedule.
2. Determine the appropriate levels of protection to be worn by personnel.
3. Conduct air monitoring in the breathing zones and screen the sample location holes once they are selected.
4. Ensure that equipment is properly decontaminated and is in working condition prior to mobilization to the site.
5. Coordinate efforts and staffing with the client or agency with which you are working.

4.0 SAMPLING PROCEDURES

4.1 Sampling Preparation

1. Perform a general site reconnaissance to verify actual site conditions consistent with the HASP.
2. Identify and mark all sampling locations using sample flags or stakes as specified in the sampling plan. *All sample locations should be measured, documented, and mapped in reference to a permanent marker, i.e. specified utility pole, benchmark, property marker, etc.* Note: It is often convenient (for residential properties, etc.) to hammer roofing nails into wooden stakes which can be driven below the surface; therefore, a hand-held metal detector can be used to find exact sample locations.
3. Make sure all sampling equipment is properly decontaminated prior to sampling.
4. Notify utility companies to check sampling areas for underground utilities prior to digging, boring, or coring operations. Most states require 7-day notice, and have a toll-free number to arrange a single, all-inclusive, free inspection.

4.2 Auger Sampling

1. Wear appropriate personal protective equipment (PPE), as defined by the site Health and Safety Plan (HASP). *Remember that a clean pair of sample gloves must be worn for each sample collected.*
2. Assemble the auger unit, if required.
3. Clear the area to be sampled of any surface debris.
4. Spread plastic sheeting near the sample location to allow for easy accessibility and to prevent equipment contamination.
5. Remove surface soil to expose the desired depth of soil to be sampled using a screw auger, bucket auger, post-hole auger, power auger, shovel, or backhoe. The sampling device (auger, split-spoon, etc.) can be marked with a grease pencil.
6. Stage removed soil on plastic sheeting near the hole. *Special care should be taken to avoid allowing surface soil to cross-contaminate soil at the desired sampling depth.*
7. Use auger extension rods to sample to depths up to approximately 9 feet.
8. Use a *decontaminated* auger to collect sample(s) after digging or boring to the desired sample depth. The best way to retrieve desired soil, while avoiding cross-contamination, is with a bucket auger. Each 6-inch bucket auger will hold approximately 3-4 inches of augered (“fluffed”) soil.
9. Decontaminate the augers after each sample-depth is collected. Note: Discarding the top inch of soil from the retrieved sample also reduces cross-contamination.
10. Transfer a portion of the sample directly into an appropriate, labeled sample container(s) with a stainless steel trowel if volatile organic analysis is to be performed.

11. Deposit sample into a homogenization tray for analysis other than volatile organics and thoroughly mix the soils to obtain a homogenous, representative sample.
12. Place homogenized sample into an appropriate, labeled sample container with a plastic scoop or stainless steel trowel, depending on the requested analysis.
13. Record all field notes on field data sheets and in the site logbook as specified in the sampling plan. The sample location should reference a permanent marker, and should be mapped and described in the field notes.
14. Photograph sample locations with landmarks in view. Keep in mind that sample locations may need to be referenced in the future, often years after your sampling event.
15. Abandon the sample hole(s) in accordance with state regulations. Generally, holes can be backfilled with removed soil materials.
16. Decontaminate all sampling equipment in accordance with SOP No. 301 Decontamination Procedures.

4.3 Split Spoon Sampling

1. Wear appropriate personal protective equipment (PPE), as defined by the site HASP. *Remember that a clean pair of sample gloves must be worn for each sample collected.*
2. Assemble cleaned spoon halves by aligning threads, inserting a clean sample basket, and screwing on the shoe.
3. Screw on the appropriate adapter for a slam bar or jackhammer apparatus. For manual sample collection, attach the slam bar to the adapter. For jackhammer-driven sample collection, attach the drive head to the adapter.
4. Clear the area to be sampled of any surface debris.
5. Spread plastic sheeting near the sample location.
6. Remove surface soil to expose the desired depth of soil to be sampled using a screw auger, bucket auger, post-hole auger, power auger, shovel, or backhoe.
7. Stage removed soil on plastic sheeting near the hole. *Special care should be taken to avoid allowing surface soil to cross-contaminate soil at desired sampling depth.*
8. Use a *decontaminated* split-spoon to collect the sample(s) after digging or boring to desired sample depth.
9. For the manual collection of a sample, slide the slam hammer over the slam bar. Slam the hammer repeatedly until the core is driven to desired depth.
10. For jackhammer-facilitated sampling, attach and lock the bit to the jackhammer. Carefully lift the jackhammer, slide the bit into the adapter, and drive the core to the desired depth. One person should support the spoon halves and the adapter during coring operations to insure that the apparatus remains vertical and that individual parts do not come unscrewed.
11. Attach the appropriate extraction head to the in-ground split spoon apparatus.
12. Using a heavy-duty lift-jack, carefully retrieve the sample core. This step usually requires two people: one to hold the jack in a vertical position while the other operates the jack.
13. Lay the core on clean plastic sheeting after the core is retrieved and carefully

- unscrew the extraction head and the adapter.
14. Unscrew the shoe, but *make sure the split spoon core does not open*. Often, pipe wrenches are required to unscrew the shoe.
 15. Lay out *labeled* homogenization trays alongside the sample core.
 16. Open the split spoon and take care not to spill or disturb the sample core.
 17. Measure the desired soil depths.
 18. Use clean gloves and a clean polyethylene scoop to retrieve each sample. *Do not slide the soil down the spoon to retrieve it*. Instead, use the scoop to carefully lift the sample into a homogenization tray.
 19. Transfer a portion of the sample directly into an appropriately labeled sample container(s) with a stainless steel trowel if volatile organic analysis is to be performed.
 20. Deposit the sample into a homogenization tray for analysis other than volatile organics, and thoroughly mix the soils to obtain a homogenous, representative sample.
 21. Place the homogenized sample into an appropriate, labeled sample container using a plastic scoop or stainless steel trowel, depending on the requested analysis.
 22. Record all field notes on field data sheets and the site logbook, as specified in the sampling plan. The sample location should reference a permanent marker, and should be mapped and described in the field notes.
 23. Photograph sample locations with landmarks in view. Keep in mind that sample locations may need to be referenced in the future, often years after your sampling event.
 24. Abandon the sample hole(s) in accordance with state regulations. Generally, holes can be back filled with removed soil materials.
 25. Decontaminate all sampling equipment in accordance with ERT SOP No. 2006
 26. Sampling Equipment Decontamination.

5.0 REFERENCES

- EPA. 1991. *Compendium of Emergency Response Team (ERT) Waste Sampling Procedures*. Office of Solid Waste and Emergency Response, Washington, DC. EPA/540/P-91/008.
- EPA 1991. *Removal Program Representative Sampling Guidance*. Volume 1 - Soil. Office of Solid Waste and Emergency Response, Washington, DC. 9630.4-10 P892-963408.
- WESTON® (Roy F. Weston, Inc.) 1993. *Standard Practices Manual for Soil Sampling With a Spade, Scoop and Stainless Surface Soil Sampler Auger and Tube Sampler*. West Chester, PA.

Attachment: 1

ATTACHMENT 1

SOIL SAMPLING DATA SHEET

Sample Number(s): _____
 Date: _____
 Time: _____

Soil Sampling Data Sheet

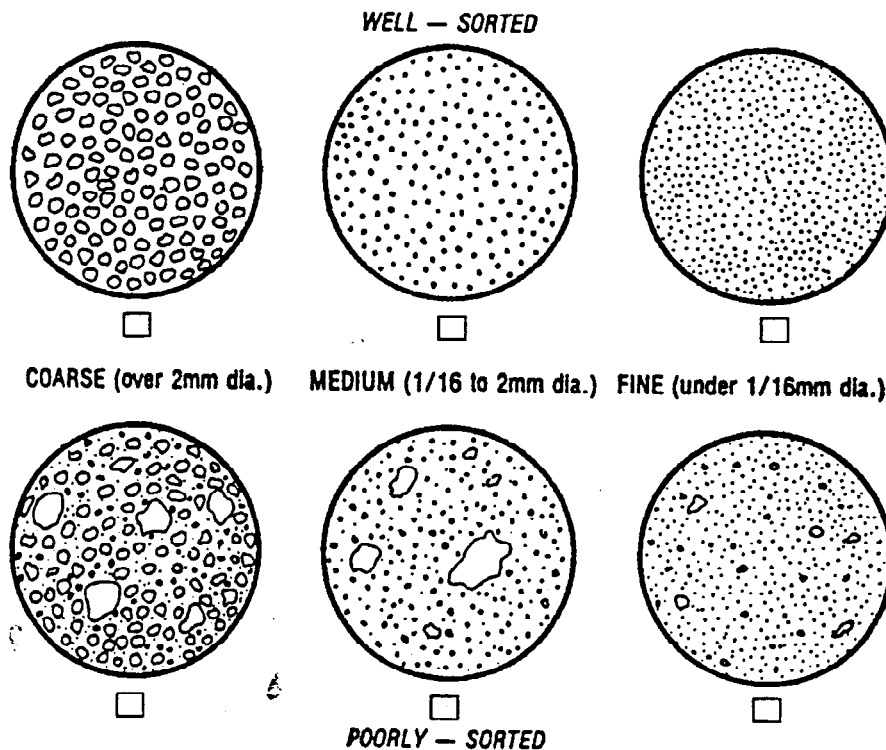
Site Name: _____ **Sampler:** _____
Sample Depth: _____ Surface (0-0.5 ft) _____ Shallow (0.5-5.0 ft)
Sample Method(Circle One): _____ Scoop (2,3,4,5,6,7,8,A,C,+) _____ Hand Auger(2,3,4,5,6,7,B,+,-)
 _____ Slide-Hammer (1,2,3,4,5,6,7,8,A,B,+,-) _____ Open Tube (A,+,-)
 _____ Split/Solid Tube (1,2,3,4,5,6,7,8,A,B,-) _____ Thin-Wall Tube(8,A,-)

Preferred Methods

| | | | |
|--------------------|------------------|--------------------------|-------------|
| 1 - Volatiles | 5 - PCBs | A - Grab | + - Surface |
| 2 - Semi-Volatiles | 6 - TPH | B - Composite (Vertical) | - - Shallow |
| 3 - Primary Metals | 7 - Rad | C - composite (Areal) | |
| 4 - Pesticides | 8 - Geotechnical | | |

Soil Description (Munsell): Chart _____ Value _____ Hue _____

Grain Size and Distribution:



APPENDIX C
ANALYTICAL SOPs



Pace Analytical Services, Inc.

7726 Moller Road
Indianapolis, IN 46268

Phone: 317.875.5894

Fax: 317.872.6189

STANDARD OPERATING PROCEDURE

THE DETERMINATION OF METALS BY INDUCTIVELY COUPLED PLASMA (ICP)

REFERENCE METHOD: EPA SW-846 METHOD 6010B

SOP NUMBER: S-IN-I-019-rev.10

EFFECTIVE DATE: Date of Final Signature

SUPERSEDES: S-IN-I-019-rev.09

APPROVAL

General Manager

September 27, 2012

Date

Quality Manager

September 26, 2012

Date

Department Manager

September 25, 2012

Date

PERIODIC REVIEW

SIGNATURES BELOW INDICATE NO CHANGES HAVE BEEN MADE SINCE APPROVAL.

Signature

Title

Date

Signature

Title

Date

Signature

Title

Date

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1. Purpose

- 1.1 The purpose of this SOP is to provide a laboratory specific procedure for determining the concentration of metals in aqueous and solid environmental samples while meeting the requirements specified in SW-846 method 6010B.

2. Summary of Method

- 2.1 Prior to analysis, samples must be solubilized or digested using appropriate sample preparation methods. When analyzing groundwater samples for dissolved constituents, acid digestion is not necessary if the samples are filtered and acid preserved prior to analysis.
- 2.2 This method describes multielement determinations by Inductively Coupled Plasma – Atomic Emission Spectrometry (ICP-AES). The instrument measures characteristic emission spectra by optical spectrometry. Samples are nebulized and the resulting aerosol is transported to the plasma torch. Element-specific emission spectra are produced by radio-frequency inductively coupled plasma. The spectra are dispersed by a grating spectrometer, and the intensities of the emission lines are monitored by photosensitive devices.
- 2.3 Background correction may be required to compensate for spectral interferences. Background is measured adjacent to analyte lines at a wavelength selected to be free of spectral interference and which reflects the same change in background intensity as occurs at the wavelength measured. Background correction is not required in cases of line broadening where a correction would actually degrade the analytical result.

3. Scope and Application

- 3.1 This method is applicable to the determination of most trace elements, including metals, in solution. Refer to Table 1 for the list of metals and reporting limits. Refer to the LIMS for method detection limits.
- 3.2 This method is applicable to groundwater, surface water, wastewater, extract, leachate, soil, sediment, sludge and other solid samples.
- 3.3 Reporting limits, control limits, volumes/weights used, standard concentrations, vendors, instrumentation, equipment and supplies are subject to change.
- 3.4 This procedure is restricted to use by, or under the supervision of, analysts experienced in the use of ICP systems and interpretation of ICP data. Each analyst must demonstrate the capability to generate acceptable results with this method to be considered qualified to report sample results.

4. Interferences

- 4.1 **Spectral interferences:** Overlap of emission lines from another element, unresolved overlap of molecular band spectra, background contribution from continuous or recombination phenomena and stray light can contribute to spectral interferences. These interferences can typically be minimized by careful selection of quantitation wavelengths, inter-element corrections, and background correction.
- 4.2 **Physical interferences:** Changes in sample viscosity, surface tension, or other effects associated with sample transport and nebulization can produce significant inaccuracies, especially in samples containing high concentrations of dissolved solids and acids. Dissolved solids may build up on the nebulizer tip, altering the sample flow rate and causing instrument drift. These effects can be minimized by sample dilution or use of a specially designed high-solids nebulizer.
- 4.3 **High Salt Concentrations:** high salt concentrations in sample digestates can cause signal suppression and confuse interference tests.

- 4.4. Chemical interferences:** Molecular compound formation, ionization effects, and solute vaporization effects are typically not significant with ICP determinations. If observed, they can be minimized by careful selection of plasma and spectrometer operating parameters.
- 4.5. Memory interferences:** Sample deposition on the nebulizer tubing, spray chamber, and plasma torch can cause apparent sample carryover. Memory interferences can be minimized by flushing the system with rinse blanks between samples. If memory interference is suspected for a sample, the sample must be re-analyzed after a sufficient rinse period.

5. Safety

- 5.1. Standards and Reagents:** The toxicity and carcinogenicity of standards and reagents used in this method have not been fully defined. Each chemical compound should be treated as a potential health hazard. Reduce exposure by the use of gloves, lab coats and safety glasses. Material Safety Data Sheets (MSDSs) are on file in the laboratory and available to all personnel. Standard solutions should be prepared in a hood whenever possible. The stock metals standards are toxic and must be handled with extreme care. Also handle concentrated acids with care, making sure to wear appropriate personal protective equipment.
- 5.2. Samples:** Take precautions when handling samples. Samples must always be treated as potentially hazardous “unknowns”. The use of personal protective equipment such as gloves, lab coats and safety glasses is required when handling samples. In the event a sample container must be opened, it is recommended to perform this in a hood whenever possible. All digestions must be conducted under a fume hood.
- 5.3. Equipment:** Portions of the preparation and analytical equipment operate at high temperatures. Care must be taken to minimize accidents and injuries when working on or with this equipment. Equipment should be turned off or the temperatures lowered to reduce the risk of thermal burns. Allow adequate time for the equipment to cool prior to working on equipment.

6. Definitions

- 6.1.** Refer to Glossary section of the Pace Quality Assurance Manual (QAM) for a comprehensive list of terms and definitions.

7. Sample Collection, Preservation, and Handling

Table 7.1 – Sample Collection, Preservation, Storage and Hold time.

| Sample type | Collection per sample | Preservation | Storage | Hold time |
|----------------------------|--|---|-----------------------|--|
| Aqueous - Total | 250mL in plastic container | - HNO ₃ to pH of <2 - Samples received at pH>2 must be preserved to pH<2 with HNO ₃ and equilibrate for 24 hours before being prepared for analysis. | Ambient or 0°C to 6°C | Must be analyzed within 6 months of the collection date. |
| Aqueous - Dissolved | 250mL in plastic container | - Filter; HNO ₃ to pH<2 | Ambient or 0°C to 6°C | Must be analyzed within 6 months of the collection date. |
| Solid | 50 grams in glass or plastic container | - No chemical preservation | Ambient or 0°C to 6°C | Must be analyzed within 6 months of the collection date. |

Samples must be stored separately from all standards, reagents, and highly contaminated samples. To avoid contamination, no food or drink products can be located near samples.

8. Equipment and Supplies

8.1. Equipment/Instrumentation

| Equipment | Vendor | Description / Comments |
|-----------|--------------------------------------|---|
| ICP-AES | Thermo-Fisher iCAP6500 or equivalent | Equipped with autosampler and data system |

8.2. General Supplies

| Item | Vendor | Description |
|----------------------|-------------------------------------|------------------------------|
| Volumetric Flasks | Class A | Various capacities |
| Volumetric Pipettors | Eppendorf or equivalent | Various sizes |
| Autosampler Vials | Environmental Express or equivalent | |
| Analytical Balance | Ohaus or equivalent | Capable of weighing to 0.01g |
| Graduated Cylinders | Class A | Various capacities |
| pH strips | Fisher or equivalent | Full range |

9. Reagents and Standards

9.1. Reagents

| Reagent | Concentration/ Description |
|-------------------|--|
| Reagent water | ASTM Type II |
| Argon | High purity, liquefied |
| Nitric acid | Concentrated, trace metal analyzed or equivalent |
| Hydrochloric acid | Concentrated, trace metal analyzed or equivalent |

9.2. Analytical Standards

9.2.1. Definitions

Standards are required for initial calibration, calibration verification standards, second source verification, and for preparing LCS, MS, and MSD samples.

Table 9.2 Standard Definitions

| Standard | Description | Comments |
|--|---|--|
| Initial Calibration Standards | Standards prepared at varying levels to determine calibration range of the instrument. | ICAL |
| Initial Calibration Verification Standard | A standard prepared from a source other than that used for the initial calibration. This standard verifies the accuracy of the calibration curve. | ICV |
| Continuing Calibration Verification Standard | A calibration standard prepared at mid-level concentration for all target compounds. This standard is used to verify the initial calibration. | CCV |
| Spiking Standard | This solution contains the target analytes and is used to spike MS/MSD sets.. | Same solution can be used for the LCS and MS/MSD |
| Internal Standard | A solution added to all standards, samples, spikes, control samples, and method blanks prior to analysis. This standard is used to adjust response ratios to account for instrument drift. | Yttrium |
| Interference Check Standards | Prepared to contain a known amount of interfering elements that will provide an accurate test of the interelement correction factors. If the ICP will display overcorrection as a negative number, the additional spiking with interfered elements is not necessary.. | ICSA (ICSAB for BP Samples only) |

9.2.3 Storage Conditions

Table 9.3 – Analytical Standard Storage Conditions

| Standard Type | Description | Expiration | Storage |
|--|---|---|---|
| Stock Calibration Standards | SPEX; catalog #'s MIXSTD1-100; MIXSTD2-100; MIXSTD3-100; MIXSTD4-100; MIXSTD5-100; PLS19-2Y; CLSN2-2Y; PLTI9-2Y; CLAG2-2Y or equivalent | Manufacturer's recommended expiration date | Manufacturer's recommended storage conditions |
| Working Calibration Standards | Refer to Section 9.2.3.2 | Must be prepared fresh weekly | Same as stock standards |
| Stock ICV Standard | Inorganic Ventures; catalog #s PA-STD-1B; PA-STD-2B; PA-STD-3B or equivalent | Manufacturer's recommended expiration date | Manufacturer's recommended storage conditions |
| Working ICV Standard | Refer to Section 9.2.3.4 | Must be prepared fresh weekly | Same as stock standard |
| Working Second Source Spiking Solution | Refer to Section 9.2.3.5 | Standard is good for 6 months from date of preparation. | Same as stock standard |
| Stock Interference Check Standard A | SPEX; catalog # INT-A1 or equivalent | Manufacturer's recommended expiration date | Manufacturer's recommended storage conditions |
| Working Interference Check Standard A (ICSA) | Refer to Section 9.2.3.6 | Must be prepared fresh weekly | Same as stock standards |
| Stock Interference Check Standard AB | SPEX; catalog #INT-A1, Mix-1B, Mix-2B, or equivalent | Manufacturer's recommended expiration date | Manufacturer's recommended storage conditions |
| Working Interference Check Standard AB (ICSAB) | Refer to Section 9.2.3.8 | Must be prepared fresh weekly | Same as stock standards |
| Stock CRDL standard | CPI; catalog # 4400-132804, or equivalent | Manufacturer's recommended expiration date | Manufacturer's recommended storage conditions |
| Working CRDL standard | Refer to Section 9.2.3.10 | Must be prepared fresh weekly | Same as stock standards |
| Stock Internal Standard | SPEX; catalog # PLY2-2X; 1000mg/L yttrium or equivalent VHG; catalog # TLIN-500; 10,000mg/L lithium or equivalent | Manufacturer's recommended expiration date | Manufacturer's recommended storage conditions |
| Working Internal Standard | Refer to Section 9.2.3.11 | Must be prepared fresh weekly | Same as stock standards |

9.2.3 Standard Preparation Procedures

9.2.3.1 Stock Calibration Standard Details

The following table shows the seven stock standard mixes that may be used to prepare the initial calibration and calibration check standards:

| Analyte | Concentration (mg/L) |
|--|----------------------|
| <i>Catalog # MIXSTD1-100</i> | |
| Lead | 500 |
| Selenium | 200 |
| Cadmium | 150 |
| Zinc | 150 |
| Manganese | 100 |
| Beryllium | 50 |
| <i>Catalog # MIXSTD2-100</i> | |
| Iron | 10,000 |
| Barium | 100 |
| Cobalt | 100 |
| Copper | 100 |
| Vanadium | 100 |
| <i>Catalog # MIXSTD3-100 + silicon PLSI9-2Y</i> | |
| Arsenic | 500 |
| Molybdenum | 100 |
| Silicon | 100 |
| <i>Catalog # MIXSTD4-100</i> | |
| Calcium | 1000 |
| Potassium | 400 |
| Aluminum | 200 |
| Sodium | 200 |
| Chromium | 20 |
| Nickel | 20 |
| <i>Catalog # MIXSTD5-100</i> | |
| Magnesium | 1000 |
| Antimony | 200 |
| Thallium | 200 |
| Boron | 100 |
| Silver | 50 |
| <i>Mix #6 (combines the following: CLSN2-2Y; PLTI9-2Y)</i> | |
| Tin | 1000 |
| Titanium | 1000 |
| <i>Mix #7- Catalog #CLAG2-2Y</i> | |
| Silver | 1000 |

9.2.3.2 Working Calibration Standards Preparation

Prepared fresh weekly and diluted from the stock standard mixes listed above, using a reagent water mixture that is 5% nitric acid and 2% hydrochloric acid unless otherwise noted.

| Working Std. ID | Stock Standard | Vol. of Stock Std. | Final Volume |
|------------------------|-----------------------------------|--------------------|---------------------------|
| Calibration Std. Mix 1 | MIXSTD1-100 | 2mL | 100mL |
| Calibration Std. Mix 2 | MIXSTD2-100 | 1mL | 100mL |
| Calibration Std. Mix 3 | MIXSTD3-100 Silicon PLSI9-2Y | 2mL 0.8mL | 100mL |
| Calibration Std. Mix 4 | MIXSTD4-100 | 5mL | 100mL |
| Calibration Std. Mix 5 | MIXSTD5-100 | 2mL | 100mL |
| Calibration Std. Mix 6 | Tin CLSN2-2Y Titanium PLTI9-2Y | 1mL 1mL | 100mL |
| Calibration Std. Mix 7 | Silver CLAG2-2Y | 0.2mL | 100mL in 10% HCl solution |

9.2.3.3 Stock ICV Standard Details

The following table shows the concentrations of the stock standards purchased from Inorganic Ventures as three mixes:

| Analyte | Concentration (mg/L) |
|---|----------------------|
| <i>Inorganic Ventures PA-STD1B</i> | |
| Arsenic | 200 |
| Barium | 200 |
| Beryllium | 200 |
| Cadmium | 200 |
| Cobalt | 200 |
| Chromium | 200 |
| Copper | 200 |
| Manganese | 200 |
| Nickel | 200 |
| Phosphorus | 200 |
| Lead | 200 |
| Selenium | 200 |
| Thallium | 200 |
| Lithium | 200 |
| Strontium | 200 |
| Vanadium | 200 |
| Zinc | 200 |
| <i>Inorganic Ventures PA-STD2B</i> | |
| Silicon | 1000 |
| Boron | 200 |
| Molybdenum | 200 |
| Antimony | 200 |
| Tin | 200 |
| Titanium | 200 |
| Zirconium | 200 |
| Silver | 100 |
| <i>Inorganic Ventures PA-STD3B</i> | |
| Aluminum | 2000 |
| Calcium | 2000 |
| Iron | 2000 |
| Potassium | 2000 |
| Magnesium | 2000 |
| Sodium | 2000 |

9.2.3.4 Working ICV Standard Preparation

Add 0.5mL of each Stock ICV Standard mix to a 100mL volumetric flask and dilute to volume with a reagent water solution that is 5% nitric acid and 2% hydrochloric acid.

9.2.3.5 Working Second Source Spiking Solution

Add 25.0 mL of each Stock ICV Standard mix to a 100 mL volumetric flask and dilute to volume with reagent water solution that is 2% nitric acid.

9.2.3.6 Stock Interference Check Standard A (ICSA) Details

| <i>SPEX Interference Check Standard A (ICSA)</i> | |
|---|------|
| Aluminum | 5000 |
| Calcium | 5000 |
| Magnesium | 5000 |
| Iron | 2000 |

9.2.3.7 Working Interference Check Standard A (ICSA) Preparation

Dilute 10mL of the Stock ICSA Standard to 100mL with a reagent water solution that is 5% nitric acid and 2% hydrochloric acid.

9.2.3.8 Stock Interference Check Standard AB (ICSAB) Details

| <i>SPEX INT-A1</i> | |
|---|------|
| Al, Ca, Mg | 5000 |
| Fe | 2000 |
| <i>SPEX Mix 1B</i> | |
| As, Ba, Be, B, Cd, Co, Cr, Cu, Mn, Ni, Pb, Se, Tl, V, Zn | 100 |
| <i>SPEX Mix 2B</i> | |
| Mo, Sb, Sn, Ti | 100 |
| Ag | 50 |
| Si | 500 |

9.2.3.9 Working Interference Check Standard AB (ICSAB) Preparation

Dilute 10mL of the stock INT-A1 standard and 0.5mL of the stock Mix 1B and Mix 2B to 100mL with a reagent water solution that is 5% nitric acid and 2% hydrochloric acid.

9.2.3.10 Stock Contract Required Detection Limit (CRDL) Standard Details

When specified by client or program requirements, a low-level check standard, also known as a CRDL standard, must be analyzed prior to sample analysis and at the end of each analytical batch to bracket the client samples. Acceptance limits for all target elements is 50-150% recovery. The composition of the CRDL standard is listed as follows:

| Element | Conc. (ug/mL) | Element | Conc. (ug/mL) |
|-----------|---------------|------------|---------------|
| Aluminum | 100 | Manganese | 5 |
| Antimony | 5 | Molybdenum | 15 |
| Arsenic | 5 | Nickel | 20 |
| Barium | 1.5 | Potassium | 2500 |
| Beryllium | 5 | Selenium | 5 |
| Boron | 75 | Silver | 5 |
| Cadmium | 0.5 | Sodium | 1000 |
| Calcium | 250 | Thallium | 10 |
| Chromium | 10 | Tin | 35 |
| Cobalt | 10 | Titanium | 10 |
| Copper | 5 | Vanadium | 15 |
| Iron | 50 | Zinc | 20 |
| Lead | 3 | Gold | 10 |
| Magnesium | 250 | Strontium | 10 |

9.2.3.11 Working Contract Required Detection Limit (CRDL) Standard Preparation

Dilute 0.2mL of the stock CRDL solution to 100mL with a reagent water solution that is 5% nitric acid and 2% hydrochloric acid.

9.2.3.12 Working Internal Standard Preparation

Dilute 5mL of yttrium stock standard (1000mg/L) and 10mL of lithium stock standard (10,000mg/L) to 1L with a reagent water solution that is 2% nitric acid for a final concentration of 5mg/L of yttrium and 100mg/L of lithium.

10. Calibration

10.1. Initial Calibration: Calibrate the ICP each working day according to the instrument manufacturer's recommended procedures. Flush the system with the Calibration Blank solution prepared by acidifying reagent water to the same concentrations of the acids found in the standards and samples. The calibration curve must consist of a minimum of a calibration blank and a standard.

10.2. Linear Calibration: Using the instrumentation software, prepare a standard curve for each element by plotting absorbance versus concentration. The analyst may employ a regression equation that does not pass through the origin. The regression will produce the slope and intercept terms for a linear equation in the form:

$$y = ax + b$$

where: y = instrument response (peak area)
a = slope of the line (the coefficient of x)
x = concentration of the calibration standard
b = intercept of the line

10.3. If a multi-point calibration is performed, the regression calculation will generate a correlation coefficient (r) that is the measure of the "goodness of fit" of the regression line to the data. In order to be used for quantitative purposes, the correlation coefficient must be ≥ 0.995 .

10.4. Initial Calibration Corrective Action: If the curve does not meet the acceptance criteria, then a new calibration curve must be analyzed. If the second curve attempt does not meet the acceptance criteria, the analyst must consult the department manager and instrument maintenance and/or preparation of new standards must be considered. Samples associated with a failed initial calibration must be reanalyzed.

- 10.5. Initial Calibration Verification (ICV):** In addition to meeting the linearity requirement, any new calibration curve must be assessed for accuracy in the values generated. To assess the accuracy, a single standard from a secondary source must be analyzed and the results obtained must be compared to the known value of the standard. This step is referred to as Initial Calibration Verification. The ICV is analyzed immediately following an initial calibration curve. Acceptable recovery range for the ICV is 90-110% and the RSD of replicate readings must be <5%.
- 10.6. ICV Corrective Action:** If the ICV is not acceptable, another ICV may be analyzed. If the second ICV fails, then a new initial calibration curve must be analyzed. Instrument maintenance and/or preparation of new standards must also be considered. Samples associated with a failed ICV must be reanalyzed. **Exception:** If the ICV is outside of the upper control limit, indicating high bias, associated samples determined to be <RL may be reported.
- 10.7. Initial Calibration Blank (ICB):** The ICB consists of a reagent water solution that is 5% HNO₃ and 2% HCl. An ICB must be analyzed immediately following the ICV. If the ICB result is above the reporting limit, another ICB may be analyzed. If the second ICB fails, then a new initial calibration curve must be analyzed. Samples associated with a failed ICB must be reanalyzed. **Exception:** If the ICB is >RL, associated samples determined to be <RL are reportable. **For BP, the ICB must be evaluated. If the absolute value of a negative concentration exceeds twice the established MDL, the ICB is considered to be unacceptable. Samples associated with a failed ICB must be re-analyzed unless the concentration of the target analyte is greater than 10 times the absolute value of the ICB result.**
- 10.9 Continuing Calibration Verification (CCV):** A CCV must be analyzed immediately following the ICB, after every 10 samples and at the end of the analytical batch to verify the system is still calibrated. The acceptable recovery range for the CCV is 90-110% and the RSD of replicate readings must be <5%.
- 10.10 CCV Corrective Action:** If a CCV fails the acceptance criteria, another CCV may be analyzed. If the second CCV fails, then a new initial calibration curve must be analyzed. Samples must be bracketed by acceptable CCVs in order to be reportable. Samples associated with a failed CCV must be reanalyzed. **Exception:** If the CCV is outside of the upper control limit, indicating high bias, associated samples determined to be <RL are reportable.
- 10.11 Contract Required Detection Limit (CRDL) Standard:** When specified by client or program requirements, a CRDL standard must be analyzed prior to sample analysis and at the end of each analytical batch to bracket the associated samples. Acceptance limits for all target elements is 50-150% recovery. This standard is required for BP samples only.
- 10.12 CRDL Corrective action:** BP Samples associated with a failed CRDL must be re-analyzed unless the concentration of the target has failed high, then the associated samples determined to be <RL are reportable
- 10.13 Interference Check Standard A (ICSA):** An ICSA must be analyzed at the beginning of each analytical run. ICSA must be 80-120% of the true value for the elements in the mix. Non-ICSA elements must be within +/-2x the reporting limit.
- 10.14 ICSA Corrective Action:** If the ICSA fails the acceptance criteria, another ICSA may be analyzed. If the second ICSA fails, then a new calibration curve must be analyzed. Instrument maintenance and/or preparation of new standards must also be considered. Samples associated with a failed ICSA must be reanalyzed. **Exception:** If the ICSA is >120% for any element in the mix or if any non-ICSA element is >2x the reporting limit, indicating high bias, associated samples determined to be <RL are reportable.
- 10.15 Interference Check Standard AB (ICSAB):** An ICSAB must be analyzed at the beginning of each analytical run. ICSAB must be 80-120% of the true value for the elements in the mix. **ICSAB is required for BP samples only.**

- 10.16 ICSAB Corrective Action:** If the ICSAB fails the acceptance criteria, another ICSAB may be analyzed. If the second ICSAB fails, then a new calibration curve must be analyzed. Instrument maintenance and/or preparation of new standards must also be considered. Samples associated with a failed ICSAB must be reanalyzed. **Exception:** If the ICSAB is >120% for any element in the mix, indicating high bias, associated samples determined to be <RL are reportable.
- 10.17 Continuing Calibration Blank (CCB):** The CCB consists of a reagent water solution that is 5% HNO₃ and 2% HCl. A CCB must be analyzed after every 10 samples following the CCV. If the CCB result is above the reporting limit, another CCB may be analyzed. If the second CCB fails, then a new calibration curve must be analyzed. Samples associated with a failed CCB must be reanalyzed. **Exception:** If the CCB is >RL, associated samples determined to be <RL are reportable. **For BP, the CCB must be evaluated. If the absolute value of a negative concentration exceeds twice the established MDL, the CCB is considered to be unacceptable. Samples associated with a failed CCB must be re-analyzed unless the concentration of the target analyte is greater than 10 times the absolute value of the CCB result.**

11 Procedures

- 11.1** Before using this procedure to analyze samples, there must be data available documenting initial demonstration of performance. The required data document the selection criteria of background correction points; analytical dynamic ranges; the applicable equations, and the upper limits of those ranges; the method and instrument detection limits; and the determination and verification of interelement correction equations or other routines for correcting spectral interferences. This data must be generated using the same instrument, operating conditions and calibration routine to be used for sample analysis.
- 11.2** Configure the ICP per manufacturer's instructions and allow it to become thermally stable.
- 11.3** Approximately 10mL portions of each standard, Method Blank, LCS, sample and MS/MSD are poured into autosampler tubes for analysis.
- 11.4** Establish initial calibration as described in Section 10.
- 11.5** Once initial calibration is established, analyze each sample, Method Blank, LCS and MS/MSD. An example sequence may be as follows:

Initial calibration blank
Mix 1
Mix 2
Mix 3
Mix 4
Mix 5
Mix 6
Mix 7
ICV
ICB
CRDL (if required)
ICSA
ICSAB (if required)
Method blank
LCS
Client samples
CCV
CCB
Client samples
CRDL (if required)

ICSA (if required)
ICSAB (if required)
CCV
CCB

- 11.6** The instrument performs two replicate readings for each analysis and the average of the two readings is used to derive the concentration. For samples, the difference between the two readings must be $\leq 20\%$ RSD for values that are $>4x$ the reporting limit. If the RSD is $>20\%$ for values that are $>4x$ the reporting limit, the sample must be reanalyzed.
- 11.7** Calculations for water samples are performed directly by the instrument software since initial sample aliquot and final digestate volumes are the same. If dilutions were performed, the appropriate factors must be applied to sample values.
- 11.8** The data system calculates the soil samples using the following equation:

$$C_s = \frac{C * V * D}{W}$$

Where: Cs = sample concentration (mg/kg, dry-weight basis)
C = concentration in extract (average of two readings taken)
V = volume of extract
D = dilution factor
W = dry weight of solid sample extracted (kg)

$$\text{Moisture corrected concentration} = \frac{(\text{Final concentration as received})}{(100 - \% \text{Moisture})} \times 100$$

- 11.9** Samples with analyte concentrations above the upper linear range must be diluted and reanalyzed or the over range results must be qualified as estimated.

12 Quality Control

12.1 Batch Quality Control

Table 12.1 – Batch Quality Control Criteria

| QA Sample | Components | Frequency | Acceptance Criteria | Corrective Action |
|---|--------------------------------|--|--|---|
| Method Blank (MB) | Reagent water or boiling chips | One per preparation batch of up to 20 samples, per matrix. | Target analyte must be less than reporting limits | Re-digest and reanalyze if target compound is >RL in method blank and associated samples. <u>Exceptions:</u> 1) If no additional sample remains for reanalysis or if reanalysis cannot take place within holding time, the reported method blank and samples must be qualified. 2) If a contaminant is present only in the method blank and not the samples, no action is required. |
| Laboratory Control Sample (LCS) | Applicable target analyte | One per preparation batch of up to 20 samples, per matrix. | 80-120% Recovery | Re-digest and reanalyze associated samples if original LCS is outside acceptance limits. <u>Exceptions:</u> 1) If no additional sample remains for reanalysis or if reanalysis cannot take place within holding time, reported data must be qualified. 2) If LCS recovery is >QC limits and sample results are non-detect, the sample data may be reported without qualifiers. The LCS data must be qualified. |
| Matrix Spike (MS)/Matrix Spike Duplicate (MSD) | Applicable target analyte | One MS/MSD set per preparation batch of up to 20 samples, per matrix. | 75-125% Recovery ≤20% RPD | No corrective actions necessary. If LCS recovery is in range, the system is considered in-control and the out-of-control MS/MSD must be qualified appropriately. |
| Internal Standard | Yttrium | Automatically added to each sample, blank, and standard as part of the analysis. | No acceptance criteria – used to monitor interferences. 60-140% | No corrective action required. Sample may be analyzed at a dilution if interference is indicated. |

12.2 LCS equation:

$$R = (C/S) * 100$$

Where R = percent recovery
C = spiked LCS concentration
S = concentration of analyte added to the clean matrix

12.3 MS/MSD equation:

$$R = \frac{(C_s - C)}{S} * 100$$

Where R = percent recovery
Cs = spiked sample concentration
C = sample concentration
S = concentration of analyte added to the sample

12.4 RPD equation:

$$RPD = \frac{|D_1 - D_2|}{[(D_1 + D_2)/2]} * 100$$

Where RPD = relative percent difference

D₁ = first sample result

D₂ = second sample result

- 12.5 Post-Digestion Spike Addition:** An analyte spike added to a portion of a prepared sample, or its dilution, should be recovered to within 75% to 125% of the known value. The spike addition should produce a minimum level of 10 times and a maximum of 100 times the instrument detection limit. If the spike is not recovered within the specified limits, a matrix effect should be suspected.
- 12.6 Dilution test:** If the analyte concentration is sufficiently high, minimally, a factor of 10 above the instrument detection limit after dilution, an analysis of a 1:5 dilution should agree within +/-10% of the original determination. If not, a chemical or physical interference effect should be suspected.

13 Method Performance

- 13.1 Method Detection Limit (MDL) Study:** An MDL study must be conducted every 12 months for each matrix per instrument.
- 13.2 Demonstration of Capability (DOC):** Every analyst who performs this method must first document acceptable accuracy and precision by passing a demonstration of capability study (DOC).
- 13.3 Linear Dynamic Range Study:** A linear dynamic range study must be conducted for each element by analyzing increasing concentrations of at least three, preferably five different concentration standards across the range. One of these should be near the upper limit of the range. The upper range limit should be an observed signal no more than 10% below the level extrapolated from lower standards. Samples determined to be above the upper range limit must be diluted and reanalyzed. New dynamic ranges should be determined whenever there is a significant change in instrument response. For those analytes that periodically approach the upper limit, the range should be checked every six months. Refer to Section 7.2.5.4 of Method 6010B for more information.
- 13.4 Interelement Correction Factors** must be verified and updated every 6 months or when an instrumentation change occurs. Refer to Section 3.1 of Method 6010B for more information.

14 Method Modifications

- 14.1** Mixed standard solutions are purchased as certified standards.
- 14.2** Instrument conditions may vary from those stated in the method.
- 14.3** Calibration blanks are evaluated to the reporting limit and not to three times the IDL.

15 Pollution Prevention and Waste Management

- 15.1** Procedures for handling waste generated during this analysis are addressed in S-IN-S-002, Waste Handling or other applicable SOP. All wastes are accumulated, managed and disposed of in accordance with all federal and state laws and regulations.
- 15.2** In order to minimize the amount of waste generated during this procedure, analyst should prepare reagents in an amount which may be used in a reasonable amount of time (i.e. before a reagent expires).
- 15.3** The company wide Chemical Hygiene and Safety Manual contains additional information on pollution prevention.

16 References

- 16.1** “Test Methods for Evaluating Solid Waste, Physical/Chemical Methods”, EPA SW-846, latest revision, Method 6010B.
- 16.2** Pace Analytical Quality Manual; latest revision.
- 16.3** TNI Standard; Quality Systems section; latest revision.

17 Tables, Diagrams, Flowcharts, Attachments, Appendices, etc.

- 17.1** Table 1: Target Metals and Reporting Limits

18 Revisions

| Document Number | Reason for Change | Date |
|------------------|--|-----------|
| S-IN-I-019-rev.7 | <ol style="list-style-type: none"> General: revised SOP references where applicable. Table 7.1 (aqueous): changed time period to 24 hours per updated 40CFR part 136 (2007). Table 12.1: added “or lab-generated limits” to LCS and MS limit description. Table 12.1: added line in chart for internal standards with acceptance limits. Table 12.1: added language to the ICSA requirements for non-ICSA elements (within +/-2X the RL). New Section 11.10: added requirement for one serial dilution per batch for BP samples. Section 10.3: added sentence about rerunning samples (to match other SOPs). Section 10.4: added “preceding or” before “following” (to match other SOPs). Added new section 10.6 to address BP requirement for calibration blanks. Section 11.8: added corrective action when CCV fails RPD requirement. Also added BP requirement to monitor RPD limits for samples and the corrective action to rerun samples. | 21Jan2008 |
| S-IN-I-019-rev.8 | <ol style="list-style-type: none"> Section 8.0: revised equipment and supplies section to a list from a table and added “or equivalent” to specific supply information. Section 9.0: removed 1:1 acid references and added “or equivalent” to specific reagent information. Table 9.2: changed Interference Check Standard to Interference Check Standard A (ICSA) and added ICSAB information Table 9.3: Added “or equivalent” to specific catalog numbers, changed Stock and Working Inter-Element Standard to Stock and Working Interference Check Standard A (ICSA), and added Stock and Working Interference Check Standard AB (ICSAB). Sections 9.2.3.7 and 9.2.3.8.: changed Stock and Working Inter-Element Correction Solution to Stock and Working Interference Check Standard A (ICSA). Sections 9.2.3.11 and 9.2.3.12.: added Stock and Working Interference Check Standard AB (ICSAB). Table 12.1: changed Inter-Element Correction Check to Interference Check Standard A (ICSA), added Interference Check Standard AB (ICSAB), and removed reference to lab-generated limits for LCS and MS/MSD. | 08May2009 |

| | | |
|-------------------|--|-----------|
| S-IN-I-019-rev.09 | <ol style="list-style-type: none"> 1. Section 2: streamlined summary of method 2. Section 3: removed SOP references and added a statement that weights, volumes, limits, etc... are subject to change. 3. Section 7: updated storage requirements, clarified hold time requirements and added requirements for dissolved metals. 4. Section 8: changed to a tabular format and updated information. Removed supplies relating to sample digestion. 5. Section 9: changed to a tabular format 6. Section 10: completely revised to clarify each QC sample and its corrective action, added calibration calculations and added Arsenic profiling criteria. 7. Section 11: removed table of ICP settings and added final concentration calculations. 8. Table 12.1: removed CCB, CRDL, ICSA, ICSAB and As profiling. Added RPD criteria for MS/MSD 9. Section 13: removed SOP references, added IDL information and expanded LDR information 10. Section 15: changed NELAC to TNI 11. Section 16: updated title of Attachment 1 12. Attachment 1: removed wavelengths and 200.7 information and update RLs. | 23Jun2011 |
| S-IN-I-019-rev.10 | <ol style="list-style-type: none"> 1. Section 3.1: added reference to MDLs. 2. Table 9.2: removed reference to Profiling Standard 3. Table 9.3: removed reference to Profiling Standard 4. Section 9.2.3: removed reference to Profiling Standard 5. Section 9.2.3.2: changed to a tabular format 6. Section 10: removed reference to Profiling Standard. 7. Section 11.9: added language that over range results can be reported if qualified as estimated. 8. Table 12.1: revised method blank corrective action. 9. Inserted new Method Modifications section. | 19Sep2012 |

Table 1: Target Metals and Reporting Limits

| Metals | Aqueous (µg/L) | Solid (mg/kg) |
|-----------------|---------------------------|--------------------------|
| Aluminum - Al | 1000 | 50 |
| Antimony - Sb | 6 | 2 |
| Arsenic - As | 10 | 2 |
| Barium - Ba | 100 | 2 |
| Beryllium - Be | 4 | 0.5 |
| Boron - B | 100 | 5 |
| Cadmium - Cd | 5 | 2 |
| Calcium - Ca | 1000 | 50 |
| Chromium - Cr | 10 | 2 |
| Cobalt - Co | 50 | 2 |
| Copper - Cu | 20 | 2 |
| Iron – Fe | 100 | 50 |
| Lead – Pb | 10 | 2 |
| Magnesium – Mg | 1000 | 50 |
| Manganese – Mn | 50 | 2 |
| Molybdenum - Mo | 50 | 2 |
| Nickel – Ni | 50 | 2 |
| Potassium - K | 1000 | 50 |
| Selenium - Se | 10 | 2 |
| Silver – Ag | 50 | 2 |
| Sodium – Na | 1000 | 50 |
| Thallium - Tl | 50 | 2 |
| Tin – Sn | 10 | 2 |
| Titanium - Ti | 50 | 2 |
| Vanadium - V | 50 | 2 |
| Zinc – Zn | 50 | 2 |



STANDARD OPERATING PROCEDURE

ACID DIGESTION OF SOLID SAMPLES FOR ICP ANALYSIS

REFERENCE METHOD: EPA SW-846 METHOD 3050B

SOP NUMBER: S-IN-I-031-rev.10
EFFECTIVE DATE: Date of Final Signature
SUPERSEDES: S-IN-I-031-rev.9

APPROVAL

Karl Anderson

General Manager

August 30, 2011

Date

Beck Schrage

Quality Manager

August 30, 2011

Date

Daniel D. Mark

Department Manager

August 30, 2011

Date

PERIODIC REVIEW

SIGNATURES BELOW INDICATE NO CHANGES HAVE BEEN MADE SINCE APPROVAL.

Signature

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1. Purpose

- 1.1** The purpose of this SOP is to provide a laboratory specific procedure for acid-digesting solid samples and wipes for metals analysis while meeting the requirements specified in EPA method 3050B. The digestates are then analyzed by inductively coupled plasma (ICP).

2. Summary of Method

- 2.1.** A sample is digested with nitric acid and hydrogen peroxide in a hot block digester. After the addition of hydrochloric acid, the samples are digested further, cooled, then brought to volume with reagent water.

3. Scope and Application

- 3.1.** This procedure is restricted to use by, or under the supervision of, analysts experienced in the use of metals digestion equipment. Each analyst must demonstrate the capability to generate acceptable results with this method to be considered qualified to report sample results.

4. Interferences

- 4.1.** Not applicable to this SOP.

5. Safety

5.1. Standards and Reagents

The toxicity and carcinogenicity of standards and reagents used in this method have not been fully defined. Each chemical compound should be treated as a potential health hazard. Reduce exposure by the use of gloves, lab coats and safety glasses. Material Safety Data Sheets (MSDSs) are on file in the laboratory and available to all personnel. Standard solutions should be prepared in a hood whenever possible. The stock metals standards are toxic and should be handled with extreme care. Also handle concentrated acids with care, making sure to wear appropriate personal protective equipment.

5.2. Samples

Take precautions when handling samples. Samples should always be treated as potentially hazardous "unknowns". The use of personal protective equipment such as gloves, lab coats and safety glasses is required when handling samples. In the event a sample container must be opened, it is recommended to perform this in a hood whenever possible. All digestions must be conducted under a fume hood.

5.3. Equipment

Portions of the preparation and analytical equipment operate at high temperatures. Care must be taken to minimize accidents and injuries when working on or with this equipment. Equipment should be turned off or the temperatures lowered to reduce the risk of thermal burns. Allow adequate time for the equipment to cool prior to working on equipment.

6. Definitions

- 6.1** Refer to Glossary section of the Pace Quality Assurance Manual for a comprehensive list of terms and definitions.

7. Sample Collection, Preservation, and Handling

Table 7.1 – Sample Collection, Preservation, Storage and Hold time.

| Sample type | Collection per sample | Preservation | Storage | Hold time |
|--------------|---|-----------------------------------|------------|--|
| Solid | 50 grams in glass or plastic container | No chemical preservation required | 0°C to 6°C | Must be analyzed within 6 months of the collection date. |
| Wipe | Individual wipe in glass or plastic container | No chemical preservation required | 0°C to 6°C | Must be analyzed within 6 months of the collection date. |

Samples should be stored separately from all standards, reagents, and highly contaminated samples. To avoid contamination, no food or drink products can be located near samples.

8. Equipment and Supplies

8.1. Equipment/Instrumentation

| Equipment | Vendor | Description / Comments |
|--------------------|-------------------------------------|--|
| Hot Block Digester | Environmental Express or equivalent | Adjustable and capable of maintaining a temperature of 90°C to 95°C. |
| Analytical Balance | Ohaus or equivalent | Capable of weighing to 0.01g |

8.2. General Supplies

| Item | Vendor | Description |
|----------------------|-------------------------------------|---|
| Volumetric Flasks | Class A | Various capacities |
| Volumetric Pipettors | Eppendorf or equivalent | Various sizes |
| Digestion Tubes | Environmental Express or equivalent | Volumetrically certified and contaminant free |
| Teflon Chips | Fisher or equivalent | For use as a clean solid matrix |
| Thermometer | Ever Safe or equivalent | Calibrated, used for monitoring Hot Block temperature |
| Plunger Filters | Environmental Express or equivalent | |
| Graduated Cylinders | Class A | Various capacities |
| pH strips | Fisher or equivalent | Full range |
| Ghost Wipes | Environmental Express or equivalent | Mixed cellulose ester wipe |

9. Reagents and Standards

9.1. Reagents

| Reagent | Concentration/ Description |
|-------------------|--|
| Reagent water | ASTM Type II |
| Nitric acid | Concentrated, trace metal analyzed or equivalent |
| Hydrochloric acid | Concentrated, trace metal analyzed or equivalent |
| Hydrogen Peroxide | 30% solution, trace metal analyzed or equivalent |

9.2. Analytical Standards

9.2.1. Definitions

Standards are required for initial calibration, calibration verification, second source verification, and for preparing LCS, MS, and MSD samples.

Table 9.2 Standard Definitions

| Standard | Description | Comments |
|-------------------|---|--|
| Spiking Standard | This solution contains the target analytes and is generally prepared using a standard source secondary to the standards used for calibration. | Same solution can be used for the LCS and MS/MSD |
| Internal Standard | A solution added all standards, samples, spikes, control samples, and method blanks prior to analysis. This standard is used to adjust response ratios to account for instrument drift. | Yttrium |

9.2.2. Storage Conditions

| Standard Type | Description | Expiration | Storage |
|--------------------------|---|--|---|
| Stock Spiking Standard | Inorganic Ventures; catalog #s PA-STD-1B; PA-STD-2B; PA-STD-3B, or equivalent | Manufacturer's recommended expiration date | Manufacturer's recommended storage conditions |
| Working Spiking Standard | Refer to Section 9.2.3.1. | Expires 6 months from date of preparation. | Same as stock standard |

Table 9.3 – Analytical Standard Storage Conditions

9.2.3. Preparation Procedures

Table 9.3 - Stock Spiking Standard Details

| Analyte | Concentration (mg/L) |
|---|----------------------|
| <i>Inorganic Ventures PA-STD1B</i> | |
| Arsenic | 200 |
| Barium | 200 |
| Beryllium | 200 |
| Cadmium | 200 |
| Cobalt | 200 |
| Chromium | 200 |
| Copper | 200 |
| Manganese | 200 |
| Nickel | 200 |
| Phosphorus | 200 |
| Lead | 200 |
| Selenium | 200 |
| Thallium | 200 |
| Lithium | 200 |
| Strontium | 200 |
| Vanadium | 200 |
| Zinc | 200 |
| <i>Inorganic Ventures PA-STD2B</i> | |
| Silicon | 1000 |
| Boron | 200 |
| Molybdenum | 200 |

| | |
|---|------|
| Antimony | 200 |
| Tin | 200 |
| Titanium | 200 |
| Zirconium | 200 |
| Silver | 100 |
| <i>Inorganic Ventures PA-STD3B</i> | |
| Aluminum | 2000 |
| Calcium | 2000 |
| Iron | 2000 |
| Potassium | 2000 |
| Magnesium | 2000 |
| Sodium | 2000 |

9.2.3.1. Working Spiking Standard Preparation

Dilute 50mL of each stock spiking standard (solutions 1B, 2B and 3B) and 4mL concentrated Nitric acid to 200mL with reagent water. One mL of this solution is used to spike the LCS, MS and MSD for 50mL initial volumes. For 15mL initial volumes use 0.3mL of this spike for LCS, MS and MSD.

10. Calibration

10.1. Not applicable to this SOP.

11. Procedures

- 11.1. Weigh 1g of well-mixed sample into a labeled digestion tube and add 5mL of reagent water and 5mL of concentrated nitric acid. Place the digestion tube into the Hot Block and set the temperature to achieve 95°C +/- 3°C in the digestion vessels. For ghost wipes, place the wipe in a labeled digestion tube and add 20mL of reagent water to completely cover the wipe. Add 5mL of concentrated nitric acid. Place the digestion tube into the Hot Block and set the temperature to achieve 95°C +/- 3°C in the digestion vessels.
- 11.2. Digest the sample until the volume is below 10mL but not dry. If brown fumes are generated, add 5mL of nitric acid and continue to digest, do not let the sample go dry. Continue adding nitric acid and digesting to below 10 mL until no brown fumes are given off, indicating a complete digestion.
- 11.3. Add 1mL of 30% hydrogen peroxide solution and warm slowly until effervescing stops. Continue to add 30% hydrogen peroxide solution in 1mL aliquots while warming until the effervescing is at a minimum or the general appearance of the sample is unchanged. Do not add more than 5mL total of the hydrogen peroxide solution.
- 11.4. Continue heating the acid –peroxide digestate until the volume has been reduced to approximately 5mL. Do not allow the digestate to go to dryness.
- 11.5. Allow the samples to cool. Add 5mL concentrated hydrochloric acid to each sample and warm the samples for another 15 minutes in order to dissolve any precipitate.
- 11.6. Adjust the volume to 50mL in the digestion tube with reagent water. Filter the samples to remove particulates using a plunger filter. The associated Method Blank and LCS must also be filtered.

- 11.7. Record all information including standard numbers, reagent numbers, digestion tube lot numbers, filter lot numbers, Hot Block number and digestion temperature in the metals digestion logbook and deliver the digestates to the ICP analyst.

12. Quality Control

12.1. Batch Quality Control

Table 12.1 – Batch Quality Control Criteria

| QA Sample | Components | Frequency | Acceptance Criteria | Corrective Action |
|---|---|---|---|---|
| Method Blank (MB) | 1g Teflon chips for solid samples or one unused ghost wipe for wipe samples | One per preparation batch of up to 20 samples, per matrix. | Target analyte must be less than reporting limits | Re-digest and reanalyze if target compound is >RL in method blank and associated samples. <u>Exceptions:</u> 1) If no additional sample remains for reanalysis or if reanalysis cannot take place within holding time, the reported method blank and samples must be qualified. 2) If a contaminant is present only in the method blank and not the samples, the sample data may be reported without qualifiers, the method blank must be qualified. |
| Laboratory Control Sample (LCS) | Applicable target analyte + 1g Teflon chips for solid samples or one unused ghost wipe for wipe samples | One per preparation batch of up to 20 samples, per matrix. | 80-120% Recovery | Re-digest and reanalyze associated samples if original LCS is outside acceptance limits. <u>Exceptions:</u> 1) If no additional sample remains for reanalysis or if reanalysis cannot take place within holding time, reported data must be qualified. 2) If LCS recovery is >QC limits and sample results are non-detect, the sample data may be reported without qualifiers. The LCS data must be qualified. |
| Matrix Spike (MS)/Matrix Spike Duplicate (MSD) | Applicable target analyte + sample | One MS/MSD set per preparation batch of up to 20 samples, per matrix. | 75-125% Recovery ≤20% RPD | No corrective actions necessary. If LCS recovery is in range, the system is considered in-control and the out-of-control MS/MSD must be qualified appropriately. |

12.2 Method Blank Preparation: For the Method Blank, add 1g of Teflon chips or one unused ghost wipe.

12.3 LCS Preparation: For LCS, add 1mL of the Working Spiking Standard plus 1g of Teflon chips or one unused ghost wipe.

12.4 Matrix Spike Preparation: Add 1mL of the Working Spiking Standard plus 1g of sample.

12.5 LCS calculation:

$$R = (C/S) * 100$$

Where R = percent recovery

C = spiked LCS concentration

S = concentration of analyte added to the clean matrix

12.6 MS/MSD calculation:

$$R = \frac{(C_s - C)}{S} * 100$$

Where R = percent recovery
Cs = spiked sample concentration
C = sample concentration
S = concentration of analyte added to the sample

12.7 RPD calculation:

$$RPD = \frac{|D_1 - D_2|}{[(D_1 + D_2)/2]} * 100$$

Where RPD = relative percent difference
D₁ = first sample result
D₂ = second sample result

12.8 Wipe Calculation:

$$\text{Total ug analyte per wipe} = (\text{analyte conc., ug/L}) \times (0.050 \text{ Liter/wipe})$$

13 Method Performance

- 13.2 Method Detection Limit (MDL) Study:** An MDL study must be conducted every 12 months for each matrix per instrument.
- 13.3 Demonstration of Capability (DOC):** Every analyst who performs this method must first document acceptable accuracy and precision by passing a demonstration of capability study (DOC).

14 Pollution Prevention and Waste Management

- 14.2** Procedures for handling waste generated during this analysis are addressed in S-IN-S-002, Waste Handling, or other applicable SOP.
- 14.3** In order to minimize the amount of waste generated during this procedure, analyst should prepare reagents in an amount which may be used in a reasonable amount of time (i.e. before a reagent expires)
- 14.4** The company wide Chemical Hygiene and Safety Manual contains additional information on pollution prevention.

15 References

- 15.2** "Test Methods for Evaluating Solid Waste, Physical/Chemical Methods", EPA SW-846, latest revision, Method 3050B.
- 15.3** Pace Analytical Quality Manual; latest revision.
- 15.4** TNI Standard; Quality Systems section; latest revision.

16 Tables, Diagrams, Flowcharts, Attachments, Appendices, etc.

- 16.2** Not applicable to this SOP.

17 Revisions

| Document Number | Reason for Change | Date |
|-------------------|--|-----------|
| S-IN-I-031-rev.8 | <ol style="list-style-type: none">1. Table 8.2, Table 12.1 and section 9.2.3.2: added wording about solid LCS material.2. Table 12.1: changed batch time from 12 hours to 8 hours.3. General: updated SOP references where applicable. Referenced 3050B as method revision.4. New section 11.2 added describing the process of additional nitric acid additions until no brown fumes are generated. | 21Jan2008 |
| S-IN-I-031-rev.9 | <ol style="list-style-type: none">1. Table of Contents: fixed to include Sections 10 and 11.2. Sections 8 and 9: revised to be in list form and removed catalog number references.3. Section 9.2.3.2: reworded for clarity.4. Table 9.3: changed working standard expiration to 6 months.5. Table 12.1: added 10x rule exception for Method Blank corrective action. | 04Jan2010 |
| S-IN-I-031-rev.10 | <ol style="list-style-type: none">1. Cover page revised to most recent format.2. Table 7.1: revised storage requirements and volume required and added wipes.3. Section 8: converted to a tabular format and updated information, added wipes.4. Section 9: converted to a tabular format and updated information.5. Section 11.1: added "well-mixed" and procedure for wipes6. Section 11.3: updated peroxide volumes added.7. Section 11.6: added LCS after method blank in last sentence.8. Section 11.7: added Hot Block number and digestion temperature as information to be recorded.9. Section 12: revised Table 12.1 and added preparation instructions for Method Blank, LCS and Matrix Spike.10. Section 13: removed SOP references.11. Section 15: changed NELAC to TNI. | 30Aug2011 |

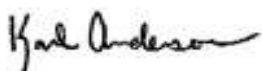
STANDARD OPERATING PROCEDURE

THE DETERMINATION OF MERCURY BY COLD VAPOR ATOMIC ABSORPTION SPECTROSCOPY

REFERENCE METHODS: EPA SW-846 METHODS 7470A AND 7471A

SOP NUMBER: S-IN-I-040-rev.13
EFFECTIVE DATE: Date of Final Signature
SUPERSEDES: S-IN-I-040-rev.12

APPROVAL



General Manager

November 2, 2012

Date



Quality Manager

October 30, 2012

Date



Department Manager

October 29, 2012

Date

PERIODIC REVIEW

SIGNATURES BELOW INDICATE NO CHANGES HAVE BEEN MADE SINCE APPROVAL.

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1. Purpose

- 1.1. The purpose of this SOP is to provide a laboratory specific procedure for determining total mercury concentration while meeting the requirements specified in EPA method 7470A for aqueous samples and method 7471A for solid samples.

2. Summary of Method

- 2.1. Prior to analysis, all samples are digested by heating with appropriate acids and oxidizing agents to dissolve and oxidize mercury contents.
- 2.2. This cold-vapor method is based on the absorption of radiation at 253.7nm by mercury vapor. The mercury is reduced to the elemental state and aerated from solution in a closed system. The mercury vapor passes through a cell positioned in the light path of an atomic absorption spectrophotometer. Absorbance (peak height) is measured as a function of mercury concentration.

3. Scope and Application

- 3.1. This method is applicable for the measurement of mercury in groundwater, surface and saline waters, domestic and industrial wastes, TCLP extracts, soil, sediment, bottom deposits and sludge-type materials.
- 3.2. The reporting limits for mercury are 0.002 mg/L for aqueous samples and 0.33 mg/Kg for solid samples. Refer to the LIMS for method detection limits.
- 3.3. Reporting limits, control limits, volumes/weights used, standard concentrations, vendors, instrumentation, equipment and supplies are subject to change.
- 3.4. This procedure is restricted to use by, or under the supervision of, analysts experienced in the use of mercury analysis equipment and reagents. Each analyst must demonstrate the capability to generate acceptable results with this method to be considered qualified to report sample results.

4. Interferences

- 4.1. High concentrations of sulfide may interfere in some water or solid samples. Potassium permanganate is added during digestion to eliminate sulfide interference. Concentrations as high as 20mg/L in water or 20mg/kg in soils have been demonstrated to cause no interference in spiked samples.
- 4.2. High concentrations of copper have been reported to interfere with mercury determinations. Concentrations as high as 10mg/L in water or 10mg/kg in soil have been demonstrated to cause no interference in spiked samples.
- 4.3. High concentrations of chloride, present in samples require additional potassium permanganate. The free chlorine produced during digestion should be removed with excess hydroxylamine hydrochloride solution.

5. Safety

5.1. Standards and Reagents

The toxicity and carcinogenicity of standards and reagents used in this method have not been fully defined. Each chemical compound should be treated as a potential health hazard. The use of gloves, lab coats and safety glasses is required. Material Safety Data Sheets (MSDSs) are on file in the laboratory and available to all personnel. Standard solutions should be prepared in a hood whenever possible.

5.2. Samples

Take precautions when handling samples. Samples should always be treated as potentially hazardous “unknowns”. The use of personal protective equipment such as gloves, lab coats and safety glasses is required when handling samples. In the event a sample container must be opened, it is recommended to perform this in a hood whenever possible. All distillations should be conducted under a fume hood.

6. Definitions

- 6.1. Refer to Glossary section of the Pace Quality Assurance Manual (QAM) for a comprehensive list of terms and definitions.

7. Sample Collection, Preservation, and Handling

Table 7.1 – Sample Collection, Preservation, Storage and Hold time.

| Sample type | Collection per sample | Preservation | Storage | Hold time |
|----------------------------|-------------------------------|---|------------|---|
| Aqueous - Total | 500 mL in plastic container. | HNO ₃ to pH<2 Samples received at pH>2 must be preserved to pH<2 with HNO ₃ and be allowed to equilibrate for 24 hours before being prepared for analysis. | Ambient | Analysis must be completed within 28 days of collection date. |
| Aqueous - Dissolved | 500 mL in plastic container | Filter; HNO ₃ to pH<2 | Ambient | Analysis must be completed within 28 days of collection date. |
| Solid | 100g in a 4oz glass container | None | 0°C to 6°C | Analysis must be completed within 28 days of collection date. |

Samples must be stored separately from all standards, reagents, and highly contaminated samples. To avoid contamination, no food or drink products can be located near samples.

8. Equipment and Supplies

8.1. Equipment/Instrumentation

| Equipment | Vendor | Model / Version | Description / Comments |
|----------------------------|-----------------------|-----------------------|---|
| Automated Mercury Analyzer | CETAC | M-7500 or equivalent | To include an atomic absorption spectrophotometer, mercury lamp, absorption cell, air pump, flow meter, drying tube, autosampler and data system. |
| Hot Block | Environmental Express | 56-well or equivalent | Adjustable and capable of maintaining a temperature of 90°C to 95°C. |
| Balance | Ohaus | GT400 or equivalent | Readability to 0.01g |

8.2. General Supplies

| Item | Vendor | Description |
|--------------------|-------------------------------------|---|
| Auto-pipettes | Eppendorf or equivalent | Various sizes |
| Volumetric flasks | Class A | 100mL |
| Graduated cylinder | Class A | 25mL |
| Digestion cups | Environmental Express or equivalent | 50mL capacity, volumetrically certified |
| Autosampler tubes | Moldpro, Inc or equivalent | 17x100 mm |

9. Reagents and Standards

9.1. Reagents

| Reagent | Concentration/ Description |
|---|---|
| Reagent water | ASTM Type II |
| Sulfuric acid | Concentrated, trace metal grade or equivalent |
| Hydrochloric acid | Concentrated, trace metal grade or equivalent |
| Hydrochloric acid (3%) | Dilute 30 mLs of concentrated HCl to 1L with reagent water. Used for dilution preparation. |
| Nitric acid | Concentrated, trace metal grade or equivalent |
| Aqua Regia | Carefully add one volume of nitric acid to three volumes of hydrochloric acid. Must be prepared in a hood and must be prepared immediately before use on each day. |
| Stannous Chloride solution | Add 100g stannous chloride and 70 mL conc. HCl in reagent water and dilute to 1L. This solution is good for 3 days. Refrigerate when not in use. |
| Sodium Chloride | Granular |
| Hydroxylamine hydrochloride | Granular |
| Sodium Chloride/ Hydroxylamine Hydrochloride solution | Dissolve 120g sodium chloride and 120g hydroxylamine hydrochloride in reagent water and dilute to 1L. This solution is good for 6 months from preparation (hydroxylamine sulfate may be substituted for hydroxylamine hydrochloride). |
| Potassium permanganate solution (5%) | Commercially purchased Mercury-free, 5% solution (w/v) |
| Potassium persulfate | Granular |
| Potassium persulfate solution | Dissolve 5g Potassium persulfate in reagent water and dilute to 100mL. |
| Rinse/Probe Wash Solution | Add 50mL HCL and 20mL HNO ₃ to 500mL reagent water and dilute to 1L. |
| Boiling chips | Or equivalent to be used as a simulated soil matrix. |

9.2. Analytical Standards

9.2.1. Definitions

Standards are required for initial calibration, calibration verification standards, second source verification, and for preparing LCS, MS, and MSD samples.

Table 9.2 Standard Definitions

| Standard | Description | Comments |
|--|---|--|
| Initial Calibration Standards | Standards prepared at varying levels to determine response and retention characteristics of instrument | |
| Initial Calibration Verification Standard | A standard prepared from a source other than that used for the initial calibration. This standard verifies the accuracy of the calibration curve. | ICV |
| Reporting Limit Verification Standard | A standard prepared at a concentration equivalent to the reporting limit for verification at that level. | RLVS |
| Continuing Calibration Verification Standard | A calibration standard prepared at mid-level concentration for all target compounds. This standard is used to verify the initial calibration. | CCV |
| Spiking Standard | This solution contains the target analyte and is used to spike MS/MSD sets. | Same solution can be used for both the LCS and MS/MSD. |

9.2.2. Storage Conditions

Table 9.3 – Analytical Standard Storage Conditions

| Standard Type | Description | Expiration | Storage |
|--|---|---|---|
| Stock Mercury Calibration standard | Fisher; catalog # SM114-500; 1000mg/L or equivalent | Manufacturer's recommended expiration date | Manufacturer's recommended storage conditions |
| Intermediate #1 Mercury Calibration standard | Refer to Section 9.2.3.1 | Solution expires 6 months from date of preparation. | Same as for stock standard. |
| Intermediate #2 Mercury Calibration standard | Refer to Section 9.2.3.1 | Must be prepared fresh daily. | Not Applicable |
| Working Mercury Calibration standards | Refer to Section 9.2.3.2 | One-time use standards. | Not Applicable |
| Stock Mercury ICV/Spiking standard | SPEX; catalog # PLHG4-2Y; 1000mg/L or equivalent | Manufacturer's recommended expiration date | Manufacturer's recommended storage conditions |
| Intermediate #1 Mercury ICV/Spiking standard | Refer to Section 9.2.3.3 | Solution expires 6 months from date of preparation. | Same as for stock standard. |
| Intermediate #2 Mercury ICV/Spiking standard | Refer to Section 9.2.3.3 | Must be prepared fresh daily. | Not Applicable |
| Working Mercury ICV standard | Refer to Section 9.2.3.4 | One-time use standard. | Not Applicable |
| Working RLVS standard | Refer to Section 9.2.3.2 | One-time use standard. | Not Applicable |

9.2.3. Standard Preparation Procedures

Refer to the standard preparation logbook or database for specific instructions regarding preparation of standards for Mercury analysis

9.2.3.1 Intermediate Mercury Calibration Standard Preparation

Intermediate #1 Mercury Calibration Standard: Dilute 1mL of the Stock Mercury Calibration Standard (1000mg/L) to 100mLs with 2% HNO₃ for a final concentration of 10mg/L. This standard is good for 6 months from the date of preparation.

Intermediate #2 Mercury Calibration Standard: Dilute 1mL of the Intermediate #1 Mercury Calibration Standard (10mg/L) to 100mLs with 2% HNO₃ for a final concentration of 100ug/L. This standard must be prepared fresh daily.

9.2.3.2 Working Mercury Calibration Standards Preparation

Working calibration standards are one-time use and are prepared by diluting the Intermediate #2 Mercury Calibration Standard with reagent water. Examples of possible calibration standards are as follows:

Aqueous:

| Standard ID | Amount of Int. Std. #2 | Final Volume in reagent water | Final Concentration |
|-------------------|------------------------|-------------------------------|---------------------|
| Standard 1 (RLVS) | 0.06mL | 30mL | 0.2ug/L |
| Standard 2 | 0.3mL | 30mL | 1.0ug/L |
| Standard 3 | 0.6mL | 30mL | 2.0ug/L |
| Standard 4 (CCV) | 1.5mL | 30mL | 5.0ug/L |
| Standard 5 | 2.25mL | 30mL | 7.5ug/L |
| Standard 6 | 3.0mL | 30mL | 10.0ug/L |

Solid:

| Standard ID | Amount of Int. Std. #2 | Final Volume in reagent water | Final Concentration |
|-------------------|------------------------|-------------------------------|---------------------|
| Standard 1 (RLVS) | 0.1mL | 50mL | 0.2ug/L |
| Standard 2 | 0.5mL | 50mL | 1.0ug/L |
| Standard 3 | 1.0mL | 50mL | 2.0ug/L |
| Standard 4 (CCV) | 2.5mL | 50mL | 5.0ug/L |
| Standard 5 | 3.75mL | 50mL | 7.5ug/L |
| Standard 6 | 5.0mL | 50mL | 10.0ug/L |

9.2.3.3 Intermediate Mercury ICV/Spiking Standard Preparation

Intermediate #1Mercury ICV Standard: Dilute 1mL of the Stock Mercury ICV/Spiking Standard (1000mg/L) to 100mLs with 2% HNO₃ for a final concentration of 10mg/L. This standard is good for 6 months from the date of preparation.

Intermediate #2Mercury ICV Standard: Dilute 1mL of the Intermediate #1 Mercury ICV/Spiking Standard (10mg/L) to 100mLs with 2% HNO₃ for a final concentration of 100ug/L. This standard must be prepared fresh daily and is also used to prepare the LCS and MS/MSD.

9.2.3.4 Working Mercury ICV/LCS Standard Preparation

Aqueous: Dilute 1.5mL of the Intermediate #2 Mercury ICV Standard to 30mL with reagent water for a standard concentration of 5.0ug/L. This standard is a one-time use standard and is also used as the LCS.

Solid: Dilute 1.5mL of the Intermediate #2 Mercury ICV Standard to 50mL with reagent water for a standard concentration of 3.0ug/L. This standard is a one-time use standard and is also used as the LCS.

10. Calibration

10.1. Initial Calibration: A minimum of 5 calibration standards is required. The lowest calibration standard must be at or below the reporting limit. A new initial calibration curve with freshly prepared standard is analyzed on each working day. Refer to the Quality Manual for more information regarding calibration curves.

10.2. Linear Calibration: Using the instrumentation software, prepare a standard curve by plotting absorbance versus mercury concentration of each calibration standard. The analyst may employ a regression equation that does not pass through the origin. The regression will produce the slope and intercept terms for a linear equation in the form:

$$y = ax + b$$

where: y = instrument response (peak area)
a = slope of the line (the coefficient of x)
x = concentration of the calibration standard
b = intercept of the line

10.3. The regression calculation will generate a correlation coefficient (r) that is the measure of the “goodness of fit” of the regression line to the data. In order to be used for quantitative purposes, the correlation coefficient must be ≥ 0.995 .

$$r = \frac{\sum_{i=1}^N (X_i - \bar{X}) * (Y_i - \bar{Y})}{\sqrt{\left(\sum_{i=1}^N (X_i - \bar{X})^2 \right) * \left(\sum_{i=1}^N (Y_i - \bar{Y})^2 \right)}}$$

- 10.4. Initial Calibration Corrective Action:** If the curve does not meet the acceptance criteria, then a new calibration curve must be digested analyzed. If the second curve attempt does not meet the acceptance criteria, the analyst must consult the department manager and instrument maintenance and/or preparation of new standards must be considered. Samples associated with a failed initial calibration must be reanalyzed. Refer to Section 10.13 for additional information.
- 10.5. Initial Calibration Verification (ICV):** In addition to meeting the linearity requirement, any new calibration curve must be assessed for accuracy in the values generated. To assess the accuracy, a single standard from a secondary source must be analyzed and the results obtained must be compared to the known value of the standard. This step is referred to as Initial Calibration Verification. The ICV is analyzed immediately following an initial calibration curve. Acceptable recovery range for the ICV is 90-110%.
- 10.6. ICV Corrective Action:** If the ICV is not acceptable, another ICV may be analyzed. If the second ICV fails, then a new initial calibration curve must be analyzed. Instrument maintenance and/or preparation of new standards must also be considered. Samples associated with a failed ICV must be reanalyzed. **Exception:** If the ICV is outside of the upper control limit, indicating high bias, associated samples determined to be <RL may be reported. Refer to Section 10.13 for additional information.
- 10.7. Initial Calibration Blank (ICB):** The ICB consists of reagent water that is prepared per Section 11. An ICB must be analyzed immediately following the ICV. If the ICB result is above the reporting limit, the ICB may be reanalyzed. If the second ICB fails, then a new initial calibration curve must be analyzed. Samples associated with a failed ICB must be reanalyzed. **Exception:** If the ICB is >RL, associated samples determined to be <RL are reportable. Refer to Section 10.13 for additional information.
- 10.8. Continuing Calibration Verification (CCV):** A CCV must be analyzed after every 10 samples and at the end of the analytical batch to verify the system is still calibrated. The CCV should be from the same material as the curve standards. The acceptable recovery range for the CCV is 90-110%.
- 10.9. CCV Corrective Action:** If a CCV fails the acceptance criteria, another CCV may be analyzed. If the second CCV fails, then a new initial calibration curve must be analyzed. Samples must be bracketed by acceptable CCVs in order to be reportable. Samples associated with a failed CCV must be reanalyzed. **Exception:** If the CCV is outside of the upper control limit, indicating high bias, associated samples determined to be <RL are reportable. Refer to Section 10.13 for additional information.
- 10.10. Continuing Calibration Blank (CCB):** The CCB consists of reagent water that is prepared per Section 11. A CCB must be analyzed after each CCV. If the CCB result is above the reporting limit, the CCB may be reanalyzed. If the second CCB fails, then a new calibration curve must be analyzed. Samples associated with a failed CCB must be reanalyzed. **Exception:** If the CCB is >RL, associated samples determined to be <RL are reportable. Refer to Section 10.13 for additional information.
- 10.11. Reporting Limit Verification Standard (RLVS):** The RLVS is a check standard at or below the concentration of the reporting limit. The RLVS must be analyzed at the beginning of an analytical run, after every 20 samples, and at the end of the analytical run. The acceptable recovery range for the RLVS is 50-150%.

10.12. RLVS Corrective Action: If an RLVS fails the acceptance criteria, another RLVS may be analyzed. If the second RLVS fails, then a new initial calibration curve must be analyzed. Sample associated with a failed RLVS must be reanalyzed. **Exception:** If the RLVS is outside of the upper control limit, indicating high bias, associated samples determined to be <RL are reportable. Refer to Section 10.13 for additional information.

10.13. Failure of the initial calibration, ICV, CCV, ICB or CCB that is due to improper or inadequate preparation requires the re-digestion and reanalysis of the associated preparation batch(es). Failure of the initial calibration, ICV, CCV, ICB or CCB due to instrument malfunction requires the instrument to be restored to proper working order and the reanalysis of samples associated with the failed QC.

11. Procedures

11.1. Aqueous Sample Preparation

- 11.1.1** Transfer a 30mL aliquot of well-mixed sample to a 50mL graduated digestion cup. Prepare an MS and MSD set by transferring 30mL aliquots of well-mixed sample to separate digestion cups and adding 1.5mL of the Intermediate #2 Mercury ICV/Spiking Standard (100ug/L) for a spike concentration of 5.0ug/L.
- 11.1.2** Prepare a Method Blank by adding 30mL of reagent water to a digestion cup. Prepare an LCS by adding 1.5mL of the Intermediate #2 Mercury ICV/Spiking Standard (100ug/L) to a digestion cup and diluting to 30mL with reagent water for a spike concentration of 5.0ug/L.
- 11.1.3** Add 0.75mL concentrated nitric to each digestion cup then add 1.5mL concentrated sulfuric acid to each digestion cup, mixing after each addition.
- 11.1.4** Add 5mL potassium permanganate solution to each digestion cup. Ensure that equal amounts of permanganate solution are added to Method Blank and LCS. Swirl to mix and add additional portions of permanganate solution, up to 15mL if necessary, until the purple color persists for at least 15 minutes. If the purple color does not persist after addition of 15mL of solution, then start over at Section 11.1.1 using a diluted aliquot of sample.
- 11.1.5** Add 2.5mL potassium persulfate solution to each digestion cup, cap loosely and heat samples for 2 hours in the Hot Block at 95°C.
- 11.1.6** Cool samples and add 1.8mL of the sodium chloride/hydroxylamine hydrochloride solution to each sample to reduce the excess potassium permanganate. **CAUTION:** perform this addition in a fume hood, as chlorine gas could be produced. Proceed to Section 11.4.

11.2. Solid Sample Preparation

- 11.2.1** Weigh 0.3g of sample into a 50mL digestion cup. To ensure the sample is representative of the entire container, the analyst should weigh out three 0.1g aliquots from different parts of the same container. Prepare an MS and MSD by weighing 0.3g portions of a sample into separate digestion cups and adding 1.5mL of the Intermediate #2 Mercury ICV/Spiking Standard (100ug/L) for a spike concentration of 0.5mg/Kg.
- 11.2.2** Prepare a Method Blank by placing several boiling chips in a digestion cup. Prepare an LCS by placing several boiling chips in a digestion cup and adding 1.5mL of the Intermediate #2 Mercury ICV/Spiking Standard (100ug/L) for a final concentration of 0.5mg/Kg.
- 11.2.3** Add 5mL of reagent water to each digestion cup.

- 11.2.4 Add 2.5mL of aqua regia to each digestion cup.
- 11.2.5 Heat samples for 2 minutes in the Hot Block at 95°C.
- 11.2.6 Cool samples and add 25mL reagent water then add 7.5mL potassium permanganate solution. Loosely cap each digestion cup.
- 11.2.7 Return the samples to the Hot Block and heat for 30 minutes at 95°C.
- 11.2.8 Cool samples again and add 3mL of the sodium chloride/hydroxylamine hydrochloride solution to each sample to reduce the excess potassium permanganate. **CAUTION:** perform this addition in a fume hood, as chlorine gas could be produced.
- 11.2.9 Adjust the digestate volumes to 50mL with reagent water and mix. Proceed to Section 11.4.

11.3. Calibration Standard Preparation

- 11.3.1 Prepare calibration standards in 50mL digestion cups per the instructions in Section 9.2.3.2.
- 11.3.2 Follow steps 11.1.3 through 11.1.6 to prepare calibration standards for aqueous matrix.
- 11.3.3 Follow steps 11.2.4 through 11.2.8 to prepare calibration standards for solid matrix.

11.4. Determination of Mercury

- 11.4.1 Configure the mercury analyzer according to manufacturer's instructions. Allow the colorimeter and recorder to warm up. Run a baseline with all reagents, using reagent water to flush the tubing. Whenever new tubing is used, allow ample time to flush the tubing.
- 11.4.2 Approximately 10mL portions of each standard, Method Blank, LCS, sample and MS/MSD are poured into autosampler tubes for analysis.
- 11.4.3 Establish initial calibration as described in Sections 10.1 through 10.5.
- 11.4.4 Once initial calibration is established, analyze each sample, Method Blank, LCS and MS/MSD. An example sequence may be as follows:

Initial calibration standards

ICV

ICB

RLVS

Method blank

LCS

Client samples

CCV

CCB

Client samples

CCV

CCB

RLVS

- 11.5 Calculations are performed directly by the instrument software. If dilutions were performed, the appropriate factors must be applied.
- 11.6 The instrument software calculates the amount of Mercury in the sample aliquot as follows:

$$X_s = (y - b)/a$$

Where: X_s = Concentration of the analyte
 y = Total area or response of the analyte
 a = slope of the line (the coefficient of x)
 b = intercept of the line

11.7 Calculate the final concentration in the sample as follows:

$$\text{Aqueous Sample (mg/L)} = \frac{(X_s)(V_f)(D)}{(V_i)}$$

$$\text{Solid Sample (mg/Kg)} = \frac{(X_s)(V_f)(D)}{(W_s)}$$

Where: X_s = Mercury concentration
 V_f = Final sample volume in milliliters
 D = Dilution factor
 V_i = Initial sample volume in milliliters
 W_s = Weight of solid sample extracted in grams

$$\text{Moisture corrected concentration} = \frac{(\text{Final concentration as received})}{(100 - \% \text{Moisture})} \times 100$$

11.8 Any sample with a mercury concentration that exceeds the linear range of the calibration curve must be diluted with 3% HCl solution and re-analyzed or over range results must be qualified as estimated.

12 Quality Control

12.1 Batch Quality Control

Table 12.1 – Batch Quality Control Criteria

| QA Sample | Components | Frequency | Acceptance Criteria | Corrective Action |
|---|--------------------------------|---|---|--|
| Method Blank (MB) | Reagent water or boiling chips | One per preparation batch of up to 20 samples, per matrix. | Target analyte must be less than reporting limits | Re-digest and re-analyze if target compound is >RL in method blank and associated samples. <u>Exceptions:</u> 1) If no additional sample remains for reanalysis or if reanalysis cannot take place within holding time, the reported method blank and samples must be qualified. 2) If a contaminant is present only in the method blank and not the samples, no action is required. |
| Laboratory Control Sample (LCS) | Applicable target analyte | One per preparation batch of up to 20 samples, per matrix. | 80-120% Recovery | Re-digest and re-analyze associated samples if original LCS is outside acceptance limits. <u>Exceptions:</u> 1) If no additional sample remains for reanalysis or if reanalysis cannot take place within holding time, reported data must be qualified. 2) If LCS recovery is >QC limits and sample results are non-detect, the sample data may be reported without qualifiers. The LCS data must be qualified. |
| Matrix Spike (MS)/Matrix Spike Duplicate (MSD) | Applicable target analyte | One MS/MSD set per preparation batch of up to 20 samples, per matrix. | 75-125% Recovery ≤20% RPD | No corrective actions necessary. If LCS recovery is in range, the system is considered in-control and the out-of-control MS/MSD must be qualified appropriately. |

12.2 LCS equation:

$$R = (C/S) * 100$$

Where R = percent recovery
C = spiked LCS concentration
S = concentration of analyte added to the clean matrix

12.3 MS/MSD equation:

$$R = \frac{(C_s - C)}{S} * 100$$

Where R = percent recovery
Cs = spiked sample concentration
C = sample concentration
S = concentration of analyte added to the sample

12.4 RPD equation:

$$RPD = \frac{|D_1 - D_2|}{[(D_1 + D_2)/2]} * 100$$

Where RPD = relative percent difference
D₁ = first sample result
D₂ = second sample result

13 Method Performance

- 13.1 Method Detection Limit (MDL) Study:** An MDL study must be conducted annually for each matrix per instrument.
- 13.2 Demonstration of Capability (DOC):** Every analyst who performs this method must first document acceptable accuracy and precision by passing a demonstration of capability study (DOC).

14 Method Modifications

- 14.1** Digestion procedure modified to use digestion cups in a hot block instead of BOD bottles in a water bath.
- 14.2** Standards and some reagents purchased as certified solutions.
- 14.3** Stannous Chloride solution not stirred continually because it is a solution and not a suspension.

15 Pollution Prevention and Waste Management

- 15.1** Procedures for handling waste generated during this analysis are addressed in S-IN-S-002, Waste Handling, or other applicable SOP. All wastes are accumulated, managed and disposed of in accordance with all federal and state laws and regulations.
- 15.2** In order to minimize the amount of waste generated during this procedure, analyst should prepare reagents in an amount which may be used in a reasonable amount of time (i.e. before a reagent expires)
- 15.3** The company wide Chemical Hygiene and Safety Manual contains additional information on pollution prevention.

16 References

- 16.1** Test Methods for Evaluating Solid Waste, Physical/Chemical Methods, SW-846, Methods 7470A and 7471A .
- 16.2** Pace Analytical Quality Manual; latest revision.
- 16.3** TNI Standard; Quality Systems section; latest revision.

17 Tables, Diagrams, Flowcharts, Attachments, Appendices, etc.

- 17.1** Not Applicable

18 Revisions

| Document Number | Reason for Change | Date |
|-------------------|--|-----------|
| IN-I-040-rev.9 | <ol style="list-style-type: none"> 1. Restructured document format to new corporate template. 2. Changed several sections of information to tabular form. 3. Section 9.1: revised vendor for potassium permanganate solution. 4. Section 9.2.3: intermediate standards are good for 6 months. 5. Section 9.2.3.2: revised working standard preparation directions. 6. Section 9.2.3.4: revised ICV preparation directions. 7. Added section 10.6 (low level check standard). 8. Table 12.1: revised language about frequency of blanks. | 31Jan2007 |
| S-IN-I-040-rev.10 | <ol style="list-style-type: none"> 1. Added new section 10.2 regarding how and when to drop points from an initial calibration curve. 2. Section 10.4: added last line regarding rerunning of samples. 3. Section 10.5: added "preceding or". 4. General: revised SOP references. | 15Nov2007 |
| S-IN-I-040-rev.11 | <ol style="list-style-type: none"> 1. Cover page converted to periodic review format. 2. Table of Contents fixed to include sections 10 and 11. 3. Section 3: added language that limits, volumes, vendors, are subject to change. 4. Table 7.1: updated to clarify hold time. 5. Section 8: updated and added "or equivalent" where applicable. 6. Section 9: updated and clarified separate standards as intermediate and working. Created a table for working calibration standard examples. 7. Section 10: completely revised to individually clarify calibration requirement including corrective actions. 8. Section 11: clarified procedure including intended run sequence and added calculations for final concentration. 9. Section 12: updated limits and corrective actions. 10. Section 15: changed NELAC to TNI. | 19Jun2011 |
| S-IN-I-040-rev.12 | <ol style="list-style-type: none"> 1. Section 3.2: added reference to MDLs and revised RL for solids. 2. Section 9.1: added potassium persulfate, wash solution and boiling chips. Revised or expanded other reagents. 3. Table 9.2: added RLVS 4. Table 9.3: added RLVS 5. Section 9.2.3.2: added RLVS 6. Section 10: added RLVS 7. Section 11.1.5: added that digestion cups be capped loosely for digestion. 8. Section 11.4.4: added RLVS 9. Section 11: added that calculations are performed by instrument software. 10. Section 11.8: added that over range results must be qualified. 11. Table 12.1: revised method blank corrective action. 12. Inserted new Method Modifications section. | 27Sep2012 |
| S-IN-I-040-rev.13 | <ol style="list-style-type: none"> 1. Table 9.1: revised details of Stannous Chloride reagent use and handling. 2. Section 14: added a modification for no continuous stirring of Stannous Chloride. | 29Oct2012 |



Pace Analytical Services, Inc.

7726 Moller Road
Indianapolis, IN 46268

Phone: 317.875.5894

Fax: 317.872.6189

STANDARD OPERATING PROCEDURE

THE DETERMINATION OF HEXAVALENT CHROMIUM

REFERENCE METHODS: EPA SW-846 METHOD 7196A AND STANDARD METHODS 3500-Cr B

SOP NUMBER: S-IN-I-063-rev.11
EFFECTIVE DATE: Date of Final Signature
SUPERSEDES: S-IN-I-063-rev.10

APPROVAL

General Manager

September 27, 2012

Date

Quality Manager

September 26, 2012

Date

Department Manager

September 26, 2012

Date

PERIODIC REVIEW

SIGNATURES BELOW INDICATE NO CHANGES HAVE BEEN MADE SINCE APPROVAL.

Signature

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1. Purpose

- 1.1 The purpose of this SOP is to provide a laboratory specific procedure for determining Hexavalent Chromium in aqueous and solid samples while meeting the requirements specified in EPA SW-846 method 7196A and Standard Methods 3500-Cr B. **NOTE: Standard Methods 3500-Cr B supersedes and is equivalent to Standard Methods 3500-Cr D.**

2. Summary of Method

- 2.1 Dissolved hexavalent chromium is determined colorimetrically by reaction when diphenylcarbazide in an acid solution. A red-violet color is produced and is measured colorimetrically at 540nm.

3. Scope and Application

- 3.1 This method is applicable for the measurement of hexavalent chromium in groundwater, surface water, soil, sediment and domestic and industrial wastes. Refer to SOP S-IN-I-070 Alkaline Digestion of Solid Samples for Hexavalent Chromium or its replacement for procedures associated with the preparation of solid samples.
- 3.2 The reporting limit for Hexavalent Chromium is 0.01mg/L for aqueous samples and 4mg/kg for solid samples. Refer to the LIMS for method detection limits.
- 3.3 Reporting limits, control limits, volumes/weights used, standard concentrations, vendors, instrumentation, equipment and supplies are subject to change.
- 3.4 This procedure is restricted to use by, or under the supervision of, analysts experienced in the use of hexavalent chromium analysis equipment and reagents. Each analyst must demonstrate the capability to generate acceptable results with this method to be considered qualified to report sample results.

4. Interferences

- 4.1 The chromium reaction with the diphenylcarbazide is usually free from interferences. However, hexavalent molybdenum and mercury salts also react to form color complexes with the reagent. Vanadium can also interfere, but concentrations up to 10 times that of chromium can be tolerated.

5. Safety

- 5.1 **Standards and Reagents:** The toxicity and carcinogenicity of standards and reagents used in this method have not been fully defined. Each chemical compound should be treated as a potential health hazard. Reduce exposure by the use of gloves, lab coats and safety glasses. Material Safety Data Sheets (MSDSs) are on file in the laboratory and available to all personnel. Standard solutions should be prepared in a hood whenever possible. The stock hexavalent chromium standards are toxic and should be handled with extreme care.
- 5.2 **Samples:** Take precautions when handling samples. Samples must always be treated as potentially hazardous "unknowns". The use of personal protective equipment such as gloves, lab coats and safety glasses is required when handling samples. In the event a sample container must be opened, it is recommended to perform this in a hood whenever possible. All digestions must be conducted under a fume hood.

6. Definitions

- 6.1 Refer to Glossary section of the Pace Quality Assurance Manual (QAM) for a comprehensive list of terms and definitions.

7. Sample Collection, Preservation, and Handling

Table 7.1 – Sample Collection, Preservation, Storage and Hold time.

| Sample type | Collection per sample | Preservation | Storage | Hold time |
|----------------------------|----------------------------|--|------------|--|
| Aqueous Unpreserved | 250mL in plastic container | None required | 0°C to 6°C | Analyze samples within 24 hours of collection date. |
| Aqueous Preserved | 250mL in plastic container | NaOH to pH >9 Alternatively, Ammonium Sulfate Buffer to pH >9 | 0°C to 6°C | Analyze samples within 28 days of collection date. |
| Solid | >100g in a glass container | None required | 0°C to 6°C | Digest samples within 30 days of collection date and analyze digestates within 7 days of digestion date. |

Samples must be stored separately from all standards, reagents, and highly contaminated samples. To avoid contamination, no food or drink products can be located near samples.

8. Equipment and Supplies

8.1. Instrumentation

| Equipment | Vendor | Description / Comments |
|-------------------|---------------------------|---|
| Spectrophotometer | Hach DR5000 or equivalent | For use at 540nm with a flow-through cell providing a light path of 1cm or longer |

8.2. General Supplies

| Item | Vendor | Description |
|--------------------|-------------------------|---------------|
| Graduated Cylinder | | Class A, 50mL |
| Mechanical pipets | Eppendorf or equivalent | Various sizes |
| Filter membrane | Whatman or equivalent | 0.45um |
| Disposable beakers | | 20mL |
| Transfer pipets | | Disposable |

9. Reagents and Standards

9.1. Reagents

| Reagent | Concentration/ Description |
|---|--|
| Reagent water | ASTM Type II |
| ChromaVer 3 Chromium reagent powder pillows | Hach cat.#12710-99 or equivalent |
| 1:1 Sulfuric Acid | Reagent grade or equivalent |
| Sodium Hydroxide (50%) | Fisher cat#SS254-4 or equivalent |
| Ammonium Sulfate Crystal | Fisher cat#A702-500 or equivalent |
| Ammonium Hydroxide | Fisher cat#A512-500 or equivalent |
| Ammonium Sulfate Buffer | Dissolve 33 g of ammonium sulfate in 75 mL reagent water and add 6.5 mL ammonium hydroxide. Dilute to 100 mL with reagent water. |

9.2. Analytical Standards

9.2.1. Definitions

Standards are required for initial calibration, calibration verification standards, second source verification, and for preparing LCS, MS, and MSD samples.

Table 9.2 Standard Definitions

| Standard | Description | Comments |
|--|---|---|
| Initial Calibration Standards | Standards prepared at varying levels to determine calibration range of the instrument. | ICAL |
| Initial Calibration Verification Standard | A standard prepared from a source other than that used for the initial calibration. This standard verifies the accuracy of the calibration curve. | ICV |
| Continuing Calibration Verification Standard | A calibration standard prepared at mid-level concentration. This standard is used to verify the initial calibration. | CCV |
| Spiking Standard | This solution contains all target analytes and should be prepared from a different source than the calibration standards. | This solution is used for the LCS, MS/MSD, and post-digestion spikes. |

9.2.2. Storage Conditions

Table 9.3 – Analytical Standard Storage Conditions

| Standard Type | Description | Expiration | Storage |
|--|---|--|---|
| Stock Cr(VI) Calibration Standard | Ricca catalog #2095-47; 100mg/L or equivalent | Manufacturer's recommended expiration date | Manufacturer's recommended storage conditions |
| Intermediate Cr(VI) Calibration Standard | Refer to Section 9.2.3.1 | Must be prepared fresh daily | Not Applicable |
| Working Cr(VI) Calibration Standards | Refer to Section 9.2.3.2 | Must be prepared fresh daily | Not Applicable |
| Stock Cr(VI) ICV/LCS Standard | Hach; catalog #810-42H; 50mg/L or equivalent | Manufacturer's recommended expiration date | Manufacturer's recommended storage conditions |

9.2.3. Standard Preparation Procedures

Refer to the standard preparation logbook or database for additional instructions regarding preparation of standards for Cr(VI) analysis. Instructions for preparation of fresh daily standards are detailed below. Refer to SOP S-IN-I-070 Alkaline Digestion of Solid Samples for Hexavalent Chromium or its replacement for reagents and standards associated with the preparation of solid samples.

9.2.3.1. Intermediate Cr(VI) Calibration Standard Preparation

Dilute 1mL of the Stock Cr(VI) Calibration Standard (100mg/L) to 100mL with reagent water to give a final concentration of 1.0mg/L.

9.2.3.2. Working Cr(VI) Calibration Standard Preparation

Working calibration standards must be prepared fresh daily by diluting the Intermediate Cr(VI) Calibration Standard (1mg/L) to 10mL with reagent water. Examples of possible calibration standards are as follows:

| Standard ID | Amt. of Intermediate Calibration Std. Used | Final Volume | Final Concentration |
|------------------|--|----------------------|---------------------|
| Standard 1 | 0.1mL | 10mL | 0.01mg/L |
| Standard 2 | 0.5mL | 10mL | 0.05mg/L |
| Standard 3 | 1.0mL | 10mL | 0.10mg/L |
| Standard 4 (CCV) | 5.0mL | 10mL | 0.50mg/L |
| Standard 5 | 7.5mL | 10mL | 0.75mg/L |
| Standard 6 | 10mL | No dilution required | 1.0mg/L |

9.2.3.3. Working Cr(VI) ICV/LCS Standard Preparation

Dilute 0.1mL of the Stock Cr(VI) ICV/LCS Standard (50mg/L) to 10mL with reagent water for an ICV concentration of 0.50mg/L. This standard must be prepared fresh daily. This standard is also used as the LCS.

10. Calibration

10.1. Initial Calibration: A minimum of 5 calibration standards is required. The lowest calibration standard must be at or below the reporting limit. A new initial calibration must be analyzed every 6 months at a minimum. Refer to the Quality Manual for more information regarding calibration curves.

10.2. Linear Calibration: After zeroing the spectrophotometer with a reagent blank, use the instrumentation software to prepare a standard curve by plotting absorbance versus Cr(VI) concentration of each calibration standard. The analyst may employ a regression equation that does not pass through the origin. The regression will produce the slope and intercept terms for a linear equation in the form:

$$y = ax + b$$

where: y = instrument response (peak area)
a = slope of the line (the coefficient of x)
x = concentration of the calibration standard
b = intercept of the line

10.3. The regression calculation will generate a correlation coefficient (r) that is the measure of the “goodness of fit” of the regression line to the data. In order to be used for quantitative purposes, the correlation coefficient must be ≥ 0.995 .

$$r = \frac{\sum_{i=1}^N (X_i - \bar{X}) * (Y_i - \bar{Y})}{\sqrt{\left(\sum_{i=1}^N (X_i - \bar{X})^2 \right) * \left(\sum_{i=1}^N (Y_i - \bar{Y})^2 \right)}}$$

10.4. Initial Calibration Corrective Action: If the curve does not meet the acceptance criteria, then a new calibration curve must be analyzed. If the second curve attempt does not meet the acceptance criteria, the analyst must consult the department manager and instrument maintenance and/or preparation of new standards must be considered. Samples associated with a failed initial calibration must be reanalyzed.

- 10.5. Initial Calibration Verification (ICV):** In addition to meeting the linearity requirement, any new calibration curve must be assessed for accuracy in the values generated. To assess the accuracy relative to the purity of the standards, a single standard from a secondary source must be analyzed and the results obtained must be compared to the known value of the standard. This step is referred to as Initial Calibration Verification. The ICV is analyzed immediately following an initial calibration curve. Acceptable recovery range for the ICV is 90-110%.
- 10.6. ICV Corrective Action:** If the ICV is not acceptable, another ICV may be analyzed. If the second ICV fails, then a new initial calibration curve must be analyzed. Instrument maintenance and/or preparation of new standards must also be considered. Samples associated with a failed ICV must be reanalyzed. **Exception:** If the ICV is outside of the upper control limit, indicating high bias, associated samples determined to be <RL may be reported.
- 10.7. Continuing Calibration Verification (CCV):** When an ICAL is not analyzed, the calibration must be verified by analyzing a CCV at the beginning of the analytical sequence. In all cases, a CCV must also be analyzed after every 10 samples and at the end of the analytical sequence to verify the system is still calibrated. The CCV should be from the same material as the curve standards. The acceptable recovery range for the CCV is 90-110%.
- 10.8. CCV Corrective Action:** If a CCV fails the acceptance criteria, another CCV may be analyzed. If the second CCV fails, then a new initial calibration curve must be analyzed. Samples must be bracketed by acceptable CCVs in order to be reportable. Samples associated with a failed CCV must be reanalyzed. **Exception:** If the CCV is outside of the upper control limit, indicating high bias, associated samples determined to be <RL are reportable.
- 10.9. Continuing Calibration Blank (CCB):** A CCB consists of 10mL reagent water to which a Chromaver 3 reagent powder pillow has been added. A CCB must be analyzed after each ICV or CCV. If the CCB result is above the reporting limit, another CCB may be analyzed. If the second CCB fails, then a new calibration curve must be analyzed. Samples associated with a failed CCB must be reanalyzed. **Exception:** If the CCB is >RL, associated samples determined to be <RL are reportable.

11. Procedures

- 11.1.** If samples are turbid, centrifuge samples to remove turbidity. If after centrifugation samples remain turbid, filtration of samples through a 0.45um filter may be performed.
- 11.2. Unpreserved aqueous samples:** Place 10mL of sample into a 20mL disposable beaker and add contents of one Chromaver 3 chromium reagent powder pillow. Cap the container and shake or stir for about 30 seconds to dissolve the powder.
- 11.3. Preserved aqueous samples and soil digestates:** Place 10mL of aqueous sample or soil digestate into a 20mL disposable beaker. Add 0.2mL of 1:1 sulfuric acid and add the contents of one Chromaver 3 chromium reagent powder pillow. Cap the container and shake or stir for about 30 seconds to dissolve the powder.
- 11.4.** Allow 5 to 10 minutes for the color to develop but do not wait beyond 20 minutes to take the reading of the sample on the spectrophotometer. A red-violet color will be observed in the presence of Cr(VI).
- 11.5.** Adjust the wavelength control of the spectrophotometer to 540nm. Zero the spectrophotometer using the reagent blank. Measure the absorbance of the standards, samples and blanks. A typical run sequence may be as follows:

ICAL Standards

ICV

(If ICAL not run, CCV would replace the ICAL and the ICV in the sequence)

CCB

Method blank

LCS

Client samples

CCV

CCB

Client samples

CCV

CCB

- 11.6.** To correct for background in samples, use an aliquot of the sample containing all reagents except the Chromaver 3 reagent powder pillow.
- 11.7.** Any sample with a Cr(VI) concentration that exceeds the linear range of the calibration curve must be diluted and reanalyzed or over range results must be qualified as estimated.
- 11.8.** From the corrected absorbance, determine the concentration of chromium present using the calculation below:

$$X = [(y)(a)] + b$$

Where: X = sample concentration
y = response or absorbance, corrected for background
a = slope
b = y-intercept

- 11.9.** Calculate the final concentration in the sample as follows:

$$\text{Aqueous Sample (mg/L)} = \frac{(X)(V_f)(D)}{(V_i)}$$

$$\text{Solid Sample (mg/Kg)} = \frac{(X)(V_f)(D)}{(W_s)}$$

Where: X = Sample concentration
V_f = Final sample volume in milliliters
D = Dilution factor
V_i = Initial sample volume in milliliters
W_s = Weight of solid sample extracted in grams

$$\text{Moisture corrected concentration} = \frac{(\text{Final concentration as received})}{(100 - \% \text{Moisture})} \times 100$$

12. Quality Control

12.1. Batch Quality Control

Table 12.1 – Batch Quality Control Criteria

| QA Sample | Components | Frequency | Acceptance Criteria | Corrective Action |
|---|--------------------------------------|--|--|---|
| Method Blank (MB) | Reagent or Ottawa sand | One per preparation batch of up to 20 samples per matrix. | Target analytes must be less than reporting limits | Re-digest and reanalyze if target compound is >RL in method blank and associated samples. <u>Exceptions:</u> 1) If no additional sample remains for reanalysis or if reanalysis cannot take place within holding time, the reported method blank and samples must be qualified. 2) If a contaminant is present only in the method blank and not the samples, no action is required. |
| Laboratory Control Sample (LCS) | Cr(VI) | One per preparation batch of up to 20 samples per matrix. | 80-120% Recovery | Re-digest and reanalyze associated samples if original LCS is outside acceptance limits. <u>Exceptions:</u> 1) If no additional sample remains for reanalysis or if reanalysis cannot take place within holding time, reported data must be qualified. 2) If LCS recovery is >QC limits and sample results are non-detect, the sample data may be reported without qualifiers. The LCS data must be qualified. |
| Aqueous Matrix Spike (MS)/Matrix Spike Duplicate (MSD) | Cr(VI) | One MS/MSD set per batch of up to 20 samples for waters. | 85-115% Recovery ≤20%RPD | No corrective actions necessary. If LCS recovery is in range, the system is considered in-control and the out-of-control MS/MSD must be qualified appropriately. |
| Solid Matrix Spike (MS)/Matrix Spike Duplicate (MSD) | Cr(VI) | One soluble and one insoluble MS/MSD set per preparation batch of up to 20 samples for solids. | 75-125% Recovery ≤20%RPD | If outside acceptance limits, evaluate sample for pH and Eh and compare to Figure 2 in Method 3060A. If in reducing range, no re-digestion is required. If outside reducing range, re-digestion and reanalysis of entire preparation batch is required. Refer to SOP S-IN-I-069 or equivalent for pH procedure. Refer to SOP S-IN-I-035 or equivalent for Eh procedure. |
| Sample Duplicate (DUP) Solids only | Sample | One Sample Duplicate per preparation batch of up to 20 samples. | ≤20% RPD | No corrective action is necessary if sample concentration is <4x RL. If sample concentration is >4x RL, the RPD must be qualified appropriately. |
| Post-Digestion Matrix Spike Solids only | Cr(VI) at 40mg/Kg or 2x sample conc. | One per preparation batch of up to 20 samples. | 85-115% Recovery | No corrective actions necessary. |

12.2. Aqueous Method Blank Preparation: the Method Blank consists 10mL of reagent water.

12.3. Aqueous LCS Preparation: Dilute 0.1mL of the Stock Cr(VI) ICV/LCS Standard (50mg/L) to 10mL with reagent water for an LCS concentration of 0.50mg/L. Prepare fresh daily.

12.4. Aqueous MS/MSD Preparation: Dilute 0.1mL of the Stock Cr(VI) ICV/LCS Standard (50mg/L) to 10mL with sample for a spike concentration of 0.50mg/L.

12.5. LCS equation:

$$R = (C/S) * 100$$

Where R = percent recovery

C = spiked LCS concentration

S = concentration of analyte added to the clean matrix

12.6. MS/MSD equation:

$$R = \frac{(C_s - C)}{S} * 100$$

Where R = percent recovery

C_s = spiked sample concentration

C = sample concentration

S = concentration of analyte added to the sample

12.4 RPD equation:

$$RPD = \frac{|D_1 - D_2|}{[(D_1 + D_2)/2]} * 100$$

Where RPD = relative percent difference

D₁ = first sample result

D₂ = second sample result

13. Method Performance

13.1. Method Detection Limit (MDL) Study: An MDL study must be conducted every 12 months for each matrix per instrument.

13.2. Demonstration of Capability (DOC): Every analyst who performs this method must first document acceptable accuracy and precision by passing a demonstration of capability study (DOC).

14. Method Modifications

14.1. Aqueous sample preservation to pH >9 using NaOH is performed as an alternative to preservation using Ammonium Sulfate Buffer.

14.2. Hach ChromaVer 3 combined reagent is used in place of laboratory prepared H₂SO₄ solution and Diphenylcarbazide solution.

14.3. Calibration standards are purchased as certified standards.

15. Pollution Prevention and Waste Management

- 15.1. Procedures for handling waste generated during this analysis are addressed in S-IN-S-002, Waste Handling or other appropriate SOP. All wastes are accumulated, managed and disposed of in accordance with all federal and state laws and regulations.
- 15.2. In order to minimize the amount of waste generated during this procedure, analyst should prepare reagents in an amount which may be used in a reasonable amount of time (i.e. before a reagent expires)
- 15.3. The company wide Chemical Hygiene and Safety Manual contains additional information on pollution prevention.

16. References

- 16.1. "Test Methods for Evaluating Solid Waste, Physical/Chemical Methods"; EPA SW-846, latest revision of methods 7196A and 3060A.
- 16.2. Standard Methods for the Examination of Waste and Wastewater, Method 3500-Cr B.
- 16.3. 40 CFR, Part 136, Table IB and Table II, Method Update Release, 2012
- 16.4. Pace Analytical Quality Manual; latest revision.
- 16.5. TNI Standard; Quality Systems section; latest revision.

17. Tables, Diagrams, Flowcharts, Attachments, Appendices, etc.

- 17.1. Not Applicable

18. Revisions

| Document Number | Reason for Change | Date |
|------------------|--|-----------|
| S-IN-I-063-rev.9 | <ol style="list-style-type: none">1. Table 7.1: revised hold time for soils.2. Section 9.2.2.3: adjusted wording to match actual practice.3. Section 10.2: added equation for correlation coefficient.4. Sections 10.3 and 10.5: added sentence to end of paragraph about rerunning samples.5. Section 10.4: added "preceding or" to language about bracketing CCVs.6. General: updated all SOP references7. Removed sentence from section 13.2 about regulatory limits.8. Added soil equation to section 12.5.9. Replaced Whatman filter with 0.45um filter in sections 9.4 and 12.110. Reworded sections 12.2 and 12.3 to reflect current practices.11. Added paragraph to section 13.5 about soil spikes.12. Added 3060 phase diagram to section 17. | 13Nov2007 |

| | | |
|-------------------|---|-----------|
| S-IN-I-063-rev.10 | <ol style="list-style-type: none"> 1. Cover page converted to periodic review format and added SM3500-Cr D reference. 2. Table of Contents fixed to include sections 10 and 11. 3. Section 1: added SM3500-Cr D reference. 4. Section 3: added language that limits, volumes, vendors, are subject to change. 5. Table 7.1: updated to clarify storage and separated requirements for unpreserved and preserved aqueous samples. 6. Section 8: updated and added "or equivalent" where applicable. 7. Section 9: updated and clarified separate standards as intermediate and working. Created a table for working calibration standard examples. 8. Section 10: completely revised to individually clarify calibration requirement including corrective actions. 9. Section 11: clarified procedure including intended run sequence and added calculations for final concentration. 10. Section 12: updated limits and corrective actions and added instructions for aqueous batch QC preparation. 11. Section 13: removed SOP references. 12. Section 15: changed NELAC to TNI and added SM3500-Cr D reference. | 23Jun2011 |
| S-IN-I-063-rev.10 | <ol style="list-style-type: none"> 1. Cover: revised Standard Methods reference from "D" to "B". 2. Section 1.1: revised Standard Methods reference and added a note that SM 3500-Cr B supersedes and is equivalent to SM 3500-Cr D. 3. Section 3.1: added a reference to the alkaline digestion SOP for soil procedures. 4. Section 3.2: added reference to LIMS for MDLs. 5. Table 7.1: updated for water preservation procedure and changed preserved water holding time from 30 days to 28 days. 6. Table 9.1: added reagents for Ammonium Sulfate Buffer 7. Section 9.2.3: added a reference to the alkaline digestion SOP for reagents and standards associated with the preparation of solid samples. 8. Section 11.7: added requirement to qualify over range results 9. Section 11.8: revised/fixed calculation for concentration from curve. 10. Table 12.1: revised method blank corrective actions and added references to pH and Eh procedures for solid sample MS/MSD corrective action. 11. Section 12: added/expanded aqueous LCS preparation. 12. Inserted new Method Modifications section. 13. References: revised Standard Methods reference and added reference to 40 CFR 2012 MUR. | 26Sep2012 |



Pace Analytical Services, Inc.

7726 Moller Road
Indianapolis, IN 46268

Phone: 317.875.5894

Fax: 317.872.6189

STANDARD OPERATING PROCEDURE

MEASUREMENT OF PERCENT MOISTURE IN SOILS AND SOLIDS

REFERENCE METHOD: ASTM METHOD D 2974-87

SOP NUMBER: S-IN-I-094-rev.07

EFFECTIVE DATE: Date of Final Signature

SUPERSEDES: S-IN-I-094-rev.06

APPROVAL

General Manager

September 25, 2012

Date

Quality Manager

September 24, 2012

Date

Department Manager

September 24, 2012

Date

PERIODIC REVIEW

SIGNATURES BELOW INDICATE NO CHANGES HAVE BEEN MADE SINCE APPROVAL.

Signature

Title

Date

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Date

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1. Purpose

- 1.1** The purpose of this SOP is to provide a laboratory specific procedure for measuring percent moisture in solid samples while meeting the requirements specified in ASTM method D 2974-87.

2. Summary of Method

- 2.1.** A sample aliquot is weighed before and after heating to dryness at 103°-105°C. The weight loss is calculated as percent moisture.

3. Scope and Application

- 3.1.** This method is applicable to most solid samples, including sludges, containing at least 0.1% moisture.
- 3.2.** Percent moisture is used to correct results of inorganic and organic tests to dry weight basis.
- 3.3.** This procedure is restricted to use by, or under the supervision of, analysts experienced in the measurement of percent moisture.

4. Interferences

- 4.1.** Non-representative materials, such as leaves and sticks, should be removed from the sample prior to measurement.
- 4.2.** Measurements are subject to negative bias for samples containing significant quantities of ammonium carbonate, volatile organics, or other volatile materials that could be lost during drying.

5. Safety

5.1. Standards and Reagents

The toxicity and carcinogenicity of standards and reagents used in this method have not been fully defined. Each chemical compound should be treated as a potential health hazard. Reduce exposure by the use of gloves, lab coats and safety glasses. Material Safety Data Sheets (MSDSs) are on file in the laboratory and available to all personnel.

5.2. Samples

Take precautions when handling samples. Samples should always be treated as potentially hazardous "unknowns". The use of personal protective equipment (gloves, lab coats and safety glasses) is required when handling samples. In the event a sample container must be opened, it is recommended to perform this in a hood whenever possible.

6. Definitions

- 6.1.** Refer to Glossary section of the Pace Quality Assurance Manual (QAM) for a comprehensive list of terms and definitions.

7. Sample Collection, Preservation, and Handling

Table 7.1 – Sample Collection, Preservation, Storage and Hold time.

| Sample type | Collection per sample | Preservation | Storage | Hold time |
|-------------|--|---------------|------------|---|
| Solid | >100g in a glass container with a tightly fitting lid. | None required | 0°C to 6°C | Samples must be analyzed within 28 days of collection |

Samples should be stored separately from all standards, reagents, and highly contaminated samples. To avoid contamination, no food or drink products can be located near samples.

8. Equipment and Supplies

8.1. Equipment

| Equipment | Model / Version | Description / Comments |
|--------------------|------------------------------|---|
| Analytical Balance | Ohaus AV212 or equivalent | Capable of weighing to 0.01g |
| Drying Oven | Fisher IsoTemp or equivalent | Able to maintain a temperature of 103° to 105°C |
| Desiccator | Fisher or equivalent | |

8.2. General Supplies

| Item | Model / ID | Description |
|-----------------|-------------------------|----------------------|
| Desiccant | Drier-ite or equivalent | |
| Weighing dishes | Fisher or equivalent | Disposable, aluminum |

9. Reagents and Standards

9.1. Reagents- Not applicable to this SOP.

9.2. Analytical Standards- Not applicable to this SOP.

10. Calibration

10.1. The analytical balance must be calibrated by an outside vendor at least annually and checked each day of use with Class 1 weights.

11. Procedures

11.1. Allow samples to warm to room temperature prior to processing.

11.2. For each sample, label and record the tare weight of a weighing dish to the nearest 0.01g.

11.3. Remove the top portion of material from the sample container and transfer approximately 10g of sample to the tared weighing dish.

11.4. Weigh the wet sample and dish, recording the weight to the nearest 0.01g.

11.5. Place the samples in the drying oven and dry at 103°C - 105°C for at least 4 hours, preferably overnight.

- 11.6. Remove the sample from the oven and place into a desiccator to cool.
- 11.7. After the sample has cooled, weigh the dry sample and dish to the nearest 0.01g.
- 11.8. If the sample was not dried overnight, return it to the oven and dry at 103° - 105°C for an additional hour. Remove the sample from the oven and place into a desiccator to cool. Weigh the sample and dish to the nearest 0.01g. Repeat this process until a constant weight is achieved or until weight change is less than 4% of the previous weight.
- 11.9. Calculate percent moisture using the following equations:

$$\% \text{ Moisture} = [(\text{wet sample weight} - \text{dry sample weight}) * 100] / \text{wet sample weight}$$

Where: Wet sample weight = Wet Mass – Tare Mass
Dry sample weight = Dry Mass – Tare Mass

12. Quality Control

12.1. Batch Quality Control

Table 12.1 – Batch Quality Control Criteria

| QA Sample | Components | Frequency | Acceptance Criteria | Corrective Action |
|-----------|------------|------------------------------------|---------------------|---|
| Duplicate | Sample | One per batch of up to 10 samples. | ≤5%.RPD | Analyze another sample and duplicate. Analyst can still report data from original duplicate as long as it is properly qualified. |

12.2. RPD calculation:

$$RPD = \frac{|D_1 - D_2|}{[(D_1 + D_2)/2]} * 100$$

Where RPD = relative percent difference
D₁ = first sample result
D₂ = second sample result

13. Method Performance

- 13.1. The analyst must read and understand this procedure with written documentation maintained in his/her training file.

14. Method Modifications

- 14.1. A nominal sample amount of 10g is used instead of 50g.
- 14.2. Constant weight is not determined on samples that are dried overnight.

15. Pollution Prevention and Waste Management

- 15.1. Procedures for handling waste generated during this analysis are addressed in S-IN-S-002, Waste Handling, or other applicable SOP.
- 15.2. In order to minimize the amount of waste generated during this procedure, analyst should prepare reagents in an amount which may be used in a reasonable amount of time (i.e. before a reagent expires)
- 15.3. The company wide Chemical Hygiene and Safety Manual contains additional information on pollution prevention.

16. References

- 16.1. ASTM method D 2974-87; Standard Test Methods for Moisture, Ash and Organic Matter of Peat and Other Organic Soils; American Society of Testing and Materials; March/April 1993.
- 16.2. Pace Analytical Quality Manual; latest revision.
- 16.3. TNI Standard; Quality Systems section; latest revision.

17. Tables, Diagrams, Flowcharts, Attachments, Appendices, etc.

- 17.1. Not applicable to this SOP.

18. Revisions

| Document Number | Reason for Change | Date |
|-------------------|---|-----------|
| S-IN-I-094-rev.4 | <ul style="list-style-type: none">1. Restructured document format to new corporate template.2. Changed several sections of information to tabular form. | 10Apr2007 |
| S-IN-I-094-rev.5 | <ul style="list-style-type: none">1. Updated SOP references where applicable. | 08Sep2008 |
| S-IN-I-094-rev.06 | <ul style="list-style-type: none">1. Revised cover page to reflect periodic review format.2. Table of Contents fixed to include sections 10 and 11.3. Table 7.1: clarified storage and hold time requirements.4. Section 8: updated information and added "or equivalent" where applicable.5. Section 15: changed NELAC to TNI. | 31May2011 |
| S-IN-I-094-rev.07 | <ul style="list-style-type: none">1. Section 11.8: clarified constant weight requirement.2. Section 11.9: clarified calculation and defined wet sample weight and dry sample weight to be consistent with %M LIMSlink format.3. Inserted new Method Modification section. | 24Sep2012 |



STANDARD OPERATING PROCEDURE

THE DETERMINATION OF VOLATILE ORGANICS BY GC/MS

REFERENCE METHOD: EPA SW-846 METHODS 8260B, 5030A, 5030B AND 5035A

| | |
|-------------------|-------------------------|
| LOCAL SOP NUMBER: | S-IN-O-029-rev.17 |
| EFFECTIVE DATE: | Date of Final Signature |
| SUPERSEDES: | S-IN-O-029-rev.16 |

LOCAL APPROVAL

Karl Anderson

General Manager

September 25, 2012

Date

Beck Schrage

Quality Manager

September 25, 2012

Date

Heath E. Bantier

Department Manager

September 24, 2012

Date

PERIODIC REVIEW

SIGNATURES BELOW INDICATE NO CHANGES HAVE BEEN MADE SINCE APPROVAL.

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1. Purpose

- 1.1. This Standard Operating Procedure (SOP) documents the procedures used by Pace Analytical Services – Indianapolis to determine the concentration of Volatile Organic Compounds (VOCs) in environmental samples. The laboratory utilizes purge-and-trap GC/MS and bases these documented procedures on those listed in SW-846 Method 8260B, 5030A, 5030B, and 5035A.

2. Summary of Method

- 2.1. Volatile organic compounds are introduced into the gas chromatograph by a purge-and trap method. The analytes are purged from a sample aliquot or extract using an inert gas. The purged analytes are collected on an absorbent trap. At the completion of the purge time, the trap is rapidly heated and back flushed to drive out the trapped analytes. The analytes are transferred into the inlet of a capillary gas chromatography column. The carrier gas flow through the column is controlled and the temperature is increased according to a set program to achieve optimum separation of purged analytes. The mass spectrometer is operated in a repetitive scan mode. Analytes are identified by the GC/MS retention times and by a comparison of their mass spectra with spectra of authentic standards. Analytes are quantified by comparing the response of a selected primary ion relative to an internal standard against a calibration curve.

3. Scope and Application

- 3.1. This method is applicable to most organic compounds that have boiling points below 200 °C and are insoluble or slightly soluble in water. Volatile water-soluble compounds may also be determined although quantitation limits are typically higher due to their hydrophilic properties (e.g. ketones, oxygenates). The list of compounds and reporting limits is found in Table 1. Refer to the LIMS for method detection limits.
- 3.2. This method is applicable to most water and solid samples, regardless of moisture content. Matrices are groundwater, surface water, soil, sediment and waste. Procedures may need to be adapted to address limits in the method or equipment that might hinder or interfere with sample analysis.
- 3.3. Reporting limits, control limits, volumes/weights used, standard concentrations, vendors, instrumentation, equipment and supplies are subject to change.
- 3.4. This procedure is restricted to use by, or under the supervision of, analysts experienced in the use of purge-and-trap GC/MS systems and interpretation of GC/MS data. Each analyst must demonstrate the capability to generate acceptable results with this method to be considered qualified to report sample results.

4. Interferences

- 4.1. Major contaminant sources are volatile materials in the laboratory and impurities in the inert purging gas and in the absorbent trap. The use of polytetrafluoroethylene (PTFE, Teflon) as thread sealants, tubing, or in flow controllers is highly recommended since other materials can be sources of contamination which may concentrate in the trap during the purging.
- 4.2. A common source of interfering contamination is carryover. This may occur when a sample containing low concentrations of volatile organic compounds is analyzed immediately after a sample containing high concentrations of volatile organic compounds. The preventive action to this condition is rinsing the purging apparatus and sample syringes with organic free water between samples. Analyze one or more blanks to check for contamination prior to sample analysis.

- 4.3.** Since methylene chloride and acetone are common laboratory solvents, special precautions must be taken. The volatiles analysis and sample storage area must be located as far as possible from areas where these solvents are used or stored. Where possible, the volatiles analysis and sample storage area should be served by a separate HVAC system and maintained under positive pressure to prevent intrusion of contaminants. Laboratory clothing previously exposed to methylene chloride fumes during extraction procedures can contribute to sample contamination.

5. Safety

5.1. Standards and Reagents

The toxicity and carcinogenicity of standards and reagents used in this method have not been fully defined. Each chemical compound should be treated as a potential health hazard. Reduce exposure by the use of gloves, lab coats and safety glasses. Material Safety Data Sheets (MSDSs) are on file in the laboratory and available to all personnel. Standard solutions should be prepared in a hood whenever possible.

5.2. Samples

Take precautions when handling samples. Samples must always be treated as potentially hazardous "unknowns". The use of personal protective equipment such as gloves, lab coats and safety glasses is required when handling samples. In the event a sample container must be opened, it is recommended to perform this in a hood whenever possible.

5.3. Equipment

Portions of the analytical instrumentation operate at high temperatures and under positive pressure. Care must be taken to minimize accidents and injuries when working on or with this equipment. Instruments must be turned off or the heated zone temperatures lowered to reduce the risk of thermal burns. Allow adequate time for the equipment to cool prior to working on these specific zones.

The purge and trap concentrator and autosampler use gas under pressure to purge samples and, in some cases, drive the robotic assemblies. These high pressures introduce the risk of injury due to flying glass and other objects should a vessel or line rupture. Safety glasses are required at all times when working in, on or around these pieces of equipment. Even instrumentation that is not operating may contain portions of the system under pressure.

6. Definitions

- 6.1.** Refer to Glossary section of the Pace Quality Assurance Manual (QAM) for a comprehensive list of terms and definitions.

7. Sample Collection, Preservation, and Handling

Table 7.1 – Sample Collection, Preservation, Storage and Hold time.

| Sample type | Collection per sample | Preservation | Storage | Hold time |
|--|--|---|--|---|
| 5030B Aqueous | Minimum (3) VOA vials Additional sample is required if MS/MSD is requested | Acidified w/ 1:1 HCl to pH<2, no headspace | 0 ° to 6°C | pH>2: Analysis must be completed within 7 days of collection date. pH <2: Analysis must be completed within 14 days of collection date. (pH determined post analysis) |
| 5035A Solid Terra Core Kits (Preferred) | One (1) 2-4 oz. wide mouth jar for % moisture <u>AND</u> Two (2) 5-g portions in vials with magnetic stir bar and 5.0mL reagent water plus one (1) 5 g portion in a vial with 5.0mL methanol. Additional sample is required if MS/MSD is requested. | No preservative or Methanol | 0 ° to 6°C for no more than 48 hours from collection then freeze at -7°C to -20°C. | Analysis must be completed within 48 hours if samples are not frozen or preserved with methanol prior to the expiration of the 48 hour period. The holding time may be extended to 14 days if the sample is frozen or preserved with methanol prior to the expiration of the 48 hour period. |
| 5035A Solid Coring Devices (Alternate) | One (1) 2-4 oz. wide mouth jar for % moisture <u>AND</u> Two (2) EnCore, TerraCore or similar sampling tubes. Additional sample is required if MS/MSD is requested. | No preservative Sample is extruded into a vial with a magnetic stir bar and 5.0mL reagent water. | Freeze at -7°C to -20°C within 48 hours of collection. | Analysis must be completed within 14 days of collection date. |
| 5030A Solid Bulk Jars | One (1) 2-4 oz. wide mouth jar for % moisture <u>AND</u> One bulk sample jar, usually 4 oz. or 8oz. | No preservative Sample is weighed into a vial with a magnetic stir bar and 5.0mL reagent water. | 0 ° to 6°C | Analysis must be completed within 14 days of collection date. |

Samples must be stored separately from all standards, reagents, and highly contaminated samples. To avoid contamination, no food or drink products can be located near samples.

8. Equipment and Supplies

8.1. Instrumentation

| Equipment | Vendor | Model / Version | Description / Comments |
|--------------------|--------------------------|--|------------------------|
| Gas Chromatographs | Agilent | Lab uses models 5890, 6850 and 6890 | Or equivalent system |
| P&T Concentrators | EST Analytical or Tekmar | Lab uses Tekmar 3000 series, Encons, and Encon Evolution | Or equivalent system |
| Data Systems | Agilent | Chemstation | Or equivalent system |
| Autosamplers | EST Analytical | Lab uses EST 8100s, Centurion, and Centurion WS | Or equivalent system |
| Mass Spectrometers | Agilent | Lab uses models 5971, 5973 and 5975 | Or equivalent system |

8.2. Chromatography Supplies

| Item | Vendor | Model / ID | Description |
|--------------------|---------|-----------------------|----------------------------------|
| Analytical Columns | Agilent | J&W Scientific DB-624 | 20m x 0.18mm x 1um or equivalent |
| Trap | Supelco | Trap K | Or equivalent |

8.3. General Supplies

| Item | Description | Vendor/ Item # / Description |
|--------------------|---------------------------------|----------------------------------|
| Gas tight syringes | Various sizes | Hamilton or equivalent |
| Syringe valves | 2-way with Luer ends | Supelco or equivalent |
| Standard vials | 2mL stop/go vials | Supelco or equivalent |
| Analytical balance | Able to measure to nearest 0.1g | Ohaus or Sartorius or equivalent |
| Sample vials | 40mL vials; pre-cleaned | Eagle Picher or equivalent |

9. Reagents and Standards

9.1. Reagents

| Reagent | Concentration/ Description |
|---------------|--|
| Reagent water | ASTM Type II water |
| Methanol | Purge-and trap grade or equivalent |
| Sand | Or equivalent to be used as a simulated soil matrix. |

9.2. Analytical Standards

9.2.1. Definitions

Standards are required for initial calibration, calibration verification standards, second source verification, and for preparing LCS, MS, and MSD samples.

Table 9.1 Standard Definitions

| Standard | Description | Comments |
|--|---|---|
| Tune Standard | 4-Bromofluorobenzene (BFB) solution used to verify ion response ratios prior to analysis | Must inject between 5 and 50ng |
| Initial Calibration Standards | Standards prepared at varying levels to determine response and retention characteristics of instrument | |
| Initial Calibration Verification Standard | A standard prepared from a source other than that used for the initial calibration. This standard verifies the accuracy of the calibration curve. | ICV |
| Continuing Calibration Verification Standard | A calibration standard prepared at mid-level concentration for all target compounds. This standard is used to verify the initial calibration. | CCV |
| Spiking Standard | This solution contains required spiking compounds, at a minimum, and is used to prepare MS/MSD sets. | Same solution can be used for the LCS and MS/MSD. |

9.2.2. Storage Conditions

Table 9.2 – Analytical Standard Storage Conditions

| Standard Type | Description | Expiration | Storage |
|--|--|---|---|
| Stock VOA calibration standards | Restek; catalog # 558679; 250-5000ug/mL and #554123; 500ug/mL and #30216; 2000ug/mL and #30006; 5000ug/mL or equivalent. | Manufacturer's recommended expiration date. | Manufacturer's recommended storage conditions |
| Stock Gas calibration standards | Restek; catalog # 30439; 200ug/mL and o2si; catalog#020229-09, 10,000ug/mL or equivalent. | Manufacturer's recommended expiration date. | Manufacturer's recommended storage conditions |
| Intermediate VOA calibration standard | Refer to Section 9.2.3.1. | Solution good for 1 month from preparation | Same as stock standard. |
| Intermediate Gas calibration standard | Refer to Section 9.2.3.2. | Solution good for 1 week from preparation | Same as stock standard |
| Working VOA calibration standards | Refer to Section 9.2.3.3. | One-time use | Not applicable |
| Stock VOA ICV/Spiking standards | o2si; catalog #120868-02; 250-5000ug/mL and #020201-01, 1000ug/mL or equivalent | Manufacturer's recommended expiration date. | Manufacturer's recommended storage conditions |
| Intermediate VOA ICV/Spiking standard | Refer to Section 9.2.3.4. | Solution good for 1 month from preparation | Same as stock standard |
| Working ICV/Spiking standard | Refer to Section 9.2.3.5. | One-time use | Not applicable |
| Stock VOATune/Surrogate standard | Restek; catalog #30240, 2500ug/mL or equivalent | Manufacturer's recommended expiration date. | Manufacturer's recommended storage conditions |
| Stock VOA Internal standards | Restek; catalog #30241, 2500ug/mL or equivalent | Manufacturer's recommended expiration date. | Manufacturer's recommended storage conditions |
| Working Tune/Surrogate/Internal standard mix | Refer to Section 9.2.3.7. | Solution good for 1 month from preparation | Stored on autosampler under pressure in a 5mL vial. |

9.2.3. Preparation Procedures

9.2.3.1. Intermediate VOA Calibration Standard Preparation

Dilute 500uL of Restek #558679 plus 500uL of Restek #554123 plus 250uL of Restek #30216 plus 125uL of Restek #30006 to 2.5mL with Methanol for a final nominal concentration of 50mg/L.

9.2.3.2. Intermediate Gas Calibration Standard Preparation

Dilute 500uL of Restek #30439 plus 200uL of o2si #020229-09 to 2.0mL with Methanol for a final nominal concentration of 50mg/L.

9.2.3.3. Working Calibration Standards Preparation

Refer to Table 9.3 for examples of possible one-time use calibration standards.

Table 9.3 – Working Calibration Standards (examples only)

| Standard | Int. Calibration Standard amount | Int. Gas Standard amount | Final Total Volume | Final Concentration |
|-------------------------|----------------------------------|--------------------------|--------------------|---------------------|
| Calibration Std 1 | 1uL | 1uL | 50mL | 1ppb |
| Calibration Std 2 | 2uL | 2uL | 50mL | 2ppb |
| Calibration Std 3 | 5uL | 5uL | 50mL | 5ppb |
| Calibration Std 4 | 10uL | 10uL | 50mL | 10ppb |
| Calibration Std 5 | 2uL | 2uL | 5mL | 20ppb |
| Calibration Std 6 (CCV) | 5uL | 5uL | 5mL | 50ppb |
| Calibration Std 7 | 15uL | 15uL | 5mL | 150ppb |
| Calibration Std 8 | 30uL | 30uL | 5mL | 300ppb |

9.2.3.4. Intermediate VOA ICV/Spiking Standard Preparation

Dilute 200uL of o2si #120868-02 plus 200uL of o2si #020201-01 to 2mL with Methanol for a final nominal concentration of 50mg/L.

9.2.3.5. Working ICV/Spiking Standard Preparation

Add 50uL of the Intermediate ICV standard per 50mL water for a final ICV concentration of 50ug/L.

9.2.3.6. Laboratory Control Sample (LCS) and Matrix Spike (MS/MSD) Preparation

9.2.3.6.1 Aqueous LCS: add 5uL of the Intermediate ICV/Spiking standard to 5mL reagent water for an LCS concentration of 50ug/L.

9.2.3.6.2 Aqueous MS: add 5uL of the Intermediate ICV/Spiking standard to 5mL sample for an MS concentration of 50ug/L.

9.2.3.6.3 Soil LCS: place approximately 5g of simulated soil matrix and 5mL reagent water into a vial. Add 5uL of the Intermediate ICV/Spiking standard for an LCS concentration of 50ug/Kg.

9.2.3.6.4 Soil MS: place approximately 5g of sample and 5mL reagent water into a vial. Add 5uL of the Intermediate ICV/Spiking standard for an LCS concentration of 50ug/Kg.

9.2.3.7. Working Tune/Surrogate/Internal Standard Preparation

Dilute 100uL of Restek #30240 plus 100uL of Restek #30241 to 5mL with Methanol for a final concentration of 50mg/L.

10. Calibration

10.1. Tune Verification: At the beginning of each analytical sequence, prior to the analysis of any standards or samples, the mass spectrometer must be hardware tuned by injecting 5-50ng BFB. This is done by analyzing a standard containing BFB. The tune verification standard can be combined with the CCV standard provided that the amount of BFB introduced into the system meets the criteria. Use the BFB mass intensity criteria in the table below as tuning acceptance criteria. Alternate tuning criteria may be used provided that method performance is not adversely affected.

| Mass (m/z) | Ion Abundance criteria |
|------------|------------------------------------|
| 50 | 15 to 40% of m/z 95 |
| 75 | 30 to 60% of m/z 95 |
| 95 | Base peak, 100% relative abundance |
| 96 | 5 to 9% of m/z 95 |
| 173 | <2% of m/z 174 |
| 174 | >50% of m/z 95 |
| 175 | 5 to 9% of m/z 174 |
| 176 | 95 to 101% of m/z 174 |
| 177 | 5 to 9% of m/z 176 |

If the ratios do not meet the criteria above, reanalyze the BFB tune. If the BFB still fails the criteria, instrument maintenance and/or preparation of new standards must be considered.

10.2. Initial Calibration: Initial Calibration standards are introduced into the GC/MS from the lowest to highest concentration of each working calibration standard. The lowest calibration standard must be at or below the required reporting limit. Five calibration points, at a minimum, are analyzed to evaluate linearity. Refer to the Quality Manual for more information regarding calibration curves. The response factor (RF) is calculated for each compound for each calibration standard as follows:

$$RF = \frac{(A_x)(C_{IS})}{(A_{IS})(C_x)}$$

where: A_x = Area of the quantitation ion for the compound being measured
 A_{IS} = Area of the quantitation ion for the internal standard.
 C_{IS} = Concentration of the internal standard
 C_x = Concentration of the compound being measured.

10.3. The average response factor (RF_{avg}) is determined by averaging the response factors at the different concentrations for each target analyte

10.4. The percent relative standard deviation (%RSD) is calculated as follows:

$$\%RSD = \frac{(SD)}{RF_{avg}} \times 100$$

where: SD = Standard deviation of average RF for a compound
RF_{avg} = Mean of RFs for a compound

- 10.5.** The %RSD should be should be $\leq 15\%$ for each target analyte. However, the %RSD for each individual Calibration Check Compound (CCC) must be $\leq 30\%$. If an RSD is $>30\%$ for any CCC, then instrument maintenance and/or preparation of new standards must be considered before attempting recalibration. The CCCs are:

| | |
|---------------------|----------------|
| 1,1-Dichloroethene | Toluene |
| Chloroform | Ethylbenzene |
| 1,2-Dichloropropane | Vinyl Chloride |

- 10.6.** System Performance Check Compounds (SPCCs) are checked for a minimum average response factor (RF_{avg}) to determine potential instability and/or degradation caused by contaminated lines or active sites in the system. The minimum RF_{avg} for the volatile SPCCs are as follows:

| | |
|---------------------------|------|
| Chloromethane | 0.10 |
| 1,1-Dichloroethane | 0.10 |
| Bromoform | 0.10 |
| Chlorobenzene | 0.30 |
| 1,1,2,2-Tetrachloroethane | 0.30 |

- 10.7.** If the percent relative standard deviation (%RSD) of the RFs for a compound is $\leq 15\%$ over the calibration range, then linearity through the origin is assumed and the RF_{avg} may be used to determine sample concentrations.
- 10.8.** If the % RSD for any compound is $>15\%$, the analyst may employ a regression equation that does not pass through the origin. The regression will produce the slope and intercept terms for a linear equation in the form:

$$y = ax + b$$

where: y = instrument response (peak area)
a = slope of the line (the coefficient of x)
x = concentration of the calibration standard
b = intercept of the line

- 10.9.** The regression calculation will generate a correlation coefficient (r) that is the measure of the “goodness of fit” of the regression line to the data. In order to be used for quantitative purposes, the correlation coefficient must be ≥ 0.99 . Refer to SW-846 Method 8000C for detailed information regarding equations.

$$r = \frac{\sum_{i=1}^N (X_i - \bar{X}) * (Y_i - \bar{Y})}{\sqrt{\left(\sum_{i=1}^N (X_i - \bar{X})^2 \right) * \left(\sum_{i=1}^N (Y_i - \bar{Y})^2 \right)}}$$

- 10.10. Non-linear or quadratic calibration:** A non-linear or quadratic calibration model can only be used if the compound(s) have historically exhibited a non-linear response and cannot be used to extend the calibration range for any compound that normally exhibits a linear response in a narrower range. The non-linear regression calibration curve is derived from a least squares regression analysis of the calibration points. A calibration curve based on this technique will have the format of: $y = ax^2 + bx + c$. In order to use this curve fit technique, a minimum of 6 calibration points must be used and the origin cannot be included as one of the points. Because the non-linear regression is not forced through the origin, very low levels of contaminants below the response of the lowest calibration point may generate erroneous reportable results. The “goodness of fit” of the polynomial equation is evaluated by calculating the coefficient of the determination (COD) or r^2 . The COD or r^2 from the regression equation must be ≥ 0.99 . Refer to SW-846 Method 8000C for detailed information regarding equations.

$$\text{COD} = \frac{\sum_{i=1}^N (Y_i - \bar{Y})^2 - \left(\frac{n-1}{n-p}\right) \sum_{i=1}^N (Y_i - Y_i')^2}{\dots \dots \dots \sum_{i=1}^N (Y_i - \bar{Y})^2}$$

- 10.11. Initial Calibration Corrective Action:** If the initial calibration does not meet the required criteria to be used for quantitative purposes, a new initial calibration must be analyzed. Instrument maintenance and/or preparation of new calibration standards must also be considered. Samples associated with a failed initial calibration must be reanalyzed.

- 10.12. Initial Calibration Verification (ICV):** In addition to meeting the response and linearity criteria, any new calibration curve must be assessed for accuracy in the values generated. To assess the accuracy relative to the purity of the standards, a single standard from a secondary source must be analyzed and the results obtained must be compared to the known true value. This step is referred to as the Initial Calibration Verification. The ICV must be from an alternative vendor or, in the event an alternative vendor is not available, from a different lot from the same vendor. The accuracy of the standard is assessed as a percent difference (%D) of the observed ICV response from the initial calibration average response, according to the following equation where RF_v is the response factor of the verification standard:

$$\% \text{ Difference (\%D)} = [RF_v - RF_{avg}] / RF_{avg} * 100$$

The ICV is analyzed immediately following the initial calibration curve. All compounds must have a maximum percent difference of +/- 30% from the initial calibration curve. Allowances for poor performers (ie, Acrolein, Ketones, 2 CEVE, etc) must be approved by the department manager and clearly documented as to which compounds are affected. **For BP, the ICV must meet the CCV criteria.**

- 10.13. ICV Corrective Action:** If the ICV fails the criteria, another ICV may be analyzed. If the second ICV fails, a new initial calibration curve must be analyzed. Instrument maintenance and/or preparation of new calibration standards must also be considered. Samples associated with a failed ICV must be reanalyzed. **Exception:** If the ICV fails during an overnight run and is outside of the upper control limit, indicating high bias, associated samples determined to be <RL may be reported.

- 10.14. Continuing Calibration Verification:** The initial calibration is verified every 12 hours by analyzing a BFB tune that must meet the criteria in Section 10.1, followed by a Continuing Calibration Verification (CCV) standard. The CCV is normally prepared using the same standard solution used for the initial calibration but an ICV/LCS can be used as a CCV if it passes the required criteria for a CCV.

- 10.15.** The % difference (%D) between initial calibration and CCV response factors for CCCs must be $\leq 20\%$. The response factors for all SPCCs in the CCV standard must meet the criteria in Section 10.6. An LCS may be used as a CCV provided that it passes the CCV acceptance criteria. **For BP, surrogate compounds must also be $\leq 20\% D$.**
- 10.16.** The internal standard areas in the CCV must be between 50%-200% of the internal standard areas of the corresponding standard in the initial calibration. In addition, the retention time of the internal standards in the CCV cannot shift by more than 30 seconds from the corresponding standard in the initial calibration. Failure in either of these two areas requires the analyst to evaluate their system and perform maintenance if necessary.
- 10.17. CCV Corrective Action:** If a CCV fails the acceptance criteria, check the instrument operating conditions, and if necessary, restore them to the original settings, and analyze another CCV. If the response still fails the acceptance criteria, then a new initial calibration must be prepared. Samples associated with a failed CCV must be reanalyzed. **Exception:** If the CCV is outside of the upper control limit, indicating high bias, associated samples determined to be $< RL$ may be reported.

11. Procedures

- 11.1.** Configure the purge & trap system and GC/MS system per manufacturer's instructions. All samples must be analyzed at room temperature and the system must be calibrated and free of contamination before samples are analyzed.

11.2. Sample Preparation and Handling

11.2.1. Aqueous Samples

Water samples to be analyzed using the Centurion autosampler require no sample preparation and are loaded as full 40mL VOA vials, unless they require a dilution. Refer to Section 7 for additional information regarding sample handling.

Water samples to be purged on the Archon/8100 autosampler are prepared by quickly measuring a 5mL aliquot of the sample using a 5mL gastight syringe and transferring it to a 40mL VOA vial. This is done as quickly as possible to minimize analyte loss. The syringe is thoroughly rinsed inside and out with reagent water before measuring each sample.

Dilutions on aqueous samples must be prepared in a volumetric fashion. Sample aliquots are measured in either a volumetric pipet or gas-tight syringe and brought to volume in either a volumetric flask or gas-tight syringe.

After analysis, check the residue in the vial using pH paper. The pH should be < 2 . Holding time for water samples with pH > 2 is 7 days. Appropriately footnote on the sequence log and in LIMS any sample not meeting the pH requirement and/or holding time requirement. A stamp may be used to document on sequence logs that all water samples are pH < 2 unless otherwise noted.

11.2.2 Soil Samples

11.2.2.1 Low-Level soils

Preferably, samples received for low level analysis should be contained in pre-weighed Terra Core vials with Organic Free Water (OFW). Prior to analysis the sample weight must be determined and recorded by weighing the vial and recording the weight. Subtract the tare weight indicated on the vial to determine the sample weight. The sample is ready for analysis. Refer to Section 7 for additional information regarding sample handling.

Alternatively, samples received in coring devices, such as Encore, must be extruded into a pre-weighed VOA vial either with or without 5mL OFW and a magnetic stir bar. Record the weight of the vial after the sample has been placed into it. Subtract the tare weight

determined initially to determine the sample weight. The sample is ready for analysis.

Samples received in bulk soil jars are applicable to Method 8260A only and are sub-sampled into a VOA vial. Place an empty VOA vial on the balance pan and tare the balance. Quickly add approximately 5g of the sample to the vial. Record the sample weight. Add 5mL of reagent water and a magnetic stir bar and cap the vial. The sample is ready for analysis.

11.2.2.2 Medium-Level soils

Preferably, samples received for medium-level analysis should be received in pre-weighed Terra Core vials with methanol as a preservative. Prior to analysis the sample weight must be determined and recorded by weighing the vial and recording the weight. Subtract the tare weight indicated on the vial to determine the sample weight. The sample is mixed well on a vortex mixer and allowed to settle. A maximum of 200uL of the methanol extract per 5mL OFW is used for analysis. Refer to Section 7 for additional information regarding sample handling.

Alternatively, samples received in coring devices, such as Encore, must be extruded into a pre-weighed VOA vial with 5mL methanol. Record the weight of the vial after the sample has been placed into it. Subtract the tare weight determined initially to determine the sample weight. The sample is mixed well on a vortex mixer and allowed to settle. A maximum of 200uL of the methanol extract per 5mL OFW is used for analysis.

Samples received in bulk soil jars are applicable to Method 8260A only and are sub-sampled into a VOA vial. Place an empty VOA vial on the balance pan and tare the balance. Quickly add approximately 5g of the sample to the vial. Record the sample weight. Add 5mL methanol and cap the vial. The sample is mixed well on a vortex mixer and allowed to settle. A maximum of 200uL of the methanol extract per 5mL OFW is used for analysis.

11.3 Qualitative Analysis

11.3.2 The relative retention time (RRT) of the sample component must compare within +/- 0.06 RRT units of the RRT of the CCV component.

11.3.3 The intensities of the characteristic ions of a compound must maximize in the same scan or within one scan of each other. Refer to Table 2 for the characteristic ions.

11.3.4 The relative intensities of the characteristic ions agree within 30% of the relative intensities of these ions in the reference spectrum.

11.4 Quantitative Analysis: Quantitation is based on the integrated abundance of the target analyte's quantitation ion using the internal standard technique. See Sections 11.6, 11.7, and 11.8 for equations to calculate the amount of analyte or surrogate introduced into the instrument. Calculations are subject to change based on the data reduction software used.

11.5 When the average response factor from the initial calibration is used, calculate the on-column amount:

$$X_s = (A_s/RF_{avg}) \times (C_{is}/A_{is})$$

Where:

X_s = On-column concentration of the analyte or surrogate
 A_s = Peak area or height of the analyte or surrogate in the sample
 A_{is} = Peak area or height of the internal standard in the sample
 C_{is} = Concentration of the internal standard in the sample
 RF_{avg} = The average response factor from the initial calibration

11.6 When a linear curve fit is used, calculate the on-column amount:

$$X_s = \frac{[A_s/A_{is} - b]}{a} \times C_{is}$$

where: A_s = Peak area or height of the analyte or surrogate in the sample
 A_{is} = Peak area or height of the internal standard in the sample
 X_s = On-column concentration of the analyte or surrogate
 C_{is} = Concentration of the internal standard in the sample
 a = Slope of the line
 b = The intercept

11.7 When a non-linear or quadratic curve fit is used, calculate the on-column amount:

$$X_s = \frac{-b + \sqrt{b^2 - 4a \left(c - \frac{A_s}{A_{is}} \right)}}{2a} \times C_{is}$$

where: X_s = On-column concentration of the analyte or surrogate
 A_s = Peak area or height of the analyte or surrogate in the sample
 A_{is} = Peak area or height of the internal standard in the sample
 C_{is} = Concentration of the internal standard in the sample
 a = Slope of the line
 b = The intercept
 c = coefficient of the polynomial

11.8 Calculate the final concentration in the sample as follows:

$$\text{Aqueous Sample (ug/L)} = \frac{(X_s)(V_t)(D)}{(V_i)(V_s)} \quad \text{Solid Sample (ug/Kg)} = \frac{(X_s)(V_t)(D)}{(V_i)(W_s)}$$

Where: X_s = On-column concentration of the analyte in the sample aliquot injected
 V_t = Total volume of concentrated extract
 D = Dilution factor
 V_i = Volume of the extract injected in uL
 V_s = Volume of aqueous sample extracted in milliliters
 W_s = Weight of solid sample extracted in grams

$$\text{Moisture corrected concentration} = \frac{(\text{Final concentration as received})}{(100 - \% \text{Moisture})} \times 100$$

12 Quality Control

12.1 Batch Quality Control

Table 12.1 – Batch Quality Control Criteria

| QA Sample | Components | Frequency | Acceptance Criteria | Corrective Action |
|---|--|---|---|---|
| Method Blank (MB) | Reagent water | One per preparation batch of up to 20 samples, per matrix. | Target analytes must be less than reporting limits. | Reanalyze if target compound is >RL in method blank and associated samples. <u>Exceptions:</u> 1) If no additional sample remains for reanalysis or if reanalysis cannot take place within holding time, the reported method blank and samples must be qualified. 2) If a contaminant is present only in the method blank and not the samples, no action is required. |
| Laboratory Control Sample (LCS) | Applicable target analytes | One per preparation batch of up to 20 samples, per matrix. | Lab-generated limits Refer to the LIMS for acceptance limits. | Reanalyze associated samples if original LCS is outside acceptance limits. <u>Exceptions:</u> 1) If no additional sample remains for reanalysis or if reanalysis cannot take place within holding time, reported data must be qualified. 2) If LCS recovery is >QC limits and sample results are non-detect, the sample data may be reported without qualifiers. The LCS data must be qualified. |
| Matrix Spike (MS)/Matrix Spike Duplicate (MSD) | Applicable target analytes | One MS/MSD set per preparation batch of up to 20 samples, per matrix. | Lab-generated limits Refer to the LIMS for acceptance limits. | No corrective actions necessary. If LCS recovery is in range, the system is considered in-control and the out-of-control MS/MSD must be qualified appropriately. |
| Surrogates | Applicable surrogate compounds | Added to each standard, sample, and method blank. | Lab-generated limits Refer to the LIMS for acceptance limits. | Samples with surrogate failures must be reanalyzed. <u>Exceptions:</u> 1) If no additional sample remains for reanalysis or if reanalysis cannot take place within holding time, reported surrogate data must be qualified. 2) If surrogate result is >QC limits, and sample or method blank results are non-detect, the sample or method blank results may be reported without qualifiers. The surrogate must be qualified. 3) MS/MSD surrogate recovery failures do not constitute the re-extraction or reanalysis of samples but the surrogate data must be qualified. |
| For BP only: Internal Standards | Applicable Internal Standard compounds | Added to each standard, sample, and method blank. | Sample ISTD areas must be -50% to +100% from CCV. Sample ISTD RTs must be +/-0.5 minutes from CCV. | Samples with internal standard failures must be reanalyzed at the same dilution or more concentrated. <u>Exception:</u> 1) If no additional sample remains for reanalysis or if reanalysis cannot take place within holding time, reported data must be qualified. |

12.2 Method Blank Preparation

- 12.2.1 Waters on Archon autosamplers:** The Method Blank consists of a 40mL VOA vial containing 5mL reagent water.
- 12.2.2 Waters on Centurion autosamplers:** The Method Blank consists of a 40mL VOA vial filled completely with reagent water.
- 12.2.3 Low-level soils:** The Method Blank consists of a 40mL VOA vial containing approximately 5g simulated soil matrix and 5mL reagent water.
- 12.2.4 Medium-level soils:** The Method Blank consists of a 40mL VOA vial containing 5mL reagent water and 200uL methanol.

12.3 Laboratory Control Sample (LCS) and Matrix Spike (MS/MSD) Preparation: Refer to Section 9.2.3.6.

12.4 Allowable Marginal Exceedances: If a large number of analytes are in the LCS, it becomes statistically likely that a few will be outside control limits. A marginal exceedance (ME) is defined as being beyond the LCS control limit of +/-3 standard deviations, but within the ME limits of +/-4 standard deviations around the mean. The number of allowable MEs is based on the number of analytes in the LCS. If more analytes exceed the LCS control limits than is allowed, or if any one analyte exceeds the ME limits, the LCS fails and correction action is necessary. If the same analyte exceeds the LCS control limit consecutively, it is an indication of a systemic problem. The source of the error shall be located and corrective action taken.

The number of allowable marginal exceedances is as follows:

| Number of Analytes in LCS | Number Allowed as Marginal Exceedances |
|---------------------------|--|
| > 90 | 5 |
| 71 – 90 | 4 |
| 51 – 70 | 3 |
| 31 – 50 | 2 |
| 11 – 30 | 1 |
| < 11 | 0 |

NOTE: For BP, the LCS shall be allowed to be outside the control limits but $\geq 10\%$ for up to four additional volatile compounds with the exception of benzene, toluene, ethylbenzene, m-xylene, p-xylene, o-xylene, total xylenes and any requested oxygenate without corrective action.

12.5 LCS equation:

$$R = (C/S) * 100$$

Where R = percent recovery
C = spiked LCS concentration
S = concentration of analyte added to the clean matrix

12.6 MS/MSD equation:

$$R = \frac{(C_s - C)}{S} * 100$$

Where R = percent recovery
Cs = spiked sample concentration
C = sample concentration
S = concentration of analyte added to the sample

12.7 RPD equation:

$$\text{RPD} = \frac{|D_1 - D_2|}{[(D_1 + D_2)/2]} * 100$$

Where RPD = relative percent difference

D₁ = first sample result

D₂ = second sample result

13 Method Performance

13.1 Method Detection Limit (MDL) Study: An MDL study must be conducted annually for each matrix per instrument.

13.2 Demonstration of Capability (DOC): Every analyst who performs this method must first document acceptable accuracy and precision by passing a demonstration of capability study (DOC).

14 Method Modifications

14.1 GC columns and chromatographic conditions may differ from those recommended.

14.2 Calibration solutions are purchased as certified standards.

15 Pollution Prevention and Waste Management

15.1 Procedures for handling waste generated during this analysis are addressed in S-IN-S-002, Waste Handling, or other applicable SOP. All wastes are accumulated, managed and disposed of in accordance with all federal and state laws and regulations.

15.2 In order to minimize the amount of waste generated during this procedure, analyst should prepare reagents in an amount which may be used in a reasonable amount of time (i.e. before a reagent expires)

15.3 The company wide Chemical Hygiene and Safety Manual contains additional information on pollution prevention.

16 References

16.1 Test Methods for Evaluating Solid Waste, Physical/Chemical Methods, SW-846, Methods 8000B, 8000C, 8260A, 8260B, 5030A, 5030B, and 5035A.

16.2 Pace Analytical Quality Manual; latest revision.

16.3 TNI Standard; Quality Systems section; latest revision.

17 Tables, Diagrams, Flowcharts, Attachments, Appendices, etc.

17.1 Table 1: Method 8260B Target Compounds and Reporting Limits

17.2 Table 2: Characteristic Ions of Target Compounds

18 Revisions

| Document Number | Reason for Change | Date |
|-------------------|--|-----------|
| S-IN-O-029-rev.12 | <ol style="list-style-type: none"> Added new section 11.6.2 regarding signal-to-noise ratio. Added new section 10.2.3 regarding when and how to drop points from an initial calibration curve (copied from Quality Manual). Added new section 10.5 regarding the inability to use Grand Mean Averaging. Section 10.2.2: added last paragraph stating why the lab does not curve its surrogate compounds. Table 11.1: revised EPIC Pro instrument names | 11Sep2007 |
| S-IN-O-029-rev.13 | <ol style="list-style-type: none"> Table 10.2: revised internal standard retention time limits (for BP samples only) and added line for client samples regarding internal standard acceptance and that BP samples that have failing internal standards must be rerun. Updated ICV acceptance criteria (same as CCV). Section 11.4.1.1: added language that all water samples must have the pH measured and recorded. Section 9.2.4.4: adjusted recipe for surrogates per HEB. Section 10.3: changed 'direct injecting' to 'purging'. Table 11.1: added new instrument (50MSV4) and removed old naming convention. Section 11.4.1.3.2: revised maximum amount of methanol to 200uL. | 11Mar2008 |
| S-IN-O-029-rev.14 | <ol style="list-style-type: none"> Section 2: changed helium to nitrogen purge gas. Table 9.2: changed gas standard expiration to 1 month. Table 10.2: revised ICV and CCV criteria Table 11.1: removed specific instrument IDs – made generic. Section 11.4: added pre-screening procedure. Section 11.4.1: added requirement that samples that are pH>2 and analyzed after 7 days be qualified. Section 11.4.1.2.1: removed requirement that soil subsampling be done using an Encore sampling device. Table 12.1: revised LCS/MS compounds and criteria. | 31Mar2009 |
| S-IN-O-029-rev.15 | <ol style="list-style-type: none"> Removed SOP template from cover page Section 6: removed SOP specific definitions Table 7.1: replaced with a more detailed table. Section 8: added "or equivalent" where applicable Section 9.2.4: added wording regarding expiration dates. Table 10.1: removed Section 10.3.3: clarified RL wording Table 10.2: added use of LCS as CCV Section 10.5.3: added use of LCS as CCV Section 11.1.1: revised to consult manufacturer's instructions Table 11.1: removed Section 11.3: removed Note Section 11.4.2.1: improved wording regarding samples pH Section 11.4.2.2: replaced "weight" with "mass" Section 11.4.2.3.2.1: added use of gastight syringe Table 12.2: reworded exceptions for clarity Table 12.3: reworded IS Area criteria for clarity | 12Mar2010 |

| | | |
|-------------------|---|-----------|
| S-IN-O-029-rev.16 | <ol style="list-style-type: none">1. Section 3: added language that limits, volumes, vendors, are subject to change.2. Table 7.1: updated to clarify hold time and acceptable preservation. Deleted Table 7.2.3. Section 8: updated and added "or equivalent" where applicable.4. Section 9: updated and clarified separate standards as intermediate and working. Created a table for all calibration standards5. Section 10: completely revised to individually clarify calibration requirement including corrective actions. Simplified BFB tune information.6. Section 11: clarified procedure including calibration calculations and added calculations for final concentration. Removed information about TICs.7. Section 12: updated information and corrective actions and added instructions for batch QC preparation. Clarified marginal exceedance criteria.8. Section 13: removed SOP references.9. Section 15: changed NELAC to TNI.10. Attachments: removed previous Att. 1 and added Att. 1 and Att. 2. | 13Jun2011 |
| S-IN-O-029-rev.16 | <ol style="list-style-type: none">1. Section 3.1: added reference to MDLs2. Section 9.1: added simulated soil matrix.3. Section 9.2.3.6: added detail for preparation of LCS and MS to include simulated soil matrix.4. Table 12.1: revised method blank corrective action.5. Section 12.2.3: revised low-level soil method blank prep to include simulated soil matrix.6. Inserted new Method Modifications section. | 24Sep2012 |

Table 1: Method 8260B Target Compounds and Reporting Limits¹

| Analyte | RL water (ug/L) | RL soil Low-level (ug/kg) | RL soil Medium-level (ug/kg) |
|---|--------------------|---------------------------------|------------------------------------|
| Dichlorodifluoromethane | 5 | 5 | 125 |
| Chloromethane | 5 | 5 | 125 |
| Vinyl Chloride | 2 | 5 | 125 |
| Bromomethane | 5 | 5 | 125 |
| Chloroethane | 5 | 5 | 125 |
| Trichlorofluoromethane | 5 | 5 | 125 |
| Methylene Chloride | 5 | 20 | 500 |
| 1,1-Dichloroethene | 5 | 5 | 125 |
| trans-1,2-Dichloroethene | 5 | 5 | 125 |
| 1,1-Dichloroethane | 5 | 5 | 125 |
| 2,2-Dichloropropane | 5 | 5 | 125 |
| cis-1,2-Dichloroethene | 5 | 5 | 125 |
| Chloroform | 5 | 5 | 125 |
| Bromochloromethane | 5 | 5 | 125 |
| 1,1,1-Trichloroethane | 5 | 5 | 125 |
| Carbon Tetrachloride | 5 | 5 | 125 |
| 1,1-Dichloropropene | 5 | 5 | 125 |
| Benzene | 5 | 5 | 125 |
| 1,2-Dichloroethane | 5 | 5 | 125 |
| Trichloroethene | 5 | 5 | 125 |
| 1,2-Dichloropropane | 5 | 5 | 125 |
| Bromodichloromethane | 5 | 5 | 125 |
| Dibromomethane | 5 | 5 | 125 |
| Toluene | 5 | 5 | 125 |
| 1,1,2-Trichloroethane | 5 | 5 | 125 |
| Tetrachloroethene | 5 | 5 | 125 |
| 1,3-Dichloropropane | 5 | 5 | 125 |
| Dibromochloromethane (Chlorodibromomethane) | 5 | 5 | 125 |
| 1,2-Dibromoethane (EDB) | 5 | 5 | 125 |
| Chlorobenzene | 5 | 5 | 125 |
| 1,1,1,2-Tetrachloroethane | 5 | 5 | 125 |
| Ethylbenzene | 5 | 5 | 125 |
| m&p-Xylene | 5 | 5 | 125 |
| o-Xylene | 5 | 5 | 125 |
| Styrene | 5 | 5 | 125 |
| Bromoform | 5 | 5 | 125 |
| Isopropylbenzene | 5 | 5 | 125 |
| 1,1,2,2-Tetrachloroethane | 5 | 5 | 125 |
| Bromobenzene | 5 | 5 | 125 |
| 1,2,3-Trichloropropane | 5 | 5 | 125 |
| n-Propylbenzene | 5 | 5 | 125 |
| 2-Chlorotoluene | 5 | 5 | 125 |
| 1,3,5-Trimethylbenzene | 5 | 5 | 125 |
| 4-Chlorotoluene | 5 | 5 | 125 |
| 1,2,4-Trimethylbenzene | 5 | 5 | 125 |
| sec-Butylbenzene | 5 | 5 | 125 |
| tert-Butylbenzene | 5 | 5 | 125 |
| p-Isopropyltoluene | 5 | 5 | 125 |
| 1,3-Dichlorobenzene | 5 | 5 | 125 |
| 1,4-Dichlorobenzene | 5 | 5 | 125 |

| Analyte | RL water (ug/L) | RL soil Low-level (ug/kg) | RL soil Medium-level (ug/kg) |
|-----------------------------|--------------------|---------------------------------|------------------------------------|
| n-Butylbenzene | 5 | 5 | 125 |
| 1,2-Dichlorobenzene | 5 | 5 | 125 |
| 1,2,4-Trichlorobenzene | 5 | 5 | 125 |
| Hexachlorobutadiene | 5 | 5 | 125 |
| Naphthalene | 5 | 5 | 125 |
| 1,2,3-Trichlorobenzene | 5 | 5 | 125 |
| trans-1,3-Dichloropropene | 5 | 5 | 125 |
| cis-1,3-Dichloropropene | 5 | 5 | 125 |
| Acetone | 100 | 100 | 2500 |
| 2-Butanone (MEK) | 25 | 25 | 625 |
| 4-Methyl-2-pentanone (MIBK) | 25 | 25 | 625 |
| Acrolein | 50 | 100 | 2500 |
| Acrylonitrile | 100 | 100 | 2500 |
| 2-Hexanone | 25 | 100 | 2500 |
| Vinyl Acetate | 10 | 100 | 2500 |
| Iodomethane | 10 | 100 | 2500 |
| Methyl tert-butyl ether | 4 | 5 | 125 |
| Carbon Disulfide | 10 | 10 | 250 |
| trans-1,4-Dichloro-2-butene | 100 | 100 | 2500 |
| Ethyl Methacrylate | 100 | 100 | 2500 |

¹Target Compounds and Reporting Limits are subject to change.

Table 2: Characteristic Ions of Target Compounds²

| Analyte | Primary Ion | Secondary Ion(s) |
|---|-------------|------------------|
| Dichlorodifluoromethane | 85 | 87 |
| Chloromethane | 50 | 52 |
| Vinyl Chloride | 62 | 64 |
| Bromomethane | 94 | 96 |
| Chloroethane | 64 | 66 |
| Trichlorofluoromethane | 101 | 103 |
| Methylene Chloride | 84 | 86, 49 |
| 1,1-Dichloroethene | 96 | 61, 63 |
| trans-1,2-Dichloroethene | 96 | 61, 98 |
| 1,1-Dichloroethane | 63 | 65, 83 |
| 2,2-Dichloropropane | 77 | 97 |
| cis-1,2-Dichloroethene | 96 | 61, 98 |
| Chloroform | 83 | 85 |
| Bromochloromethane | 49 | 128 |
| 1,1,1-Trichloroethane | 97 | 99, 61 |
| Carbon Tetrachloride | 117 | 119, 121 |
| 1,1-Dichloropropene | 75 | 110, 77 |
| Benzene | 78 | 52, 77 |
| 1,2-Dichloroethane | 62 | 98, 64 |
| Trichloroethene | 95 | 97, 130, 132 |
| 1,2-Dichloropropane | 63 | 62, 112 |
| Bromodichloromethane | 83 | 85, 127 |
| Dibromomethane | 93 | 95, 174 |
| Toluene | 91 | 92 |
| 1,1,2-Trichloroethane | 83 | 97, 85 |
| Tetrachloroethene | 166 | 129, 168 |
| 1,3-Dichloropropane | 76 | 78 |
| Dibromochloromethane (Chlorodibromomethane) | 129 | 127 |
| 1,2-Dibromoethane (EDB) | 107 | 109 |
| Chlorobenzene | 112 | 77, 114 |
| 1,1,1,2-Tetrachloroethane | 131 | 133, 119 |
| Ethylbenzene | 106 | 91 |
| m&p-Xylene | 106 | 91 |
| o-Xylene | 106 | 91 |
| Styrene | 104 | 78 |
| Bromoform | 173 | 175, 254 |
| Isopropylbenzene | 105 | 120 |
| 1,1,2,2-Tetrachloroethane | 83 | 131, 85 |
| Bromobenzene | 77 | 156, 158 |
| 1,2,3-Trichloropropane | 75 | 77, 110 |
| n-Propylbenzene | 91 | 120 |
| 2-Chlorotoluene | 91 | 126 |
| 1,3,5-Trimethylbenzene | 105 | 120 |
| 4-Chlorotoluene | 126 | 91 |
| 1,2,4-Trimethylbenzene | 105 | 120 |
| sec-Butylbenzene | 105 | 134 |
| tert-Butylbenzene | 119 | 91, 134 |
| p-Isopropyltoluene | 119 | 134, 91 |
| 1,3-Dichlorobenzene | 146 | 111, 148 |
| 1,4-Dichlorobenzene | 146 | 111, 148 |
| n-Butylbenzene | 91 | 92, 134 |

| Analyte | Primary Ion | Secondary Ion(s) |
|-----------------------------|-------------|------------------|
| 1,2-Dichlorobenzene | 146 | 111, 148 |
| 1,2,4-Trichlorobenzene | 180 | 182, 145 |
| Hexachlorobutadiene | 225 | 223, 227 |
| Naphthalene | 128 | 127 |
| 1,2,3-Trichlorobenzene | 180 | 182, 145 |
| trans-1,3-Dichloropropene | 75 | 77 |
| cis-1,3-Dichloropropene | 75 | 77 |
| Acetone | 43 | 58 |
| 2-Butanone (MEK) | 43 | 57, 72 |
| 4-Methyl-2-pentanone (MIBK) | 43 | 58, 85 |
| Acrolein | 56 | 55 |
| Acrylonitrile | 53 | 52, 51 |
| 2-Hexanone | 43 | 58, 100 |
| Vinyl Acetate | 43 | 86 |
| Iodomethane | 142 | 127 |
| Methyl tert-butyl ether | 73 | 57 |
| Carbon Disulfide | 76 | 78 |
| trans-1,4-Dichloro-2-butene | 53 | 88, 75 |
| Ethyl Methacrylate | 69 | 99, 114 |
| Surrogates | Primary Ion | Secondary Ion(s) |
| Dibromofluoromethane | 113 | 111 |
| Toluene-d8 | 98 | 99, 100 |
| 4-Bromofluorobenzene | 95 | 174, 176 |
| Internal Standards | Primary Ion | Secondary Ion(s) |
| Fluorobenzene | 96 | - |
| Chlorobenzene-d5 | 117 | 82, 119 |
| 1,4-Dichlorobenzene-d4 | 152 | 115, 150 |

²Target compounds subject to change.

STANDARD OPERATING PROCEDURE

ANALYSIS OF WHOLE AIR SAMPLES FOR VOLATILE ORGANIC COMPOUNDS BY GC/MS

Reference Methods: EPA Compendium Method TO-15/TO-14

| | |
|-------------------|-------------------------|
| Local SOP Number: | S-MN-A-013-Rev.14 |
| Effective Date: | Date of Final Signature |
| Supersedes: | S-MN-A-013-Rev.13 |

APPROVALS


Laboratory General Manager

4/2/2014
Date


Laboratory Quality Manager

07 Apr 2014
Date

PERIODIC REVIEW

SIGNATURES BELOW INDICATE NO CHANGES HAVE BEEN MADE SINCE PREVIOUS APPROVAL.

| | | |
|-----------|-------|------|
| Signature | Title | Date |
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| Signature | Title | Date |
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| Signature | Title | Date |
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1. PURPOSE/IDENTIFICATION OF METHOD

- 1.1. The purpose of this Standard Operating Procedure (SOP) is to provide quality control and analytical guidance for the analysis of whole air samples and soil vapor samples contained in Summa ® passivated canisters, Silco ® lined canisters (or equivalent), or sampling bags using gas chromatography/mass spectrometry. This SOP is based on Environmental Protection Agency (EPA) Compendium Method TO-15/14.

2. SUMMARY OF METHOD

- 2.1. Samples are received in Summa ® canisters or Silco ® lined canisters (or equivalent). The gauge pressure upon arrival is measured and recorded. The canister is then pressurized to 5 psi gauge pressure using an inert gas. The canister is connected to an autosampler tree, which concentrates the sample prior to injection into a GC/MS. The data is then analyzed for the desired volatile organic compounds.
- 2.2. This method addresses an extensive set of VOCs by incorporating a multisorbent, dry purge technique for water management.
- 2.3. An aliquot of the whole air sample is concentrated prior to gas chromatographic (GC) separation and mass spectrometry (MS) full scan detection. Samples expected to contain VOCs in a range of 0.1 parts per billion by volume (ppbv) to 500 ppbv can be analyzed by this technique.
- 2.4. If samples are received in sampling bags, see section 7.0 for appropriate holding times and actions to transfer the samples to a Summa ® canister to complete analysis as described in this SOP.

3. SCOPE AND APPLICATION

- 3.1. Personnel: The policies and procedures contained in this SOP are applicable to all personnel involved in the analytical method.
- 3.2. Parameters: This procedure is designed to analyze whole air samples collected in Summa ® canisters, Silco ® lined canisters (or equivalent), or sampling bags for some of the volatile organic compounds (VOCs), or hazardous air pollutants (HAPs), found in Title III of the Clean Air Act Amendments of 1990. This SOP is related to only those VOCs that have been found to be stable when collected in Summa ® polished stainless steel canisters, Silco ® lined canisters or sampling bags (or equivalent). VOCs are defined as organic compounds having a vapor pressure greater than 10-1 Torr. Attachment I lists target VOCs applicable to this method.
- 3.3. This SOP is based on the EPA Compendium Method TO15 which can also be applied to TO14. As such, this SOP serves to cover both analyses. See EPA Compendium Method TO15 Section 3 and Attachment I for compound list.

4. APPLICABLE MATRICES

- 4.1. This SOP is applicable to whole air samples and soil vapor samples contained in Summa ® passivated canisters, Silco ® lined canisters (or equivalent), or sampling bags using gas chromatography/mass spectrometry.

5. LIMITS OF DETECTION AND QUANTITATION

- 5.1. The most current reporting and detection limits can be found in the Laboratory Information Management System (LIMS).

6. INTERFERENCES

- 6.1. Carrier gas potentially contains small amounts of contaminants and is filtered prior to use in instrumentation. Other interferences are sample specific and are dealt with as they occur.

- 6.2. Interferences in samples can result from contamination of the canisters. To minimize this problem, processes must be implemented to ensure that the canisters are contamination free. See SOP S-MN-A-004 - Procedure for Cleaning, Certification, Leak Checking, and Preparation for Shipment of SUMMA Passivated Canisters, or equivalent replacement.
- 6.3. Contamination of analytical equipment can also occur when samples containing high concentrations of VOCs are analyzed. The resulting "carryover" contamination varies from system to system. The analyst needs to use best judgment when evaluating sample data following samples with large detection levels.

7. SAMPLE COLLECTION, PRESERVATION, SHIPMENT AND STORAGE

7.1. Collection, Preservation, Storage and Holding Time Table

- 7.1.1. The holding time indicated below is the maximum allowable time from collection to analysis per the analytical method. If the samples fail to meet the holding time, data will be qualified accordingly on the analytical checklist and on the final report with the appropriate footnote.
- 7.1.2. Note For Ohio VAP: As applicable, to the best of the laboratory's knowledge, if holding times are not met the laboratory will qualify the data accordingly indicating the bias present due to the exceedance.

| Sample type | Collection per sample | Preservation | Storage | Hold time |
|-------------|--|--------------|------------------------|---|
| Air | Samples are collected into evacuated Summa ® canisters, Silco ® canisters (or equivalent). The canisters are then shipped back to Pace Analytical Services, Inc. for analysis. | None | Ambient sample storage | <p>Samples collected in Summa ® canisters, Silco ® canisters (or equivalent) must be analyzed within 28 days from collection.</p> <p>Samples collected in Minnesota are to be collected in canisters and must be analyzed within 14 days of collection per the Minnesota Pollution Control Agency (MPCA).</p> <p>If samples have been collected in bags, the samples need to be transferred to a Summa Canister within 48 hours to maintain a 28 day holding time. The holding time is potentially extended to 72 hours per client specific QAPPS. Collection in a bag results in higher reporting limits. See Attachments VIII-X for instructions and documentation for the transfer procedure.</p> <p>Ohio VAP samples must be transferred to a Summa Canister within 48 hours from collection to extend the holding time of collection to analysis to 28 days.</p> |

8. DEFINITIONS

- 8.1. Definitions of terms found in this SOP are described in the Pace Analytical Services Quality Manual, Glossary Section.

- 8.2. Absolute canister pressure = $P_g + P_a$, where P_g = gauge pressure in the canister (kPa, psig) and P_a = barometric pressure.
- 8.3. Absolute pressure - Pressure measured with reference to absolute zero as opposed to atmospheric pressure, usually expressed as kPa, mm Hg or psia.
- 8.4. Cryogen - A refrigerant used to obtain very low temperatures for sample concentration. A typical cryogen is liquid nitrogen (bp - 195.8°C).
- 8.5. Dynamic calibration - Calibration of an analytical system using calibration gas standard concentrations in a form identical or very similar to the samples to be analyzed and by introducing such standards into the inlet of the sampling or analytical system in a manner very similar to the normal sampling or analytical process.
- 8.6. Gauge pressure - Pressure measured above ambient atmospheric pressure as opposed to absolute pressure. Zero gauge pressure is equal to ambient atmospheric (barometric) pressure.
- 8.7. MS-SCAN - The GC is coupled to a MS programmed in the SCAN mode to scan all ions repeatedly during the GC run. As used in the current context, this procedure serves as a qualitative identification and characterization of the sample.
- 8.8. MS-SIM - The GC is coupled to a MS that is programmed to scan a selected number of ions repeatedly.
- 8.9. Qualitative accuracy - The ability of an analytical system to correctly identify compounds.
- 8.10. Quantitative accuracy - The ability of an analytical system to correctly measure the concentration of an identified compound.

9. EQUIPMENT AND SUPPLIES (INCLUDING COMPUTER HARDWARE AND SOFTWARE)

9.1. Equipment and Supplies Table

| Supply | Description | Vendor/ Item # / |
|---|---|----------------------------------|
| Gas Tight Syringes | 0.010, 0.025, 0.05, 0.1, 0.25, 0.5, 1, 5, and 10 mL | Fisher, or equivalent |
| Neat liquid standards | at least 95% | O2Si, or equivalent |
| Glass static dilution flask | 2L, equipped with a Mini-inert cap | Fisher, or equivalent |
| Oven | capable of maintaining a temperature of 65°C | Fisher, or equivalent |
| Summa ® passivated canisters or Silco ® lined canisters (or equivalent) | 6L or 15L capacity | Restek |
| Dual pressure/vacuum gauge | high accuracy | Omega Engineering, or equivalent |
| Nitrogen | | Praxair |
| Organic free water | DI Water | n/a |
| Gas Chromatograph | Equipped with a split/splitless injection port and electronic pressure control (EPC) or equivalent. See 9.1.1 and 9.1.2 for operating parameters. | Agilent Technologies 6890N |
| Mass Selective Detector | With Chemstation operating software and WinTarget data processing software or equivalent. See 9.1.3 for operating parameters. | Hewlett Packard 5973 |
| Pre-concentrator | With 7016 canister manifold autosampler. See 9.1.4 for operating parameters. | Entech 7100A |

| | | |
|------------------|---|-------------------------------------|
| Capillary Column | DB-5 60m x 0.32mm capillary column or DB-624 60m x 0.32mm with a 1.8 µm film thickness or equivalent. | J & W Scientific |
| Helium Cylinder | High purity grade high-pressure helium cylinder for column carrier gas equipped with a dual stage pressure regulator. | Praxair |
| Chemstation | Data Acquisition Software | See master list for current version |
| Target | Data Processing Software | See master list for current version |
| EPIC Horizon | Data Reporting Software (LIMS) | See master list for current version |
| Gandalph | Data Packaging Software | See master list for current version |

9.1.1. Chromatograph Suggested Operating Parameters:

- 9.1.1.1. Initial temp: 40°C for 2.0 min.
- 9.1.1.2. Ramp A: 8°C/min to 150°C
- 9.1.1.3. Ramp B: 15°C/min to 200°C
- 9.1.1.4. Hold 2 min
- 9.1.1.5. EPC Pressure: 9 psi
- 9.1.1.6. Temp 250°C
- 9.1.1.7. Split Flow 20mL/min

9.1.2. Injection port parameters:

- 9.1.2.1. EPC pressure: 9 psi
- 9.1.2.2. Temperature: 250°C
- 9.1.2.3. Purge valve: Initial value On, Off time 0.0 min.
- 9.1.2.4. Split flow: 20 mL/min.

9.1.3. Suggested Mass spectrometer parameters:

- 9.1.3.1. Electron volts: 70 nominal
- 9.1.3.2. Scan range: 29 to 300 amu
- 9.1.3.3. Scan time: At least 2 scans/peak, not to exceed 1 sec/scan
- 9.1.3.4. Interface temp: 250°C
- 9.1.3.5. The GC/MS system must be set up to meet manufacturer's specification. The mass calibration and resolution of the GC/MS are verified by the analysis of the tune standard, p-bromofluorobenzene (BFB). For more information refer to the Chemsystem User's Guide and the GC/MS User's Guide.

9.1.4. Entech Pre-Concentrator suggested settings:

9.1.4.1.

| During Concentration | Temperature (°C) |
|---------------------------------------|------------------|
| Module No. 1, Glass Bead Cryotrap | -150 |
| Module No. 2, Sorbent Packed Cryotrap | -20 |
| Focusing Trap | -160 |

9.1.4.2.

| Desorb/Transfer/Inject | Preheat (°C) | Final Temp(°C) |
|------------------------|--------------|----------------|
|------------------------|--------------|----------------|

| | | |
|---------------------------------------|-----|-----|
| Module No. 1, Glass Bead Cryotrap | 10 | 10 |
| Module No. 2, Sorbent Packed Cryotrap | 50 | 180 |
| Focusing Trap | N/A | N/A |

9.1.4.3.

| Media Concentrated/Transferred | Volume (cc) | Flow Rate (sccm) |
|--------------------------------|-------------|------------------|
| Internal Standard & Surrogate | 50 | 200 |
| Sample | 25 to 500 | 250 |
| Sweep/Dry Purge | 75 | 100 |
| Transfer to Packed Column | 40 | 10 |

9.1.4.4. Sample Transfer

| | |
|--|-------------|
| Line Conditioning Sample Flush Before Trapping | 20 sec |
| Carrier Flush Before Trapping | 2 to 4 min. |
| Sample Transfer to Focusing Trap | 2 to 4 min. |
| Sample Injection | 2 to 5 min. |

9.1.4.5.

| System Bakeout | Temperature (°C) | Time (min.) |
|----------------|------------------|-------------|
| Module No. 1 | 150 | 10 |
| Module No. 2 | 190 | 10 |

9.1.4.6.

| Regulated Zones | Temperature (°C) |
|--------------------------|------------------|
| 8-Port Valve | 100 |
| GC Transfer Line | 110 |
| Manifold Transfer Line | 100 |
| 16-Position Select Valve | 100 |
| Sample Container | Ambient |

10. REAGENTS AND STANDARDS

10.1. Target analyte standards are obtained from various vendors and verified for accuracy.

10.1.1. Calibration Mix is used for Initial Calibration (ICAL), Continuous Calibration (CCAL) and Laboratory Control Spike (LCS). Second Source is used for initial calibration verification. Surrogate, Tuning and Internal standard solutions are obtained from vendors in solution form. These solutions are stored per manufacturer's specifications, and have an expiration date of one year after being opened or the manufacturer's expiration date if that date is prior to the one year date.

10.2. Reagents and Standards Table

| Reagent/Standard | Concentration/Description | Requirements/ Vendor/ Item # |
|----------------------|--|--|
| Calibration Standard | This is a custom mix that includes all compounds of interest at 1ppmv. | The calibration standard is purchased in the form of a pressurized cylinder from a source independent of the second source verification mix (Spectra Gas, Linde, Custom Gas Solutions, or equivalent). |

| | | |
|---|--|--|
| | | |
| Initial Calibration Verification (second source standard) | This is a custom mix that includes all compounds of interest at 1ppmv. | The ICV is purchased in the form of a pressurized cylinder from a source independent of the calibration mix. |
| Internal Standard/ Surrogate/ BFB Standard | Neat standards per each individual component. | The internal, surrogate, and BFB standards are purchased as separate neat standards from specific vendors; such as Chem-Service, Sigma-Aldrich or equivalent. See Section 10.3.5 on preparation of working standard. |

10.3. Working Standard Dilutions and Concentrations

- 10.3.1. All standards prepared into canisters in the air lab will be assigned a 28 day holding time, similar to the air samples. Upon expiration, the expired standard is removed from use and a new standard must be prepared.

| Standard | Standard(s) Amount | Concentration of Std | Solvent | Solvent Volume | Final Total Volume | Final Concentration |
|---|--------------------|----------------------|----------|----------------|--------------------|---------------------|
| 2 PPBV Calibration Standard | 90cc | 1ppmv | Nitrogen | 15 L Can | 30psig | 2 ppbv |
| 20 PPBV Calibration Standard | 900cc | 1 ppmv | Nitrogen | 15 L Can | 30 psig | 20 ppbv |
| Initial Calibration Verification Standard (ICV) (Second Source) | 900 cc | 1ppmv | Nitrogen | 15L Can | 30psig | 20 ppbv |
| Method Blank | value less than RL | na | Nitrogen | 500cc | 500cc | less than RL |
| LCS | 250cc | 20ppbv | na | na | 250cc | 10ppbv |

| Ical Level | Concentration | Calibration Standard Used | Amt of Cal Standard Used |
|------------|---------------|---------------------------|--------------------------|
| Level 1 | 0.10 ppbv | 2.0 ppbv std | 25 cc |
| Level 2 | 0.20 ppbv | 2.0 ppbv std | 50 cc |
| Level 3 | 0.50 ppbv | 2.00 ppbv std | 125 cc |
| Level 4 | 1.00 ppbv | 2.00 ppbv std | 250 cc |
| Level 5 | 10.00 ppbv | 20.00 ppbv std | 250 cc |
| Level 6 | 20.00 ppbv | 20.00 ppbv std | 500 cc |
| Level 7 | 30.00 ppbv | 20.00 ppbv std | 750 cc |

- 10.3.2. Calibration Standard 2 PPBV: Using the 1000cc gas tight syringe, pull 90cc from TO15 Stock Standard Cylinder into a clean, evacuated 15L canister, that has been humidified with 50ul H₂O. Pressurize the canister to 30 psig with clean nitrogen. This yields a final concentration of 2 ppbv. Record the standard ID, date created, analyst initial, canister number, canister volume, stock standard ID, volume used, water volume added, final pressure (psig), final concentration, and expiration date in the Pace Standard Preparation Logbook.
- 10.3.3. Calibration Standard 20 PPBV: Using the 1000cc gas tight syringe, pull 900cc from TO15 Stock Standard Cylinder into a clean, evacuated 15L canister, that has been humidified with 50 µL H₂O. Pressurize the canister to 30 psig with clean nitrogen. This yields a final concentration of 20 ppbv. Record the standard ID, date created, analyst initial, canister number, canister volume, stock standard ID, volume used, water volume added, final pressure (psig), final concentration, and expiration date in the Pace Standard Preparation Logbook.

10.3.4. ICV 20 PPBV: Using the 1000cc gas tight syringe, pull 900cc from TO15 Stock Standard Cylinder into a clean, evacuated 15L canister, that has been humidified with 50ul H₂O. Pressurize the canister to 30 psig with clean nitrogen. This yields a final concentration of 20 ppbv. Record the standard ID, date created, analyst initial, canister number, canister volume, stock standard ID, volume used, water volume added, final pressure (psig), final concentration, and expiration date in the Pace Standard Preparation Logbook.

10.3.5. The ICV (Second Source Standard) is analyzed by injecting 250cc from the 20 ppbv ICV/Second Source standard (see above table)

10.3.6. To prepare Internal Standard/Surrogate/BFB:

| | |
|------------------------|--------|
| Hexane D4 | 239ul |
| Toluene D8 | 195ul |
| Chlorobenzene D5 | 186ul |
| 1,4 Difluorobenzene | 179ul |
| BFB | 201ul |
| 1,4 Dichlorobenzene D4 | 0.277g |

10.3.7. Example:

Barometric Pressure: 29.92
Room Temperature : 24 °C
Flask Temperature : 65 °C
Flask Volume : 2000 mL
Canister Pressure : 30 psig
Canister Volume : 15,000 mL (15 L)
Flask Concentration : 520.015 PPM

The procedure for Internal Standard/Surrogate/BFB standard is outlined in Attachment VI.

10.3.8. Next, pressurize the 15 L canister 30 psig with clean nitrogen. This yields a final concentration of 200 ppbv. Record the standard ID, date created, analyst initial, canister number, canister volume, stock standard ID, volume used, water volume added, final pressure (psig), final concentration, and expiration date in the Pace Standard Preparation Logbook. For each analysis, 25 cc is added during the trapping.

10.3.9. The tune standard, Bromofluorobenzene (BFB), must be 50ng or less on column.

10.3.9.1. The tune standard can be evaluated from the continuing calibration verification (CCV) standard so long as all criteria can be evaluated and met, since the BFB is present in all injections.

10.3.10. Internal standard compounds and surrogate standard compounds are used in the analysis.

10.3.10.1. Internal Standards: 1,4-Difluorobenzene and Chlorobenzene-d₅

10.3.10.2. Surrogates*: Hexane-d₁₄, Toluene-d₈, and 1,2-Dichlorobenzene-d

*Surrogates are not a method requirement and therefore only reported at specific request of the client. Surrogates are not evaluated for Ohio VAP samples.

10.4. Standard Canister Preparation:

10.4.1. Static Dilution Technique

10.4.1.1. Summary: Standard preparation is accomplished by injecting an aliquot of liquid standard cocktail into a static dilution vessel (see 10.3.6). The static dilution vessel is held at a temperature of 60°C. The liquid standard vaporizes and is quickly vented to come to equilibrium. An aliquot is removed and injected into a canister. The canister is then pressurized with nitrogen to a pre-established final pressure.

10.4.1.2. Procedure

- 10.4.1.2.1. The volume of a clean 2L round-bottom flask, modified with a threaded glass neck to accept a Mininert septum cap, is determined by weighing the amount of water required to completely fill up the flask. Assuming a density of 1 g/mL for water, the weight of the flask in grams when filled with water is taken as the volume of the flask in milliliters.
- 10.4.1.2.2. The dried flask is flushed with nitrogen. After a few minutes, the glass neck is immediately capped with a Mininert septum cap.
- 10.4.1.2.3. The flask is placed in a 60°C oven and allowed to equilibrate at that temperature for about 30 minutes. Predetermined aliquots of liquid standards are injected into the flask making sure to keep the flask temperature constant at 60°C.
- 10.4.1.2.4. The contents are allowed to equilibrate in the oven for at least 30 minutes. To avoid condensation, syringes must be preheated in the oven at the same temperature prior to withdrawal of aliquots.
- 10.4.1.2.5. Sample aliquots are then to be taken from the static dilution flask for introduction into a clean, evacuated canister. The canister is then filled to a final predetermined pressure. An aliquot or aliquots totaling greater than 1 percent of the flask volume are to be avoided.
- 10.4.1.2.6. The concentration of each component in the flask is calculated using Equation 1 (see section 14).
- 10.4.1.2.7. The concentration in ppbv of each component in the flask is determined using Equations 2 and 3 (see section 14).
- 10.4.1.2.8. The concentration in ppbv of each compound in the canister can be determined using Equation 4 (see section 14).
- 10.4.1.2.9. Entech Standards Preparation has a database of compounds and their properties. The program does the necessary conversions of units and calculations (equations 1-4) to yield the amounts of neat standard put in the standard cocktail, the amount of cocktail spiked into the 2L flask, and the aliquot taken from the 2L flask to the final canister. This program can be used to make any gas standard from neat liquid standards.
- 10.4.1.2.10. See Attachment VI for a single sheet summary of Air Standard Preparation.

11. CALIBRATION AND STANDARDIZATION

11.1. Calibration Criteria

| Calibration Metric | Parameter / Frequency | Criteria | Comments |
|------------------------|--|---|---|
| Instrument Tune | Before any standard, method blank, or sample analysis can occur using the GC/MS system, it must be demonstrated that the GC/MS is capable of producing compliant spectra when p-bromofluorobenzene (BFB) is analyzed. Attachment II lists the required spectral criteria. The instrument performance check must be analyzed initially and | If the BFB spectrum meets the criteria listed in Attachment II, proceed with standard and sample analysis. If the BFB spectrum fails to meet the criteria listed in Attachment II, the MS must be retuned. Repeated failures potentially indicate the need for MS maintenance such as cleaning the ion source. | The GC/MS is set up according to the manufacturer's specifications. The MS source and mass filter are adjusted by monitoring the mass spectra of Perfluorotributylamine (PFTBA). Prepare a standard solution of BFB at a concentration that allows the collection of 50ng or less under the optimized concentration parameters (see Section 10.3). This is met by injecting 25 cc during the trapping. The BFB is introduced into the system through |

| | | | |
|---|---|---|--|
| | once every 24-hour period. The tune period begins at the time of injection of the BFB. | | <p>microscale purge and trap.</p> <p>The spectrum of BFB must be acquired by averaging three scans; the apex and the scans that immediately proceed and follow the apex.</p> <p>Background subtraction is accomplished using a single scan taken before the BFB peak.</p> |
| Initial Calibration (ICAL) | <p>All standards, method blanks, laboratory control spikes (LCS), and samples must be analyzed using the same conditions. A calibration curve must consist of a minimum of 5 standards (6 for quadratic) and spans the expected monitoring range established for each compound of interest to determine instrument response and linearity. The lowest level of the curve must be at or below the reporting limit for each analyte. A typical calibration curve can cover a range from 0.1 to 20 ppbv. Section 10.3 contains standard preparation information.</p> | <p>The %RSD for all calibrated target compounds must be $\pm 30\%$. Alternately for all target compounds, linear regression is used with an r^2 value of 0.995 or greater. A quadratic curve is utilized if the r^2 (equals COD in Equation 12) value is 0.990 or greater and six calibration points are included in the curve. Curves must not be forced through zero. <i>For Ohio VAP: quadratic curve fit is only to be used for analytes that have historically exhibited nonlinear response.</i></p> <p>The area response for each internal standard in each calibration level must be within 40% of the mean area response over the calibration range. The relative retention time (RRT) of each compound must agree within ± 0.06 RRT units of the average RRT from the initial calibration curve.</p> <p>Per the Pace Quality Assurance Manual, the reporting limit standard must be evaluated to determine if the curve fit is presenting bias. The level corresponding to the reporting limit must be quantitate back after processing the curve and be $\pm 40\%$ of the expected true value.</p> <p>See section 11.2 for corrective actions.</p> | <p>Initial calibrations are not meant to be a replacement of necessary instrument maintenance. Calibration curve fits are possible indicators of instrument performance or deterioration. Analytes that traditionally are average or linear responders that suddenly display quadratic curve fits could be a sign of a system that is deteriorating. Quadratic cannot be used to extend the calibration range for compounds that normally exhibit a linear response, perform necessary maintenance to return the system to good working order. This is not to eliminate the use of quadratic curve fits, some analytes always present a quadratic response and that is acceptable. If an analyte fails to meet ICAL criteria, the analyst should report sub list of compounds and/or re-analyze samples under compliant conditions.</p> <p>Calibration is performed using the internal standard technique. See Attachment III for internal standard groups. The data is evaluated using WinTarget. See section 11.2 for acceptance criteria.</p> |
| Initial Calibration Verification (ICV) | A second source standard must be analyzed following an initial calibration curve which contains all the analytes of interest. | <p>The spike level of the ICV must be near the midpoint level of the calibration curve. The ICV is considered acceptable if the recoveries of the analytes fall within 60-140%. If ICV fails criteria, the analyst must consult with his or her supervisor or manager before</p> | <p>The ENTECH 7000 Concentrator automatically adds 25 cc of the internal standards and surrogates (Section 10.3) to each analysis during trapping.</p> <p>Using the Target data processing software, evaluate the calibration data.</p> |

| | | | |
|--|---|---|---|
| | | <p>moving forward. Possible corrective actions include:</p> <ul style="list-style-type: none"> • The system and standards must be evaluated for potential problems. If a problem is isolated and corrected, attempt to run a second ICV. If the second attempt also does not meet criteria, perform further necessary troubleshooting and maintenance. • Check pressure on the standard canister • Check system for leaks. • Check to see that standards were made correctly. | |
| Continuing Calibration Verification (CCV) | <p>After an acceptable tune has been evaluated, the initial calibration curve for each compound of interest must be checked and verified before sample analysis can occur each day. This is accomplished by analyzing continuing calibration verification (CCV) standard at 10 ppbv (see section 10.3 Ical Level 5). The CCV is the same source as the ICAL standard.</p> <p>The CCV is analyzed after a compliant tune once every 24-hour period during sample analysis.</p> | <p>The %D for each target compound in the continuing calibration verification (CCV) standard must be less than or equal to 30 percent.</p> <p>The RRT of each compound must agree within ± 0.06 RRT units of the average RRT from the initial calibration curve.</p> <p>See 11.3 for corrective actions.</p> | <p>Calculate the RRF for each target compound from the continuing calibration standard using Equation 5 (see section 14).</p> <p>See 11,3 for corrective action. Note: If a CCV fails biased high and the associated samples are non-detect, the samples may be reported as the high bias has no impact on the non-detect results.</p> |

11.2. Corrective Action for Initial Calibration

- 11.2.1. If the technical acceptance criteria fail for the initial calibration curve, inspect the system for any possible leaks. A high baseline and reduced response potentially indicates a leak.
- 11.2.2. Examine the response factors of each calibration level. If the response factors of all the compounds for one level appear to be significantly different, analyze that same level calibration standard again.
- 11.2.3. If the same results occur after reanalysis, a new standard canister must be made and analyzed.
- 11.2.4. If a leak or other system problem cannot be found, try to clean the ion source or perform column maintenance.
- 11.2.5. No samples can be analyzed until a compliant initial calibration curve has been established and verified against a second source standard or technical justification has been given for analysis to proceed. Technical justification would be if there are samples available that require only a short list of analytes that exclude the failures, analysis may continue for those analytes. Clearly document which elements are not acceptable on the analytical checklist. If sample analysis has occurred prior to the ICAL being evaluated, samples that are unable to be reanalyzed may have to be reported. The client must be contacted and the data results must be clearly qualified indicating the associated ICAL failures.

- 11.2.6. Recalibration must be performed if any major change has been made to the GC/MS system such as replacing the GC column, cleaning the MS source or repair.
- 11.3. Corrective Action for Continuing Calibration Verification
 - 11.3.1. If the CCV does not meet criteria, the system and standards must be evaluated for potential problems. If a problem is isolated and corrected, attempt to run a second CCV. If the second attempt also does not meet criteria, perform further necessary troubleshooting and maintenance.
 - 11.3.1.1. Check pressure on the standard canister.
 - 11.3.1.2. Check system for leaks.
 - 11.3.1.3. Check to see that standards were made correctly.
 - 11.3.1.4. Document all maintenance and corrective action measures taken in the maintenance logbook, run logbook or checklist accordingly based on the actions taken.
 - 11.3.2. If corrective action attempts fail or two consecutive CCV do not meet criteria, then a new calibration curve must be analyzed.
 - 11.3.3. Samples are not to be analyzed until CCV criteria has been met or technical justification as defined in 11.2.5 has been given for the analysis to continue.

12. PROCEDURE

- 12.1. Analytical Sequence
 - 12.1.1. The following is the GC/MS analytical sequence for samples each 24-hour period:
 - 12.1.1.1. Instrument tune using (BFB); see Section 10.3.5
 - 12.1.1.2. Initial Multi-Point Calibration or CCV; see Section 10.2 or 10.3
 - 12.1.1.3. ICV or Laboratory Control Sample (LCS); see Section 10.3
 - 12.1.1.4. Laboratory Method Blank: 500cc of nitrogen from a clean 6L canister.
 - 12.1.1.5. 20 field samples
 - 12.1.1.6. Sample duplicate, minimum of one in 20 samples
 - 12.1.1.7. Any necessary dilutions from previously analyzed samples (see the dilution preparation section of Attachment V).
 - 12.1.1.8. In the event that time remains in the 24 hour tune period, an additional method blank and LCS must be analyzed in order to analyze additional reportable samples.
- 12.2. Sample Analysis
 - 12.2.1. Upon receipt, the canister pressure of each sample is measured and recorded on the canister sample tag.
 - 12.2.1.1. If the canister pressure is less than 5 psig, the canister pressure must be increased before analysis can occur.
 - 12.2.1.1.1. Add clean nitrogen or helium gas to the sample canister. For a six liter canister, 5 psig is the desired final pressure. A one liter canister requires a final pressure of 10 psig for adequate sample volume for analysis.
 - 12.2.1.1.2. Record the final canister pressure on the canister sample tag noting which gas was added. Also, note the information in the final analytical results report.
 - 12.2.1.1.3. Calculate the resultant dilution factor using Equation 16 in section 14.16.
 - 12.2.1.1.4. This dilution factor is applied to Equation 17 in section 14.17.
 - 12.2.2. Once the GC/MS system is demonstrated to be in control, an aliquot of the air sample is removed from the canister and pre-concentrated using the Entech 7100A pre-concentrator and 7016 autosampler manifold.

- 12.2.3. Analyze the samples under the same operating conditions as the instrument calibration and quality control samples.
 - 12.2.4. Analyze a duplicate sample for every 20 samples analyzed.
 - 12.2.5. If time remains in the 24-hour tune period in which an initial calibration was performed, it is possible to continue to analyze samples without the analysis of a CCV standard.
 - 12.2.6. If the tune period has expired, an instrument tune and CCV standard must be analyzed before samples can be analyzed.
 - 12.2.7. If time remains in the tune period after a batch of no more than 20 samples and its re-runs have been analyzed, it is possible to analyze additional samples after a new LCS and method blank have been analyzed.
 - 12.2.8. Technical Acceptance Criteria can be found in Section 12.7.
 - 12.2.9. Procedures for the determination of Air Phase Petroleum Hydrocarbons (APH) can be found on Attachment XII. Reporting of APH can only be conducted per each state or client acceptance of the data, i.e. Ohio VAP only allows the analysis of the TO15 analytes listed in Table 1 and as specified the Pace scope of accreditation. See the most current certificates for the approved analyte lists, any analytes not approved must be clearly indicated on the report and affidavits as being compounds not certified by the VAP program.
- 12.3. Qualitative Analysis
- 12.3.1. The compounds listed in Attachment I are identified by an analyst competent in the interpretation of mass spectra. Sample mass spectrum is compared to the mass spectrum of a standard of the suspected compound. Two criteria must be satisfied to verify the target compound identifications: (1) elution of the sample component at the same GC retention time as the standard component, and (2) correspondence of the sample component and standard component mass spectra.
 - 12.3.2. The relative retention time (RRT) of the sample component must agree within ± 0.06 RRT units of the RRT of the standard component using the CCV standard as reference.
 - 12.3.3. Standard and sample mass spectra are compared using reference spectra obtained on the GC/MS system being used. The mass spectra used for comparison are from the same standard as that being used for RRT comparison. Mass spectral requirements are as follows:
 - 12.3.3.1. All ions present in the standard mass spectra at a relative intensity greater than 10% (most abundant ion in the spectrum equals 100%) must be present in the sample spectrum.
 - 12.3.3.2. The relative intensities of ions specified above must agree within $\pm 20\%$ between the standard and sample spectra.
 - 12.3.3.3. Ions greater than 10% in the sample spectrum but not present in the standard spectrum must be considered and accounted for by the analyst making the comparison. The verification process favors false positive.
 - 12.3.4. Non-target sample components are library searched using the latest NIST library for the purpose of tentative identification. These components are referred to as TICs - Tentatively Identified Compounds) and are noted as such in any final report with a qualifier of "J" unless the client specifies differently. The "J" qualifier indicates an estimated value. Guidelines for identification are as follows:
 - 12.3.4.1. Characteristic ions in the reference spectrum (ions greater than 10% of the most abundant ion) must be present in the sample.
 - 12.3.4.2. The relative intensities of the major ions must agree within $\pm 20\%$.
 - 12.3.4.3. Ions present in the sample spectrum but not in the reference spectrum must be reviewed for background contamination or presence of co-eluting peaks.

- 12.3.4.4. If in the technical judgment of the analyst, no valid identification can be made, the compound is to be reported as an unknown with possible classification, such as hydrocarbon.
- 12.3.4.5. TIC searches are reported only upon client request. These results are considered estimated and do not fall under any scope of accreditation held by Pace.
- 12.4. Identified target analytes are quantitated using the internal standard method using the extracted ion current profile (EICP) area of the characteristic ions of analytes listed in Attachment III. This ion is referred to as the quantitation ion.
- 12.5. The RRF from the initial calibration curve analysis is used to quantitate samples and method blanks. Calculate the concentration of the sample component using Equation 17 in section 14.17.
 - 12.5.1. Additional curve fit equations are in section 14.
- 12.6. The internal standard method of quantitation is also used to determine an estimated concentration for Tentatively Identified Compounds (TIC). The nearest internal standard to the TIC is used as a reference to estimate the concentration of the TIC. If the nearest internal standard exhibits interferences, the next closest internal is used. The estimated concentration is obtained using Equation 17 with the following exceptions:
 - A_x =Total ion chromatogram area of the TIC,
 - A_i =Total ion chromatogram area of the specific internal standard;
 - R_f =1.0Estimated TIC concentrations are flagged with a qualifier of "J" which indicates that the quantitated amount is an estimate. TICs are not considered certified analytes under any scope of accreditation. If TICs are reported, they must be clearly indicated as not being certified analytes under the program that the work is related to.
- 12.7. General Technical Acceptance Criteria
 - 12.7.1. For data to be reported without qualification, the following criteria must be met for all samples, CCVs, method blanks, and laboratory control sample (LCS).
 - 12.7.1.1. The EICP area response for each internal standard must be within $\pm 40\%$ of the EICP area response in the most recent CCV. See Attachment III for a list of analytes and assigned internal standards.
 - 12.7.1.2. The retention time for each of the internal standards must be ± 0.33 minutes of each of the internal standard (IS) retention times in the most recent ICAL 10.0 Standard.
 - 12.7.1.3. Recoveries for surrogate standard compounds (where required) must fall within $\pm 30\%$ of the true value.
 - 12.7.2. If the technical acceptance criteria are not met, samples must be reanalyzed with appropriate batch QC to confirm results under compliant operating conditions. This confirmation is performed by reanalyzing the corresponding QC that was out of range with the samples and if found to be not in range, narrative for bias will be noted, as applicable. See Section 11.2 and 11.3 for corrective action for calibration failures and Section 13.1 for all other samples (including QC).
 - 12.7.3. There will be times in which analytical results are obtained outside the linear range due to highly contaminated sites. Pace makes every attempt to dilute the samples within the linear range of the instrument. If sufficient dilutions cannot be made to achieve results within the linear range because of sample size or concentration, the data will be reported qualified as estimated. If the bias can be noted, it will be qualified accordingly in the final report, i.e. biased high or biased low.

13. QUALITY CONTROL

13.1. QC Table

| QC Sample | Components | Frequency | Acceptance Criteria | Corrective Action |
|--------------------------|--|---|---|--|
| Method Blank (MB) | <p>A clean canister filled with humidified nitrogen is analyzed on the GC/MS system to demonstrate that the system is free of interferences.</p> <p>The Method Blank is prepared in the same manner as any standard or sample and analyzed in the same manner.</p> <p>See Equation 16 for the calculation on how to determine the concentration present.</p> | <p>Analyzed once every 24-hour period or every 20 samples, whichever comes first.</p> <p>The Method Blank is analyzed before samples can be analyzed, and after daily ICAL or CCAL.</p> | <p>An instrument blank analysis is allowed after any sample that has known VOCs present that exceed the upper calibration limit of the method to demonstrate that the system is free of possible carryover effects. When possible, historical data can be used to determine if there are high levels of contaminants present, possibly causing carry over in the system.</p> <p>The method blank must not contain any target analyte at a concentration greater than its reporting limit and must not contain additional compounds with elution characteristics and mass spectral features that interfere with identification and measurement of a method analyte.</p> <p>The internal standard must be within $\pm 40\%$ of the mean area response of the IS in the most recent calibration. The retention time of each of the internal standards must be within ± 0.33 minutes between the method blank and the most recent calibration standard.</p> | <p>If a Method Blank fails acceptance criteria, the source of the contamination must be identified and eliminated.</p> <p>If a source of contamination is corrected, another Method Blank must be prepared and analyzed to verify that the problem has been resolved.</p> <p>However, if the contaminant cannot be eliminated and samples are analyzed, samples containing the same artifact as that found in a method blank must be flagged accordingly.</p> <p>NOTE: For Ohio VAP samples, if the detection is above the reporting limit and corrective actions as listed in this table do not result in acceptable data, the samples must be re-analyzed. If re-analysis is not possible due to depleted sample volume, then contact the client for further instructions. The client can choose to re-submit the sample or have the lab qualify the data and narrate as appropriate. The narrative for any report that includes qualified data must also include a discussion of any bias in the results.</p> |

| | | | | |
|--|---|--|--|--|
| Laboratory Control Sample (LCS) | <p>The laboratory control standard is prepared from the same standard as the calibration standard) as outlined in section 10.3.</p> <p>The LCS standard is from the same source as the ICAL standard.</p> | <p>A LCS must be analyzed once every 24-hour period or every 20 samples, whichever is more frequent.</p> | <p>The percent recovery for each analyte in the LCS must be within the internally generated recovery limits and can be found in the LIMS system.</p> | <p>If a LCS fails to meet the recovery limit criteria, inspect the system for the possibility of a poor sampling.</p> <p>If the LCS fails and no error in sampling was found, preparation and injection of a second analysis can be conducted. If that second analysis fails, the system must be recalibrated and all affected samples must be reanalyzed.</p> <p>If the samples cannot be reanalyzed, qualify the data accordingly with an appropriate footnote on the final report indicating the bias present.</p> <p>For Ohio VAP samples, if the outlier is an analyte of interest and corrective actions as listed in this table do not result in acceptable data, the QC and samples must be re-analyzed. If re-analysis is not possible due to depleted sample volume, then contact the client for further instructions. The client can choose to re-submit the sample or have the lab qualify the data and narrate as appropriate. The narrative for any report that includes qualified data must also include a discussion of any bias in the results.</p> |
| Duplicate Samples | <p>Client provided samples.</p> | <p>Duplicate sample analysis is performed once per 20 samples</p> | <p>The RPD between the sample and the sample duplicate must be < 25%.</p> | <p>If the RPD fails to meet criteria, the instrument must be evaluated to determine if there was an error with the analysis. If there is not evidence of malfunction, the parent sample must be reanalyzed to confirm results. If the data confirms, report the original data and qualify the bias accordingly.</p> <p>Contact the client for further instructions. The client can choose to re-submit the sample or have the lab qualify the data and narrate as appropriate. The narrative for any report that includes qualified data must also include a discussion of any bias in the results.</p> |

| | | | | |
|---------------------------|---|--|---|---|
| Internal Standards | Internal standard is added to every injection at a concentration of 10ppbv. | Aliquot is added to each analysis at the pre-concentrator. | The EICP area response for each internal standard must be within $\pm 40\%$ of the EICP area response in the mid point of the initial calibration. See Attachment III for a list of analytes and assigned internal standards. | <p>Examine the instrument for possible errors or malfunctions and correct any that are discovered. Re-analyze the samples and associated batch QC samples: method blanks, laboratory control spike, and sample duplicates). Report the reanalyzed sample results accordingly.</p> <p>If there is no evidence of error or malfunction, re-analyze the affected QC and samples. If the data confirms, report the original data and qualify the bias accordingly.</p> <p>Unless a matrix interference was detected, Ohio VAP samples must be re-analyzed undiluted.</p> <p>If the outlier corrective actions do not result in acceptable data, the samples must be re-analyzed. The narrative for any report that includes qualified data must also include a discussion of any bias in the results.</p> |
| Surrogate | Labeled compounds that behave similarly to target analytes that are meant to represent the efficiency of the system related to the matrix | <p>Included in each injection per client specific QAPPs.</p> <p>Surrogates will not be injected into any samples or standard/QC solutions for analysis intended for Ohio VAP certified data.</p> | Internally generated control limits. Most current limits are found in the LIMS. | <p>Confirm that there are no errors in the calculations, surrogate solutions, and internal standards. Verify instrument performance.</p> <p>If no problems are found, reprepare and reanalyze the sample.</p> <p>If the reanalysis is within limits and holding times, then report only the reanalysis.</p> <p>If the reanalysis is within limits, but out of hold, then report both sets of data.</p> <p>If the reanalysis is still out of limits, then report both sets of data.</p> |

14. DATA ANALYSIS AND CALCULATIONS

- 14.1. See the most current Quality Manual for calculations
- 14.2. Concentration of each component in the flask (Static Dilution Technique, section 10.4.1)

Equation 1

$$\text{Concentration (mg/L)} = \frac{(V_i)(d)}{V_f}$$

where:

V_i = Volume of liquid neat standard injected into the flask in mL;
 d = Density of the liquid neat standard in mg/mL;
 V_f = Volume of the flask in liters.

Caution: In the preparation of standards by this technique, make sure that the volume of neat standard injected into the flask does not result in an overpressure due to the higher partial pressure produced by the standard compared to the vapor pressure in the flask.

- 14.3. The concentration in ppbv of each component in the flask is determined using Equations 2 and 3 (Static Dilution Technique, section 10.4.1)

- 14.3.1. First determine the volume of the compound as a gas using Equation 2:

Equation 2

$$V = \frac{nRT}{P} \quad \text{where,} \quad n = \frac{(V_i)(d)}{M}$$

where,

V = Volume of injected compound at STP in liters;

n = Moles;

R = Gas constant (0.08206 L-atm/mole °K);

T = Ambient temperature in °K;

P = Ambient pressure in atm;

V_i = Volume of liquid neat standard injected into the flask in mL;

d = Density of the neat standard in g/mL;

M = Molecular weight of the compound in g/mole.

- 14.3.2. Now calculate the concentration in the flask in ppbv using Equation 3:

Equation 3

$$\text{ppbv} = \frac{V}{V_f} (10^9)$$

where:

V = Gas volume of compound as determined in Eq. 8 in liters;

V_f = Volume of static dilution flask in liters.

- 14.4. The concentration in ppbv of each compound in the canister can be determined using Equation 4 (Static Dilution Technique, section 10.4.1)

Equation 4

$$\text{ppbv} = \frac{(V_i)(C_x)}{V_c}$$

where:

V_i = Volume removed from static dilution flask and injected into the canister in liters;

C_x = Concentration of compound x in the static dilution flask in ppbv;

V_c = Final canister volume in liters.

- 14.5. Relative Response Factor (RRF): Tabulate the area response of the primary ion (Attachment III) for each compound and the associated internal standard. Use the internal standard, which has a retention time nearest to the compound of interest. Calculate the relative response factors (RRF) for each compound using Equation 5:

Equation 5

$$\text{Relative Response Factor (RRF)} = \frac{(A_x)(C_i)}{(A_i)(C_x)}$$

where,

A_x =Area of the primary ion for compound x to be measured;

A_i =Area of the primary ion for the internal standard associated with compound x ;

C_i =Concentration of the internal standard in ppbv;

C_x =Concentration of compound x to be measured in ppbv.

- 14.6. Mean Relative Response Factor. Calculate the mean RRF for each compound using the RRF from the five (or six, where $n=6$)-point calibration using Equation 6:

Equation 6

$$\overline{R_f} = \frac{\sum_{n=5} R_f}{n}$$

where,

$\overline{R_f}$ =Average relative response factor;

R_f =Relative response factor from calibration curve;

n =Number of data points.

- 14.7. Standard Deviation ($\sigma_{(n-1)}$).

Equation 7

$$\sigma_{(n-1)} = \sqrt{\sum_{i=1}^n \frac{(x_i - \bar{x})^2}{(n-1)}}$$

- 14.8. %Relative Standard Deviation (%RSD). Using the average RRF from Equation 6 and the standard deviation from Equation 7, calculate the %RSD using Equation 8:

Equation 8

$$\%RSD = \frac{S_{(n-1)}}{\overline{R_f}} \times 100$$

- 14.9. Mean area response for Internal Standard:

Equation 9

$$\overline{y} = \sum_{i=1}^n \frac{y_i}{n}$$

where,

\overline{y} = mean area response

y = Area response for the internal standard for each initial calibration standard

- 14.10. If a linear regression is used, the regression produces the slope and intercept terms for a linear equation according to Equation 10:

Equation 10

$$y = ax + b$$

where:

y = instrument response (peak area or height)

a = Slope of the line (also called the coefficient of x)

x = Concentration of the calibration standard

b = the intercept, do not include the origin (0) as a calibration point

- 14.11. To calculate the sample concentration by the internal standard method using the linear regression equation, use Equation 11:

Equation 11

$$C_s = [(A_s C_{is} / A_{is}) - b] / a$$

where:

A_s = Area of the peak for the target analyte in the sample

A_{is} = Area of the peak of the internal standard

C_s = Concentration of the target analyte in the calibration standard

C_{is} = Concentration of the internal standard

a = Slope of the line (also called the coefficient of C_s)

b = The intercept

- 14.12. To calculate the coefficient of determination (or r²) for a quadratic curve fit, use Equation 12:

Equation 12

$$COD = \frac{\sum_{i=1}^n (y_{obs} - \bar{y})^2 - \left(\frac{n-1}{n-p} \right) \sum_{i=1}^n (y_{obs} - Y_i)^2}{\sum_{i=1}^n (y_{obs} - \bar{y})^2}$$

where:

y_{obs} = Observed response for each concentration from each initial calibration standard

y = Mean observed response from the initial calibration (See equation 6)

Y_i = Calculated response at each concentration from the initial calibration (See Equation 5)

n = Total number of calibration points in the equation, 6 points for quadratic

p = Number of adjustable parameters in the polynomial equation

- 14.13. Calculate the sample concentration by the internal standard method using the quadratic regression by comparing peak heights to the calibration curve.

Equation 13

Regression equation (quadratic):

$$y = ax^2 + bx + c$$

- 14.14. Percent Difference (%D). The % D in the RRF of the daily RRF of an individual compound compared to the mean RRF for that compound in the most recent calibration curve is determined as follows:

Equation 14

$$\%D = \frac{|R_i - R_c|}{R_i} (100)$$

where,

R_i = The average RRF from the initial calibration curve for compound x;

R_c = RRF for compound x from the CCV standard.

- 14.15. Calculate the percent recovery of the LCS using Equation 15:

Equation 15

$$\text{Percent Recovery} = \frac{C_q}{C_a} (100)$$

where:

C_q = Quantitated concentration of compound x in ppbv;

C_a = Actual concentration of compound x in ppbv.

- 14.16. Calculate the resultant dilution factor using Equation 16:

Equation 16

$$DF = (Pf + 14.7) / (Pi + 14.7)$$

Pi = Pressure reading of canister prior to pressurization (psig)

Pf = Pressure reading of canister after pressurization (psig)

DF = Dilution factor

To convert Hg to psig:

Multiply by 0.491559 or divide by 2.036

PSIG reading is converted to One Atmosphere:

One Atmosphere = 14.7 psig = 29.21 inches of Hg

See Attachment V for the application of dilution factors for filling canisters.

- 14.17. Calculate the concentration of the sample component using Equation 17:

Equation 17

$$C_x = \frac{(A_x)(C_i)(D_f)(R_f)}{(A_i)}$$

where:

C_x = Concentration of compound x in ppbv;

A_x = EICP area of the quantitation ion for compound x ;

C_i = Concentration of the internal standard associated with compound x in ppbv;

D_f = Dilution factor from Equation 12 (if no dilution was performed, D_f equals 1.)

A_i = EICP area of the quantitation ion for the internal standard associated with compound x ;

R_f = Average RRF for compound x from the most recent calibration curve.

- 14.18. The RPD between the sample and the sample duplicate can be calculated using Equation 18:

Equation 18

$$RPD = \frac{|A - B|}{(A + B)/2} \times 100$$

Where:

RPD = Relative Percent Difference

A = Sample Value

B = Duplicate Value

14.19. Convert ppbv to $\mu\text{g}/\text{m}^3$ using Equation 19:

Equation 19

Conversion of ppbv to $\mu\text{g}/\text{m}^3$:

$$\frac{(x \text{ ppbv} * MW \frac{g}{mol})}{24.055 \frac{L}{mol}} = y \frac{\mu g}{m^3}$$

MW= Molecular Weight

24.055 L/mol = Molar Volume of an Ideal Gas

$$PV = nRT$$

$$V = \frac{nRT}{P}$$

V= Volume in liters

n= mols of Ideal Gas (1 mol)

R = Ideal gas constant

P = Pressure (in atm)

T = Temperature in Kelvin

$$V = \frac{(1mol) * (0.082 \frac{L * atm}{mol * K}) * 293.15K}{1 atm}$$

$$V = 24.055 \frac{L}{mol}$$

14.20. Preparation of Working TO15 Standard can be calculated using equation 20:

Equation 20

Preparation of Working TO15 Standard:

$$\frac{X}{Y} * C = Z$$

X=Volume (L) spiked from stock

Y= Volume (L) of container

C=Concentration (ppbv) of Stock

Z = Concentration (ppbv) of working standard

15. DATA ASSESSMENT AND ACCEPTANCE CRITERIA FOR QUALITY CONTROL MEASURES

- 15.1. See tables in section 11 & 13.

16. CORRECTIVE ACTIONS FOR OUT-OF-CONTROL DATA

- 16.1. See tables in section 11 & 13.

17. CONTINGENCIES FOR HANDLING OUT-OF-CONTROL OR UNACCEPTABLE DATA

- 17.1. If not specifically listed in the tables in section 11 & 13, the contingencies are as follows. If there is no additional sample volume to perform re-analyses, all data will be reported as final with applicable qualifiers. If necessary, an official case narrative will be prepared by the Quality Manager or Project Manager.
- 17.2. Any data that is reported not meeting method specifications will be qualified accordingly using footnotes in the LIMS or custom qualifiers using the text field. These footnotes will be designated next to the analytes impacted with a letter/number combination with a summary of definition in the footnote section of the final report. Depending on the client data quality objective, an additional case narrative may be included in the final report and the qualifiers will be summarized in that section of the report as well. As indicated throughout the document, Ohio VAP requires the bias be included in the data qualification and associated case narrative.

18. METHOD PERFORMANCE

- 18.1. All applicable personnel must read and understand this SOP with documentation of SOP review maintained in their training files.
- 18.2. Three performance criteria are used to demonstrate method validity which are: (1) method detection limit (MDL), (2) replicate precision, and (3) accuracy - % recovery of LCS.
- 18.3. **Method Detection Limit (MDL) Study:** An MDL study must be conducted annually (per the method) per S-MN-Q-269 (or equivalent replacement), Method Detection Limit Studies for each matrix per instrument.
- 18.4. **Demonstration of Capability (DOC):** Every analyst who performs this method must first document acceptable accuracy and precision by passing a demonstration of capability study (DOC) S-ALL-Q-020 (or equivalent replacement), Training Procedures.
- 18.5. **Periodic performance evaluation (PE)** samples are analyzed periodically to demonstrate continuing competence per SOP S-MN-Q-258 (or equivalent replacement). Results are stored in the Quality office.

19. METHOD MODIFICATIONS

- 19.1. Pace utilizes 1,4-Difluorobenzene and Chlorobenzene-d5 as internal standards. This is a modification from the three recommended internal standards in the method. Pace has demonstrated with MDLs, ICAL/ICV and PTs that this does not impact the data results.
- 19.2. Pace utilizes clean nitrogen for cleaning and filling canisters used for samples and standards. This is a modification from the method use of zero air.
- 19.3. Pace initial calibrations are accepted with average response models, as well as linear and quadratic regression models. Pace utilizes percent drift when analyzing continuing calibrations with regression models. Acceptance criteria utilized is the same as percent difference (+/-30% of the midpoint of the most recent ICAL). This process has been adapted from EPA method 8260B. Refer to section 24.8.

20. INSTRUMENT/EQUIPMENT MAINTENANCE

- 20.1. All maintenance activities are listed daily in maintenance logs that are assigned to each separate instrument.
- 20.2. Refer to the instrument user's manual for instrument maintenance.

21. TROUBLESHOOTING

- 21.1. Not applicable to this SOP.

22. SAFETY

- 22.1. Standards and Reagents: The toxicity and carcinogenicity of standards and reagents used in this method have not been fully defined. Each chemical compound should be treated as a potential health hazard. Reduce exposure by the use of gloves, lab coats and safety glasses. Material Safety Data Sheets (MSDSs) are on file in the laboratory and available to all personnel. Standard solutions should be prepared in a hood whenever possible.
- 22.2. Samples: Take precautions when handling samples. Samples should always be treated as potentially hazardous "unknowns". The use of personal protective equipment (gloves, lab coats and safety glasses) is required when handling samples. In the event a sample container must be opened, it is recommended to perform this in a hood whenever possible.

23. WASTE MANAGEMENT

- 23.1. Procedures for handling waste generated during this analysis are addressed in S-MN-S-003, Waste Handling, or equivalent replacement.
- 23.2. In order to minimize the amount of waste generated during this procedure, analyst should prepare reagents in an amount which may be used in a reasonable amount of time (e.g., before a reagent expires).

24. POLLUTION PREVENTION

- 24.1. The company wide Chemical Hygiene and Safety Manual contains information on pollution prevention.

25. REFERENCES

- 25.1. Pace Quality Assurance Manual- most current version.
- 25.2. National Environmental Laboratory Accreditation Conference (NELAC), Chapter 5, "Quality Systems"- most current version.
- 25.3. The NELAC Institute (TNI); Volume 1, Module 2, "Quality Systems"- most current version.
- 25.4. Department of Defense (DoD) Quality Systems Manual- most current version.
- 25.5. Compendium of Methods for the Determination of Toxic Organic Compounds in Ambient Air, Second Edition; USEPA, January 1999; EPA/625/R-96/010b. Compendium Method TO15.
- 25.6. Compendium of Methods for the Determination of Toxic Organic Compounds in Ambient Air, Second Edition; USEPA, January 1999; EPA/625/R-96/010b. Compendium Method TO14A
- 25.7. MA DEP Air Phase Petroleum Hydrocarbon (APH) method, 12/2009.
- 25.8. Method 8260B: Volatile Organic Compounds by Gas Chromatography/Mass Spectrometry (GC/MS). Section 7.4.5.

26. TABLES, DIAGRAMS, FLOWCHARTS, AND VALIDATION DATA

- 26.1. ATTACHMENT I: Target Compound List

- 26.2. ATTACHMENT II: Required BFB Key Ions and Ion Abundance Criteria
- 26.3. ATTACHMENT III: Characteristic Ions for Target Compounds
- 26.4. ATTACHMENT IV: Calibration of THC as Gas
- 26.5. ATTACHMENT V: Canister Dilution Factors
- 26.6. ATTACHMENT VI: Air Laboratory Standard Preparation Procedures
- 26.7. ATTACHMENT VII: Procedures for Analyzing MPCA Samples
- 26.8. ATTACHMENT VIII: Procedure for Tedlar Bags
- 26.9. ATTACHMENT IX: Tedlar Sign-off Logbook
- 26.10. ATTACHMENT X: Tedlar Bag Transfer Log
- 26.11. ATTACHMENT XI: Common Logbook Abbreviations
- 26.12. ATTACHMENT XII: Determination of Air Phase Petroleum Hydrocarbons (APH)
- 26.13. ATTACHMENT XIII: Training Attendance Record - Unit Conversion of ppbv to $\mu\text{g}/\text{m}^3$
- 26.14. ATTACHMENT XIII: Training Attendance Record - Equation for Preparation of Working Standard

27. REVISIONS

| Document Number | Reason for Change | Date |
|-------------------|--|-----------|
| S-MN-A-013-Rev.12 | <p>Updated to new corporate-issued SOP format</p> <p>Removed incorrect CCV criteria for TO14 – criteria is 30%</p> <p>3.2 – added sampling bags</p> <p>3.3 – updated to Attachment I</p> <p>7.1 table – defined MPCA, added “for the transfer procedure”</p> <p>7.1.1 - added that holding time exceedances will be noted on the analytical checklists and qualified accordingly on the final report</p> <p>9.1 table - added Gandolph software</p> <p>10.3.1 – added</p> <p>10.3.8 and Attachment VI have been updated to 200</p> <p>10.3.8.1 – reworded for clarity</p> <p>10.3.10.2 – clarified who surrogates are reported for.</p> <p>11.1 table – added “method” before “blank” throughout</p> <p>11.1 table (ICAL) – changed spikes to laboratory control spikes (LCS)</p> <p>11.1 table (ICV) - added reference for the equation used to determine the recoveries in the SOP</p> <p>11.1 table (ICV) – Corrective action for ICV has been expanded to mimic that of the CCV which is the actions that would be taken in case of a failure.</p> <p>11.1 table (ICAL) – corrective action comment has been deleted and is covered in the existing 11.2.6 corrective action information.</p> <p>11.1 table (CCV) - added that the CCV is the Ical level 5 standard as defined in 10.3 corrected references to the daily calibration standard to CCV for consistency</p> <p>11.1 – defined RRT and PFTBA, added “for all target compounds”, added “if an analyte fails...”</p> <p>11.1 – changed “MDH rules” to “Pace Quality Assurance Manual”</p> <p>11.1.1 – added</p> <p>11.3.3 – removed “if samples were associated...”</p> <p>11.3.8.1 – defined CCV</p> <p>12.1.1.8 – added “method”</p> <p>12.2.6 – changed “performance check standard” to “tune”</p> <p>12.5 – added “method”</p> <p>12.7.1.2 – defined IS</p> <p>13.1 table – added “method” and “The narrative for any report that includes qualified data must also include a discussion of any bias in the results” throughout.</p> | 26Jul2013 |

| | | |
|-------------------|---|-----------|
| | <p>Comment d3 - 10.2 (calibration standard) - calibration is discussed in section 11.1, it is noted that the CCV is made as described in 9.1.3 Ical level 5 for the CCV. No additional changes in 10.2 were made</p> <p>Comment d4 - the amount of internal/surrogate spiked has been added to sections 10.3.7, and 11.1</p> <p>Comment d10 – 11.2.5 has further indication of the technical justification of reporting with ical failures- this is a rare situation 11.3.1.4 was added to clarify where to document the information.</p> <p>Comment d12 – 11.3.3 - technical justification referenced back to 11.2.5 in the ical section as defined. This should also resolve the comment about VAP acceptance, if the failing analytes were requested analysis of the VAP samples would be conducted on an instrument with the analytes of interest passing criteria.</p> <p>12.2.9 - add a clarifier indicating APH is not allowed by VAP and reference to refer to each state/client QAPP acceptance to cover all the various states that Pace conducts work.</p> <p>12.3.4.5 – added additional clarification of certification for TICS</p> <p>12.4 – defined EICP</p> <p>12.5 – has been corrected to indicate samples are quantitated against the associated initial calibration</p> <p>12.7.2 – reworded for clarity</p> <p>12.7.2 – added “this confirmation is performed by...”</p> <p>12.7.2 comment on corrective action is covered in table 11.1</p> <p>Removed comment on replicate precision completely (it was stated under 18.2), MDLs are defined in a separate SOP</p> <p>13.1 – In corrective action for MB – removed “undiluted” for re-analysis requirement for Ohio VAP samples.</p> <p>13.1 – added “and can be found in the LIMS system”</p> <p>13.1 table (MB and LCS) – added “as listed in this table”</p> <p>13.1 table (LCS) – clarified “if the LCS fails and no...” to be less confusing</p> <p>13.1 table – added “the bias” to duplicate and internal standard</p> <p>13.1 table (internal standards) – added “report the reanalyzed sample results accordingly”. By accordingly we mean those results that passed in the reanalysis.</p> <p>13.1 table (surrogates) – added “Surrogates will not be injected...”</p> <p>Comment d21 – 13.1 table (surrogates) – Removed the reference that Ohio VAP does not require surrogates</p> <p>13.1 table – Deleted repeated statements on outliers and removed Ohio Vap from the corrective actions associated with these sections as it applies to all clients. Added additional clarification to corrective action</p> <p>Comment d19 – 13.1 table (internal standards) – changed to laboratory control sample</p> <p>13.1 LCS C.A. – added “and all affected samples must be reanalyzed”</p> <p>13.1 DUP – removed reference to attachment VII and clarified parent sample to be reanalyzed</p> <p>17.2 added information regarding the placement and display of footnotes/qualifiers for Ohio VAP requirements</p> <p>14.19 Equation 19 added</p> <p>14.20 Equation 20 added</p> <p>19.1 added on the internal standards utilized</p> <p>Updated Attachment VI</p> | |
| S-MN-A-013-Rev.14 | <p>10.3.6/10.3.7 – updated</p> <p>Bolded QC samples in 13.1 table</p> <p>19.1-19.3 – added and revised</p> | 03Apr2014 |

ATTACHMENT I - Target Compound List

| Compound | CAS RN | TO14 compounds |
|---------------------------|------------|----------------|
| 1,1,1-Trichloroethane | 71-55-6 | X |
| 1,1,2,2-Tetrachloroethane | 79-34-5 | X |
| 1,1,2-trichloroethane | 79-00-5 | |
| 1,1-Dichloroethane | 75-34-3 | X |
| 1,1-Dichloroethene | 75-35-4 | X |
| 1,2,4-Trichlorobenzene | 95-63-6 | X |
| 1,2,4-Trimethylbenzene | 95-63-6 | X |
| 1,2-Dibromoethane | 106-93-4 | X |
| 1,2-Dichlorobenzene | 95-50-1 | X |
| 1,2-Dichloroethane | 107-06-2 | X |
| 1,2-Dichloropropane | 78-87-5 | X |
| 1,3,5-Trimethylbenzene | 108-67-8 | X |
| 1,3-Butadiene | 106-99-0 | |
| 1,3-Dichlorobenzene | 541-73-1 | X |
| 1,4-Dichlorobenzene | 106-46-7 | X |
| 4-Ethyltoluene | 622-96-8 | |
| Acetone | 67-64-1 | |
| Acrolein | 107-02-8 | |
| Acrylonitrile | 107-13-1 | |
| Benzene | 71-43-2 | X |
| Benzyl Chloride | 100-44-7 | |
| Bromodichloromethane | 75-27-4 | |
| Bromoform | 75-25-2 | |
| Bromomethane | 74-83-9 | X |
| Carbon Disulfide | 75-15-0 | |
| Carbon Tetrachloride | 56-23-5 | X |
| Chlorobenzene | 108-90-7 | X |
| Chloroethane | 75-00-3 | X |
| Chloroform | 67-66-3 | X |
| Chloromethane | 74-87-3 | X |
| Cis-1,2-Dichloroethene | 156-59-2 | X |
| Cis-1,3-Dichloropropene | 10061-01-5 | X |

ATTACHMENT I (continued)

| Compound | CAS RN | TO14 compounds |
|---------------------------|---------------|-----------------------|
| Cyclohexane | 110-82-7 | |
| Dibromochloromethane | 124-48-1 | |
| Dichlorodifluoromethane | 75-71-8 | X |
| Dichlorotetrafluoroethane | 76-14-2 | X |
| Ethanol | 64-17-5 | |
| Ethyl Acetate | 141-78-6 | |
| Ethyl Benzene | 100-41-4 | X |
| Freon 113 | 76-13-1 | X |
| Heptane | 142-82-5 | |
| Hexachlorobutadiene | 87-68-3 | X |
| Hexane | 110-54-3 | |
| Isopropyl Alcohol | 67-63-0 | |
| M,P Xylene | 106-42-3 | X |
| O-Xylene | 95-47-6 | X |
| Methyl Butyl Ketone | 591-78-6 | |
| Methyl Ethyl Ketone | 78-93-3 | |
| Methyl Isobutyl Ketone | 108-10-1 | |
| Methyl Tert Butyl Ether | 1634-04-4 | |
| Methylene Chloride | 75-0902 | X |
| Napthalene | 91-20-3 | |
| Propylene | 115-07-1 | |
| Styrene | 100-42-5 | X |
| Tetrachloroethene | 127-18-4 | X |
| Tetrahydrofuran | 109-99-9 | |
| Toluene | 108-88-3 | X |
| Trans-1,2-Dichloroethene | 156-60-5 | |
| Trans-1,3-Dichloropropene | 10061-02-6 | X |
| Trichloroethene | 79-01-6 | X |
| Trichlorofluoromethane | 75-69-4 | X |
| Vinyl Acetate | 108-05-4 | |
| Vinyl Chloride | 75-01-4 | X |

*Current reporting limits can be found in Horizon

ATTACHMENT II - Required BFB Key Ions And Ion Abundance Criteria

| Mass | |
|------------------------|---|
| Ion Abundance Criteria | |
| 50 | 8.0 - 40.0 percent of mass 95 |
| 75 | 30.0 - 66.0 percent of mass 95 |
| 95 | base peak, 100 percent relative abundance |
| 96 | 5.0 - 9.0 percent of mass 95 (See note) |
| 173 | less than 2.0 percent of mass 174 |
| 174 | 50.0 - 120.0 percent of mass 95 |
| 175 | 4.0 - 9.0 percent of mass 174 |
| 176 | 93.0 - 101.0 percent of mass 174 |
| 177 | 5.0 - 9.0 percent of mass 176 |

Note: All ion abundances must be normalized to m/z 95, the nominal base peak, even though the ion abundance of m/z 174 may be up to 120 percent that of m/z 95.

ATTACHMENT III - Characteristic Ions For Target Compounds

| Compound | Primary Ion | Secondary Ion(s) | Internal Standard Group |
|---------------------------|-------------|------------------|-------------------------|
| Propylene | 41 | 39 | 1 |
| Dichlorodifluoromethane | 85 | 87 | 1 |
| Chloromethane | 50 | 52 | 1 |
| Dichlorotetrafluoroethane | 85 | 135,87 | 1 |
| Vinyl Chloride | 62 | 64 | 1 |
| 1,3-Butadiene | 54 | 39 | 1 |
| Bromomethane | 94 | 96 | 1 |
| Chloroethane | 64 | 66 | 1 |
| Ethanol | 31 | 45 | 1 |
| Trichlorofluoromethane | 101 | 103,105 | 1 |
| Acetone | 43 | 58 | 1 |
| Isopropyl Alcohol | 45 | 43 | 1 |
| 1,1-Dichloroethene | 61 | 96 | 1 |
| Freon 113 | 101 | 103,151 | 1 |
| Methylene Chloride | 49 | 84,86 | 1 |
| Carbon Disulfide | 76 | 44,78 | 1 |
| Trans-1,2-Dichloroethene | 96 | 61,98 | 1 |
| Methyl Tert Butyl Ether | 73 | 41 | 1 |
| Vinyl Acetate | 43 | 86 | 1 |
| 1,1-Dichloroethane | 63 | 65 | 1 |
| Methyl Ethyl Ketone | 72 | 43 | 1 |
| Hexane | 57 | 41,43 | 1 |
| Cis-1,2-Dichloroethene | 96 | 61,98 | 1 |
| Ethyl Acetate | 43 | 61,70 | 1 |
| Chloroform | 83 | 85,47 | 1 |
| Tetrahydrofuran | 42 | 41,72 | 1 |
| 1,1,1-Trichloroethane | 97 | 99,61 | 1 |
| 1,2-Dichloroethane | 62 | 64 | 1 |
| Benzene | 78 | 77,50 | 1 |
| Carbon Tetrachloride | 117 | 119 | 1 |
| Cyclohexane | 56 | 84,41 | 1 |
| Heptane | 43 | 41 | 1 |
| 1,2-Dichloropropane | 63 | 41,62 | 1 |
| Trichloroethene | 130 | 132,95 | 1 |

ATTACHMENT III (continued)

| Compound | Primary Ion | Secondary Ion(s) | Internal Standard Group |
|-------------------------------|--------------------|-------------------------|--------------------------------|
| Bromodichloromethane | 83 | 85 | 1 |
| Napthalene | 128 | 127 | 1 |
| Methyl Isobutyl Ketone | 43 | 58,100 | 1 |
| Cis-1,3-Dichloropropene | 75 | 39,77 | 1 |
| Trans-1,3-Dichloropropene | 75 | 39,77 | 1 |
| Toluene | 91 | 92 | 1 |
| 1,12-trichloroethane | 97 | 83,61 | 1 |
| Methyl Butyl Ketone | 43 | 58 | 2 |
| Dibromochloromethane | 129 | 127 | 2 |
| 1,2-Dibromoethane | 107 | 109 | 2 |
| Tetrachloroethene | 166 | 164,131 | 2 |
| Chlorobenzene | 112 | 77,114 | 2 |
| Ethyl Benzene | 91 | 106 | 2 |
| M,P,& O Xylene | 91 | 106 | 2 |
| Bromoform | 173 | 171 | 2 |
| Styrene | 104 | 78,103 | 2 |
| 1,1,2,2-Tetrachloroethane | 83 | 85 | 2 |
| 4-Ethyltoluene | 105 | 120,79 | 2 |
| 1,3,5-Trimethylbenzene | 105 | 120 | 2 |
| 1,2,4-Trimethylbenzene | 105 | 120 | 2 |
| 1,3-Dichlorobenzene | 146 | 111,148 | 2 |
| Benzyl Chloride | 91 | 126 | 2 |
| 1,4-Dichlorobenzene | 146 | 148,111 | 2 |
| 1,2-Dichlorobenzene | 146 | 111,148 | 2 |
| 1,2,4-Trichlorobenzene | 180 | 182,184 | 2 |
| Hexachlorobutadiene | 225 | 227,223 | 2 |
| 1,4-Difluorobenzene | 114 | 88 | IS #1 |
| Chlorobenzene | 117 | 82 | IS #2 |
| Hexane-d14 (surr) | 66 | 64 | 1 |
| Toluene-d8 (surr) | 98 | 100 | 1 |
| 1,4-Dichlorobenzene-d4 (surr) | 150 | 152 | 2 |

ATTACHMENT IV - Calibration of THC as Gas

- IV-1 THC as gas is calibrated by using the same calibration runs that are used for all other compounds, as well as using the same acceptance criteria.
- IV-2 The original calibration files are copied to a target batch. This does not change the raw data in any way, it merely allows the same data to be processed against two different methods
- IV-3 The area response is obtained by summing the area in the total ion chromatogram from the first eluting compound of interest till the end of the run. The internal standard is included as part of this value, the response factor is not calculated using the internal standard method. It is solely based on area response and calibration concentration
- IV-4 The calibration concentration at each level is obtained by summing the values of the individual compounds present in the calibration standard.
- IV-5 A response factor is obtained as detailed earlier in this SOP. Calibration criteria are the same as stated earlier in this SOP.
- IV-6 Custom THC values may be obtained and are noted as such on final reports. These custom values can be based on calibrating using a select list of compounds or a select time frame for example. Requests for these custom values are to be evaluated on an individual basis for analytical feasibility.

ATTACHMENT V - Canister Dilution Factors (6L)

| Initial Pressure Units (inches Hg or PSIG) | Initial Pressure | Initial Pressure Converted to PSIA | Final Pressure (PSIG) | Final Pressure Conver. to PSIA | Dilution Factor |
|--|---------------------|---------------------------------------|--------------------------|--------------------------------------|--------------------|
| Hg | 0 | 14.70 | 5 | 19.7 | 1.34 |
| Hg | -1 | 14.21 | 5 | 19.7 | 1.39 |
| Hg | -2 | 13.72 | 5 | 19.7 | 1.44 |
| Hg | -3 | 13.23 | 5 | 19.7 | 1.49 |
| Hg | -4 | 12.74 | 5 | 19.7 | 1.55 |
| Hg | -5 | 12.24 | 5 | 19.7 | 1.61 |
| Hg | -6 | 11.75 | 5 | 19.7 | 1.68 |
| Hg | -7 | 11.26 | 5 | 19.7 | 1.75 |
| Hg | -8 | 10.77 | 5 | 19.7 | 1.83 |
| Hg | -9 | 10.28 | 5 | 19.7 | 1.92 |
| Hg | -10 | 9.79 | 5 | 19.7 | 2.01 |
| Hg | -11 | 9.30 | 5 | 19.7 | 2.12 |
| Hg | -12 | 8.81 | 5 | 19.7 | 2.24 |
| Hg | -13 | 8.31 | 5 | 19.7 | 2.37 |
| Hg | -14 | 7.82 | 5 | 19.7 | 2.52 |
| Hg | -15 | 7.33 | 5 | 19.7 | 2.69 |
| Hg | -16 | 6.84 | 5 | 19.7 | 2.88 |
| Hg | -17 | 6.35 | 5 | 19.7 | 3.10 |
| Hg | -18 | 5.86 | 5 | 19.7 | 3.36 |
| Hg | -19 | 5.37 | 5 | 19.7 | 3.67 |
| Hg | -20 | 4.88 | 5 | 19.7 | 4.04 |
| Hg | -21 | 4.39 | 5 | 19.7 | 4.49 |
| Hg | -22 | 3.89 | 5 | 19.7 | 5.06 |
| Hg | -23 | 3.40 | 5 | 19.7 | 5.79 |
| Hg | -24 | 2.91 | 5 | 19.7 | 6.76 |
| Hg | -25 | 2.42 | 5 | 19.7 | 8.14 |
| Hg | -26 | 1.93 | 5 | 19.7 | 10.21 |
| Hg | -27 | 1.44 | 5 | 19.7 | 13.69 |
| Hg | -28 | 0.95 | 5 | 19.7 | 20.79 |
| Hg | -29 | 0.46 | 5 | 19.7 | 43.17 |
| PSIG | 1 | 15.7 | 5 | 19.7 | 1.25 |
| PSIG | 2 | 16.7 | 5 | 19.7 | 1.18 |

Canister Dilution Equation:

$$DF = (Pf + 14.7) / (Pi + 14.7)$$

Pi = Pressure reading of canister prior to pressurization
(psig)

Pf = Pressure reading of canister after pressurization (psig)

DF = Dilution factor

To convert Hg to psig:

Divide by 2.036

PSIG reading is converted to One Atmosphere:

One Atmosphere = 14.7 psig = 29.21 inches of Hg

ATTACHMENT V (continued) - Canister Dilution Factors (1L)

| Initial Pressure Units (inches Hg or PSIG) | Initial Pressure | Initial Pressure Converted to PSIA | Final Pressure (PSIG) | Final Pressure Conver. to PSIA | Dilution Factor |
|---|-----------------------------|---|----------------------------------|---|----------------------------|
| Hg | 0 | 14.7 | 10 | 24.7 | 1.68 |
| Hg | -1 | 14.21 | 10 | 24.7 | 1.74 |
| Hg | -2 | 13.72 | 10 | 24.7 | 1.80 |
| Hg | -3 | 13.23 | 10 | 24.7 | 1.87 |
| Hg | -4 | 12.74 | 10 | 24.7 | 1.94 |
| Hg | -5 | 12.24 | 10 | 24.7 | 2.02 |
| Hg | -6 | 11.75 | 10 | 24.7 | 2.10 |
| Hg | -7 | 11.26 | 10 | 24.7 | 2.19 |
| Hg | -8 | 10.77 | 10 | 24.7 | 2.29 |
| Hg | -9 | 10.28 | 10 | 24.7 | 2.40 |
| Hg | -10 | 9.79 | 10 | 24.7 | 2.52 |
| Hg | -11 | 9.30 | 10 | 24.7 | 2.66 |
| Hg | -12 | 8.81 | 10 | 24.7 | 2.80 |
| Hg | -13 | 8.31 | 10 | 24.7 | 2.97 |
| Hg | -14 | 7.82 | 10 | 24.7 | 3.16 |
| Hg | -15 | 7.33 | 10 | 24.7 | 3.37 |
| Hg | -16 | 6.84 | 10 | 24.7 | 3.61 |
| Hg | -17 | 6.35 | 10 | 24.7 | 3.89 |
| Hg | -18 | 5.86 | 10 | 24.7 | 4.22 |
| Hg | -19 | 5.37 | 10 | 24.7 | 4.60 |
| Hg | -20 | 4.88 | 10 | 24.7 | 5.06 |
| Hg | -21 | 4.39 | 10 | 24.7 | 5.63 |
| Hg | -22 | 3.89 | 10 | 24.7 | 6.34 |
| Hg | -23 | 3.40 | 10 | 24.7 | 7.26 |
| Hg | -24 | 2.91 | 10 | 24.7 | 8.48 |
| Hg | -25 | 2.42 | 10 | 24.7 | 10.20 |
| Hg | -26 | 1.93 | 10 | 24.7 | 12.80 |
| Hg | -27 | 1.44 | 10 | 24.7 | 17.17 |
| Hg | -28 | 0.95 | 10 | 24.7 | 26.07 |
| Hg | -29 | 0.46 | 10 | 24.7 | 54.12 |
| PSIG | 1 | 15.7 | 10 | 24.7 | 1.57 |
| PSIG | 2 | 16.7 | 10 | 24.7 | 1.48 |

Canister Dilution Equation:

$$DF = (Pf + 14.7) / (Pi + 14.7)$$

Pi = Pressure reading of canister prior to pressurization (psig)

Pf = Pressure reading of canister after pressurization (psig)

DF = Dilution factor

To convert Hg to psig:

Divide by 2.036

PSIG reading is converted to One Atmosphere:

One Atmosphere = 14.7 psig = 29.21 inches of Hg

ATTACHMENT V (continued) - Canister Dilution Factors (1L)

AIR CANISTER DILUTIONS

When a sample is over the linear range of calibration for a compound of interest, several compounds of interest, or the matrix of the sample interferes with internal standard detections, a dilution is performed.

SYSTEM DILUTION

The pre-concentrator uses a digital mass flow controller to pull volume of the air sample onto the system.

1x = 500cc
2x = 250cc
5x = 100cc
10x = 50cc
20x = 25cc

SERIAL DILUTION

For samples that may require a dilution greater than 20x, the lab performs serial dilutions by emptying the pressurized air in the sample back to ambient conditions (0psig) and refilling the can to 15psig.

This doubles the volume once inside the can and is a 2x

As you multiply this process, the resultant dilution factor is multiplied out.

1. Flush to 0psig fill to 15 = **2x**
2. Flush to 0 and fill again to 15 = **4x**
3. **8x**
4. **16x**
5. **32x**
6. **64x**

ATTACHMENT VI - Air Laboratory Standard Preparation Procedures

CALIBRATION STANDARD

The calibration stock standard is purchased in the form of a pressurized cylinder from SPECTRA GASES, Inc, or equivalent. This is a custom mix that includes all compounds of interest at 1ppmv.

TO15 Standard Preparation

Standards are prepared in a 6L or 15L summa canister that has been evacuated to less than 150 mTorr. The canister is humidified with 50 µl of deionized water. A 1000cc gas tight syringe is filled with a desired volume of TO15 stock standard, depending on the desired final concentration of the summa canister. The summa canister is then pressurized to 30 psig (3 atm) with clean nitrogen from Praxair.

The standard ID, date created, analyst initials, canister number, canister volume, stock standard ID, volume used, water volume added, final pressure in psig, final concentration in ppbv and expiration date are recorded in the standard preparation logbook.

Second Source Verification

The second source stock standard is purchased in the form of a pressurized cylinder from a source independent of the calibration mix (Custom Gas, or equivalent). This includes all compounds of interest at 1ppmv.

The second source standard is prepared in a 6L or 15L summa canister following the same method as the TO15 20.0 ppbv standard.

| Canister Volume (L) | Canister Final Pressure (psig) | Canister Final Pressure (atm) | Canister Pressurized Volume (L) | Standard Volume (cc) | Standard Volume (L) | Final Standard Concentration (ppmv) | Final Standard Concentration (ppbv) |
|---------------------|--------------------------------|-------------------------------|---------------------------------|----------------------|---------------------|-------------------------------------|-------------------------------------|
| 6 | 30 | 3.04 | 18.24 | 36 | 0.036 | 0.002 | 2.00 |
| 15 | 30 | 3.04 | 45.6 | 90 | 0.09 | 0.002 | 2.00 |
| 6 | 30 | 3.04 | 18.24 | 360 | 0.36 | 0.020 | 20.00 |
| 15 | 30 | 3.04 | 45.6 | 900 | 0.9 | 0.020 | 20.00 |

*The Pressurized canister volume can be obtained from Boyle's Law, stating $P_1 V_1 = P_2 V_2$. At 3.04 atm, a 15L cylinder occupies the same volume as a 45.6L cylinder at 1.00 atm. 900 cc of a standard is put into the pressurized canister creating a 0.90L/45.6 L dilution factor to result in the standard to determine the final concentration in ppbv.

Internal Standard/ Surrogate/ BFB Standard 200ppbv:

The internal, surrogate, and bfb standards are purchased as neat standards from specific vendors; such as Chem-Service, Sigma-Aldrich or equivalent. The standard is purchased in a 1.0mL vial with the following components:

| | |
|------------------------|--------|
| Hexane D4 | 239ul |
| Toluene D8 | 195ul |
| Chlorobenzene D5 | 186ul |
| 1,4 Difluorobenzene | 179ul |
| BFB | 201ul |
| 1,4 Dichlorobenzene D4 | 0.277g |

To prepare the internal standards, 25µl of neat standard is added to a clean 2L flask. The flask is then heated at 65°C for 30 min. Following this, the flask is removed from the heat and allowed to cool to ambient temperature. A 20cc aliquot of the volatilized internals is spike from the 2L flask into a clean, humidified and evacuated 15L canister. The 15L canister is then filled to positive 30 psig with clean nitrogen, resulting in a final concentration of 200ppbv for all components. Below is a table summarizing this process:

| Compound | Volume in neat standard (mL) | Density (g/ml) | Mass (g) | Molecular Weight (g/mol) | Molar value | mol in 25 ul | Flask concentration (ppmv) | Final can Concentration (ppbv) |
|------------------------|------------------------------|----------------|----------|--------------------------|------------------------|-------------------------|----------------------------|--------------------------------|
| Chlorobenzene-d5 | 0.186 | 1.157 | 0.215 | 117.59 | 1.83×10^{-03} | 3.679×10^{-05} | 450 | 200 |
| 1,4-difluorobenzene | 0.179 | 1.17 | 0.209 | 114.09 | 1.84×10^{-03} | 3.690×10^{-05} | 450 | 200 |
| BFB | 0.201 | 1.593 | 0.320 | 175 | 1.83×10^{-03} | 3.678×10^{-05} | 450 | 200 |
| 1,4-Dichlorobenzene-d4 | | | 0.277 | 151.03 | 1.83×10^{-03} | 3.686×10^{-05} | 450 | 200 |
| n-hexane-d14 | 0.239 | 0.767 | 0.183 | 100.26 | 1.83×10^{-03} | 3.675×10^{-05} | 450 | 200 |
| toluene-d8` | 0.195 | 0.943 | 0.184 | 100.19 | 1.84×10^{-03} | 3.689×10^{-05} | 450 | 200 |

ATTACHMENT VII - Procedures For Analyzing MPCA Samples

- VII-1 Samples must be carefully monitored for carryover from previous samples with large detections. Analysts and data reviewers need to verify that each analysis has been evaluated for potential carryover.
- If a compound of interest has an on-column concentration that is greater than 10% of the previous sample, it is assumed that this value is not due to carryover.
 - If the compound of interest has an on-column concentration between 2 and 10% of the previous sample, then the analyst carefully examines other factors relating to sample analysis (i.e. the concentration of related components, the overall concentration of constituents in each sample, etc.). When in doubt, the analyst must re-analyze the sample to confirm that the results are not due to carryover.
 - When the compound of interest has an on-column concentration which is less than 2% of the previous sample's concentration, but greater than the method reporting limit, the sample must be analyzed to confirm or eliminate possible carryover.
- VII-2 Sample duplicate analysis must be performed at a minimum of 1 in 10 samples analyzed.
- VII-3 The relative detection limit for MPCA samples is 0.200 ppbv for all analytes except m&p xylene which has a relative detection limit of 0.400 ppbv.

ATTACHMENT VIII - Procedure for Tedlar Bags

Transfer of Tedlar Bags to SUMMA Canisters

In the event that a sample is collected into a tedlar bag, the client has 48 hours to get the bag to the facility for analytical testing. Pace Analytical Services recognizes a 48 hour holding time for all samples collected in tedlar bags. Upon receipt at the laboratory, the sample in the tedlar bag is transferred into a batch certified, evacuated one liter SUMMA canister for analysis. The sample is subsequently analyzed by the appropriate method within 28 days of transfer.

Procedure for transfer:

- Tedlar bag is received and logged for analysis by Pace Analytical Services
- The sample is delivered to the Air Lab, and the laboratory numbers assigned to the sample is recorded in a logbook (as delivered; see Attachment IX).
- The bag is connected to a clean, evacuated canister (105mTorr).
 - The tip of the bag valve is placed into tubing, connected by a 1/4" nut to the sample valve of the canister, secured with a wrench to insure all sample is pulled into the can.
- The bag is opened first. Second, the can is opened.
 - By opening the canister second, the sample is transferred into the can through vacuum (since the can is evacuated to 150mTorr, and the bag is at ambient room pressure).
- After the sample is transferred the sample data and canister number, time and date, is recorded into the transfer logbook (Attachment X).
- Sample is submitted to the laboratory for analysis.
- A data qualifier is added to the report, notifying the client of the transfer.

ATTACHMENT IX - Tedlar Sign-off Logbook (example)



Tedlar Signoff Logbook

[illegible]



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[illegible]

ATTACHMENT XI – Common Logbook Abbreviations

RR
DIL
previously reported sample
CONF
sample
C/O
OK
reported

Reanalysis for previously analyzed sample
Dilution for over-range compounds from a

Confirms results from a previously analyzed

Possible carryover from a prior sample
Analysis is acceptable and sample is

ATTACHMENT XII – Determination of Air Phase Petroleum Hydrocarbons (APH)

This method is designed, based on the Massachusetts APH method, to measure the gaseous-phase concentrations of volatile aliphatic and aromatic petroleum hydrocarbons in air and soil gas. Volatile aliphatic hydrocarbons are collectively quantitated within two carbon number ranges: C₅ through C₈, and C₉ through C₁₂. Volatile aromatic hydrocarbons are collectively quantitated within the C₉ to C₁₀ range. These aliphatic and aromatic hydrocarbon ranges correspond to a boiling point range between approximately 28°C and 245°C. This is a performance-based method. Modifications to this method are permissible, provided that adequate documentation exists, or has been developed, to demonstrate an equivalent or superior level of performance.

Collective Aliphatic/Aromatic ranges: Relative Response Factors are calculated for C₅-C₈ Aliphatic Hydrocarbons and C₉-C₁₂ Aliphatic Hydrocarbons based upon a correlation between the TOTAL mass of aliphatic APH Component Standards eluting within the range of interest and the total ion area count. A Relative Response Factor is calculated for C₉-C₁₀ Aromatic Hydrocarbons based upon a correlation between the TOTAL mass of aromatic APH Component Standards eluting within this range and the total area count of extracted ions 120 and 134. Specified APH Component Standards are designated “marker” compounds to define the beginning and end of the hydrocarbon ranges.

- **C₅ through C₈ Aliphatic Hydrocarbons** are defined as all aliphatic hydrocarbon compounds which elute from isopentane to just before n-nonane (C₉).
- **C₉ through C₁₂ Aliphatic Hydrocarbons** are defined as all aliphatic hydrocarbon compounds which elute from n-nonane to just after 1-methylnaphthalene.
- **C₉ through C₁₀ Aromatic Hydrocarbons** are defined as all aromatic hydrocarbon compounds which elute from just after o-xylene to just after 1-methylnaphthalene, excluding naphthalene and 2-methylnaphthalene, which are quantitated and evaluated separately as Target APH Analytes.

| Hydrocarbon Range | Beginning Marker | Ending Marker |
|--|----------------------------|------------------------------------|
| C ₅ -C ₈ Aliphatic Hydrocarbons | 0.1 min. before isopentane | 0.01 min. before n-Nonane |
| C ₉ -C ₁₂ Aliphatic Hydrocarbons | 0.01 min. before n-Nonane | 0.1 min. after 1-Methylnaphthalene |
| C ₉ -C ₁₀ Aromatic Hydrocarbons | 0.1 min. after o-xylene | 0.1 min. after 1-Methylnaphthalene |

Standard Information: All APH standards are purchased as 30 component mixtures from a known vendor, such as SPEX CertiPrep or O2Si, in methanol.

Initial Calibration (*Suggested Parameters*)

- Standard Concentration: All components 10 ppmv
- Prepare a **20 ppbv** working standard by adding 36cc to a clean, evacuated 6L canister. Fill to 30psig.

Second Source Verification

- Standard Concentration: Components range from 30-70ug/ml
- Prepare working standard by adding 7.2ul to a clean, evacuated 6L canister. Fill to 30psig.

ATTACHMENT XII (continued) – Determination of Air Phase Petroleum Hydrocarbons (APH)

Initial Calibration and SSV Table:

| | Volume (cc) | C5-C8 | C9-C12 | C9-C10 |
|---------------|-------------|-------|--------|--------|
| ICAL-1 | 10 | 5.2 | 6.4 | 2.4 |
| ICAL-2 | 25 | 13 | 16 | 6 |
| ICAL-3 | 50 | 26 | 32 | 12 |
| ICAL-4 | 125 | 65 | 80 | 30 |
| ICAL-5 | 250 | 130 | 160 | 60 |
| ICAL-6 | 500 | 260 | 320 | 120 |
| SSV | 250 | 165 | 145 | 59 |

**all expressed in ppbv*

| Component Mixture | Ions | | |
|-------------------------|---------------|--------------|--------------|
| Compound | CAS NO | Quant | Qual. |
| 1,3-Butadiene | 106990 | 54 | 39 |
| Isopentane | 78784 | 43 | 42 |
| MTBE | 1634044 | 73 | 41 |
| n-Hexane | 110543 | 57 | 41/43 |
| Benzene | 71432 | 78 | 77/50 |
| Cyclohexane | 110827 | 56 | 84/41 |
| 2,3-Dimethylpentane | 565593 | 56 | 43 |
| n-Heptane | 142825 | 43 | 41 |
| Toluene | 108883 | 91 | 92 |
| n-Octane | 111659 | 43 | 85/57 |
| Ethylbenzene | 100414 | 91 | 106 |
| 2,3-Dimethylheptane | 3074713 | 43 | 84/85 |
| m-Xylene | 108383 | 91 | 106 |
| p-Xylene | 106423 | 91 | 106 |
| o-Xylene | 95476 | 91 | 106 |
| n-Nonane | 111842 | 43 | 57 |
| Isopropylbenzene | 98828 | 105 | 120 |
| 1-Methyl-3-ethylbenzene | 620144 | 105 | 120 |
| 1,3,5-Trimethylbenzene | 108678 | 105 | 120 |
| n-Decane | 124185 | 57 | 85 |
| 1,2,3-Trimethylbenzene | 526738 | 105 | 120 |
| p-Isopropyltoluene | 99876 | 119 | 105 |
| Indene | 95136 | 115 | 116 |
| Butylcyclohexane | 1678939 | 83 | 55 |
| n-Undecane | 1120214 | 57 | 42 |
| Naphthalene | 91203 | 128 | 127 |
| n-Dodecane | 112403 | 57 | 43 |
| Hexylcyclohexane | 4292755 | 83 | 82 |
| 2-Methylnaphthalene | 91576 | 142 | 141 |
| 1-Methylnaphthalene | 90120 | 142 | 141 |

**STANDARD OPERATING PROCEDURE FOR
THE TOXICITY CHARACTERISTIC LEACHING PROCEDURE
FOR VOLATILE ORGANIC COMPOUNDS**

Revision Author: Troy Goehl

This SOP is effective upon signed approval by the following:

Troy M. Goehl

4/12/2012

Production Manager

Date

R. L. M.

4/12/2012

QA Director

Date

DISCLAIMER: This SOP has been developed for use at the Microbac Laboratories, Merrillville, Indiana facility. It is intended for use by trained analysts. As written, this SOP may not be specifically applicable to the activities of other organizations.

1.0 TABLE OF CONTENTS

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2.0 SCOPE AND APPLICATION

- 2.1 This is a sample preparation procedure for the extraction of Volatile Organic Compounds. This procedure is applicable to the analysis of aqueous, non-aqueous liquid and solid matrix samples. The applicable analytes, detection limits and routine reporting limits (PQL) are listed at the Analyte page of the applicable test codes in Element LIMS.

3.0 SUMMARY

- 3.1 A ten-gram sample is placed into a zero headspace extractor (ZHE). The ZHE unit is completely filled with extraction fluid as to leave no headspace. The ZHE is then placed on a rotating mixer and the sample tumbled for 18 hours. The sample is then transferred into a VOA vial, leaving zero headspace, and analyzed by GC/MS.
- 3.2 This procedure is based on the reference methods listed in section 17 of this document. This procedure contains the following deviation from the reference methods:
- 3.2.1 The ZHE units used have an internal volume of 200 mL. Section 4.2.1 of the reference method requires units with an internal volume of 500-600 mL. The units used are specifically manufactured for the TCLP extraction. The sample size used is proportionate to that specified in the reference method so that the 20-fold factor is maintained as required in section 7.3 of Method 1311.

4.0 DEFINITIONS

- 4.1 A list of definitions is in the Quality Assurance Plan. In addition to the terms defined in the QAP the term below is specific and critical to this procedure.
- 4.1.1 Storage Blank – An aliquot of lab pure water stored and analyzed with samples at the laboratory. It is a test for contamination in sample storage.

5.0 INTERFERENCES

- 5.1 Not applicable

6.0 SAFETY

- 6.1 Consult the current revision of the Chemical Hygiene Plan. Requirements for the use of personal protective equipment (e.g. safety glasses, lab coats, gloves) as well as other area-specific safety requirements (e.g. gas cylinders) and MSDS sheets are addressed in the CHP.

7.0 EQUIPMENT AND SUPPLIES

The following is a list of materials needed to perform the steps of this procedure as written. See the reference method(s) for equipment and supply specifications.

- 7.1 All volumetric glassware used shall be ASTM Class A. Class B glassware must be verified for accuracy on an annual basis and labeled with an appropriate correction.
- 7.2 Filters, 0.7 micron borosilicate glass fiber filter, 90 mm diameter: Thomas Scientific, #XMCL067 or equivalent.
- 7.3 Lubricant: Archer Precision with Teflon, catalog #64-2301A. Available at stores such as Radio Shack and Menards. Others may be used provided they do not contaminate the samples.
- 7.4 pH meter: Orion 3-star from Thermo Electron Corporation, or equivalent
- 7.5 Screw-top glass jars: 250 mL and 1 L
- 7.6 Thermometer, maximum/minimum registering
- 7.7 Top loading balance with 0.01 g sensitivity: Mettler BB300, or equivalent
- 7.8 Tumbler, capable of rotating at 30 ± 2 rpm: Bodine 42R5BFCI-E3
- 7.9 VOA vials
- 7.10 ZHE units (200 mL): Analytical Testing and Consulting Services, Warrington, PA

8.0 REAGENTS AND STANDARDS

- 8.1 All reagents used must be analytical reagent (AR) grade or higher. All standards must be traceable to NIST, when available. Certificates of traceability must be obtained from the manufacturer. All reagents and standards must be documented in the appropriate preparation logbook. Refer to the requirements in the Labeling of Standards, Reagents, Digestates and Extracts SOP.

8.2 Reagents

All stock reagents are stored in the Metals prep lab unless otherwise noted.

- 8.2.1 Lab pure water (DI water): Analyte free water is prepared as described in the Quality Assurance Plan. DI water may be obtained from any of the designated taps throughout the lab.
- 8.2.2 Sodium hydroxide (NaOH): EMD #SX0590-3 or equivalent
- 8.2.3 Glacial acetic acid, $\text{CH}_3\text{CH}_2\text{OOH}$: Fisher A507-212

- 8.2.4 TCLP Extraction Fluid #1: In a large carboy, dilute the following to 47 L (the "0" line on the carboy) with DI water: 268 mL $\text{CH}_3\text{CH}_2\text{OOH}$ and 120 g of NaOH (dissolve 120 g NaOH in DI water prior to adding it into the carboy). When correctly prepared, the pH of this fluid will be 4.93 ± 0.05 . Verify the pH of this solution before each use. If the fluid was not prepared the day of use, prepare new fluid if the pH is not within the specified range.

9.0 SAMPLE COLLECTION, PRESERVATION, AND HOLDING TIMES

- 9.1 The client or other trained personnel collect samples. Samples received at the laboratory are considered representative unless otherwise noted.
- 9.2 A minimum of 10 g of sample should be collected in a glass container with a Teflon-lined lid and a minimum headspace to prevent the loss of volatiles. Samples should not be opened until immediately prior to analysis. Preservation consists of storage in the range of 0.1-6°C. Samples are stored in the Sample Receipt walk-in cooler. Samples that fail to meet the preservation criteria are noted as such on the Cooler Inspection Report in the Login process.
- 9.3 TCLP leaching must be performed within 14 days of sample collection. Analysis must be performed within 14 days of leaching.

10.0 QUALITY CONTROL

- 10.1 A *Method Blank* must be prepared and analyzed with each batch of maximum 20 samples and at a minimum of one per day. No more than 20 environmental samples may be processed through a given ZHE unit before a method blank must be processed through that unit.
- 10.1.1 The acceptance criteria are < PQL. If the acceptance criteria are not met, reanalyze. If reanalysis fails to meet the acceptance criteria, stop analysis and recalibrate or report data with an appropriate qualifier. Samples for compliance with our Wisconsin DNR certification must be evaluated down to the current MDL and corrective action taken if the blank exceeds the routine PQL.
- 10.1.2 MBLK data that fail to meet the acceptance criteria turn red at DataEntry/Review and are automatically flagged with a "B" qualifier at reporting.
- 10.1.3 The reporting of data associated with a failed control sample must be documented with a CAR form. If the failure is considered to have a significant affect on the data, client notification is required using the Case Narrative of the report.
- 10.1.4 Samples associated with a MBLK that fails with positive bias can be reported without narration if the sample concentration is < PQL or greater than 10 times the blank contamination.

11.0 CALIBRATION AND STANDARDIZATION

- 11.1 The toploading balance must be calibrated/verified in accordance with the Daily Balance Calibration SOP.
- 11.2 The thermometers used must be verified in accordance with the Thermometer Calibration SOP.
- 11.3 Calibrate the pH meter according to the Electrometric Determination of pH SOP.
- 11.4 Tumbler rotation and room temperature (maximum and minimum) are measured each day of use and documented in the TCLP logbook (copy attached).

12.0 PROCEDURE

Analytical data is documented and retained using the TCLP log (copy attached). Analytical data must be maintained in accordance with the document control requirements in the Quality Assurance Plan as well as the Document Control SOP.

12.1 SIZE REDUCTION

- 12.1.1 The solids of the sample must have a surface area less than 3.1 cm^2 . (This is less than 0.375 inches per side.) If the sample is larger than 3.1 cm^2 , size reduction must be performed to alter the physical size of the solids. Use any appropriate physical means available to reduce the size of the sample. This includes, but is not limited to, cutting, tearing, crushing, or grinding. Sieving, or other rigorous techniques, should be avoided due to the possibility that volatiles may be lost. Exposure to the atmosphere should be minimized.
- 12.1.1.1 The surface area criteria are meant for filamentous materials such as paper, cloth, etc. Samples such as PVC pipe, baghouse bags, and plastic sheeting do not require size reduction. Clay-like soils that are not expected to break apart during the tumbling process should be broken apart.

12.2 SOLIDS DETERMINATION

- 12.2.1 Evaluate the sample for % solids. Classify the sample in one of three categories: 100% solid, < 0.5% solid, or > 0.5% and < 100% solid. If there is no visual liquid present, assume 100% solids. If the sample is completely liquid with no particulate, assume < 0.5% solids.
- 12.2.2 The actual % solids must be determined if the sample is neither 100% nor < 0.5% solid. To determine the % solids, weigh and record the weight of a clean 0.45 μm filter.
- 12.2.3 Assemble the filtration device and transfer a minimum of 100 g sample into the device that contains the preweighed filter. Try to cover as much of the filter area with sample as possible.

- 12.2.4 Place a clean 250 mL beaker underneath the device and slowly apply the pressure in increments of 10 psi up to a maximum of 50 psi.
- 12.2.5 When no additional liquid has passed through the filter, release the pressure on the filter device.
- 12.2.6 Weigh the filter and calculate the % solids using the equation in section 13.2.

12.3 SAMPLES HAVING 100% SOLIDS

NOTE: The processing of samples for volatiles analysis must be performed with techniques and forethought to avoid the loss of analyte as well as the potential cross-contamination of the sample and/or laboratory environment. Examples include, but are not limited to, removing the sample for Volatiles analysis from the sample container before any other aliquots are removed (i.e. first), minimizing the time the sample/extract is exposed to air, and potential airborne contamination from the sample/extract.

- 12.3.1 Place a 250 mL glass beaker on the balance.
- 12.3.2 Tare the balance, add approximately 10 g of sample and record the actual amount used.
- 12.3.3 Assemble a ZHE unit. The support screen assembly must contain a 0.7 μ m borosilicate filter between two support screens. Apply a small amount of lubricant to the o-rings.
- 12.3.4 Using 200 mL of the extraction fluid, quantitatively transfer the sample into a ZHE unit.
- 12.3.5 Place the lid on the ZHE unit and tighten the connectors so that no leaking occurs. If leaks occur while applying the pressure, tighten the connectors so to stop the leaking.
- 12.3.6 Attach the ZHE unit to the tumbler.
- 12.3.7 With the release valve in the open position, place a Kimwipe® on top of the valve and, using the handle, twist the drive screw as to raise the piston. Stop applying pressure at the first sign of liquid on the Kimwipe® and immediately close the valve.
- 12.3.8 Turn on the tumbler and allow the ZHE to rotate at $23 \pm 2^{\circ}\text{C}$ for 18 ± 2 hours at 30 rpm.
- 12.3.9 Stop the tumbler after the appropriate extraction time.
- 12.3.10 Open the release valve to verify the presence of pressure. If there is no pressure present, the unit has leaked and the sample must be re-extracted.
- 12.3.11 Using the handle, twist the drive screw and fill the VOA vial.

- 12.3.12 Label the VOA vial with the appropriate sample number.
- 12.3.13 Place the extract in the sample storage cooler in the VOA lab and notify the analyst. The analyst will fill the VOA vial, leaving zero headspace, for analysis.

12.4 SAMPLES HAVING < 0.5% SOLIDS

NOTE: The processing of samples for volatiles analysis must be performed with techniques and forethought to avoid the loss of analyte as well as the potential cross-contamination of the sample and/or laboratory environment. Examples include, but are not limited to, removing the sample for Volatiles analysis from the sample container before any other aliquots are removed (i.e. first), minimizing the time the sample/extract is exposed to air, and potential airborne contamination from the sample/extract.

- 12.4.1 Filter the sample in VOA vials and consider the filtrate as the TCLP extract.
- 12.4.2 Label the VOA vials with the appropriate sample number.
- 12.4.3 Place the filtrate in the sample storage cooler in the VOA lab and notify the analyst.

12.5 SAMPLES HAVING > 0.5% and <100% SOLIDS

NOTE: The processing of samples for volatiles analysis must be performed with techniques and forethought to avoid the loss of analyte as well as the potential cross-contamination of the sample and/or laboratory environment. Examples include, but are not limited to, removing the sample for Volatiles analysis from the sample container before any other aliquots are removed (i.e. first), minimizing the time the sample/extract is exposed to air, and potential airborne contamination from the sample/extract.

- 12.5.1 Calculate the amount of sample to use using the equation in section 13.3.
- 12.5.2 Weigh and record the weight of a clean glass beaker.
- 12.5.3 Assemble a ZHE unit. The support screen assembly must contain a 0.7 μ m borosilicate filter between two support screens. Apply a small amount of lubricant to the o-rings.
- 12.5.4 Transfer the calculated amount of sample into the ZHE unit.
- 12.5.5 Place the lid on the ZHE unit and tighten the connectors.
- 12.5.6 Filter into a 250 mL glass jar and slowly apply pressure to evacuate all liquid from the sample. If leaks occur while applying the pressure, tighten the connectors so to stop the leaking. The filtrate is the liquid phase of the sample. Retain this phase of the sample until the extraction is complete.

- 12.5.7 Weigh the container containing the liquid phase, and calculate the weight of the liquid phase.
- 12.5.8 Remove a small aliquot (~10 mL) of the liquid phase and add an equal volume of DI water. If this results in two separate layers, expect the liquid phase and the extract to not be compatible. This will be important after the extract is obtained.
- 12.5.9 Remove the lid of the ZHE unit and withdraw the piston back to its original position.
- 12.5.10 Remove the filter from the assembly and place it in the ZHE unit. Gently scrape any solid material off of the innermost screen support and add it into the unit.
- 12.5.11 Reprepare the screen assembly with a new filter.
- 12.5.12 Calculate the weight of extraction fluid needed to replace the liquid phase using the equation in section 13.4.
- 12.5.13 Add the solid phase of the sample and the calculated amount of extraction fluid to the ZHE.
- 12.5.14 Place the lid on the ZHE unit and tighten the connectors so that no leaking occurs. If leaks occur while applying the pressure, tighten the connectors so to stop the leaking.
- 12.5.15 Attach the ZHE unit to the tumbler.
- 12.5.16 With the release valve in the open position, place a Kimwipe® on top of the valve and, using the handle, twist the drive screw as to raise the piston. Stop applying pressure at the first sign of liquid on the Kimwipe® and immediately close the valve.
- 12.5.17 Turn on the tumbler and allow the ZHE to rotate for 18 ± 2 hours at 30 rpm.
- 12.5.18 Stop the tumbler after the appropriate extraction time.
- 12.5.19 Open the release valve to verify the presence of pressure. If there is no pressure present, the unit has leaked and the sample must be re-extracted.
- 12.5.20 Filter the sample.
- 12.5.21 Using the handle, twist the drive screw and completely transfer all contents of the ZHE unit to a 1 L glass jar.
- 12.5.21.1 If the extract and original liquid portion of the sample are compatible, combine the liquids.
- 12.5.22 Label the bag with the appropriate sample number.

- 12.6 Place the extract in the sample storage cooler in the VOA lab and notify the analyst NOTE: If the sample is multi-phasic (i.e. the two phases were not compatible), notify the analyst that they will be analyzing separate phases of the sample and notify the project manager so both phases can be logged in. The final concentration of multi-phasic samples is calculated using the equation in section 13.5. The analyst will fill the VOA vial, leaving zero headspace, for analysis.

13.0 **CALCULATIONS AND DATA HANDLING**

- 13.1 After review, enter final results into the LIMS system. Details on the procedure for entering analytical data are in the Analytical Data Entry – Metals SOP.
- 13.2 Calculate the % solids as follows:

$$\% \text{ Solids} = \frac{A - B}{C} \times 100$$

where: A = weight of filter + waste after filtration, g
 B = weight of filter, g
 C = amount of sample used, g

- 13.3 Calculate the amount of sample needed for >0.5% and <100% solids as follows:

$$\text{Sample needed, g} = [(10) (100) / \% \text{ Solids}]$$

- 13.4 Calculate the weight of extraction fluid as follows:

$$\text{Weight needed, g} = [(20) (\% \text{ Solids}) (\text{weight of liquid phase, g})] / 10$$

- 13.5 For multi-phasic samples, use the TCLP multi phase calculator spreadsheet located in the E:\Forms\QC Forms\TCLP multi-phase calculator folder to calculate out the final concentration. The final concentration is calculated as follows based on the analytical results of each phase:

$$\text{Conc., mg/l} = \frac{(V_1) (C_1) + (V_2) (C_2)}{(V_1 + V_2)}$$

Where: V_1 = volume of the first phase, L
 C_1 = concentration of the first phase, mg/L
 V_2 = volume of the second phase, L
 C_2 = concentration of the second phase, mg/L

14.0 METHOD PERFORMANCE

- 14.1 Not applicable

15.0 POLLUTION PREVENTION

- 15.1 The quantity of chemicals purchased should be based on expected usage during their shelf life and the disposal cost of unused material.
- 15.2 Prepare the minimum amount of reagent and standard necessary.

16.0 WASTE MANAGEMENT

- 16.1 Refer to the Sample Disposal SOP for guidance on the disposal of any resulting residue, digestate, distillate, extract or standard.

17.0 REFERENCES

- 17.1 SW-846 Method 1311
- 17.2 Microbac Laboratories Quality Assurance Plan, current revision, all sections

18.0 TABLES, FORMS, CHECKLISTS, AND OTHER ATTACHMENTS

- 18.1 TCLP Preparation Logsheet (1 page)
- 18.2 SOP Revision Notification Form denoting changes made to this revision (1 page)

SECTION 18.1: TCLP PREPARATION LOGSHEET

| Microbac Laboratories - Chicagoland Division TOXICITY CHARACTERISTIC LEACHING PROCEDURE (TCLP) EPA Method 1311 for Volatiles | | | |
|--|--|---|--|
| Sample ID: _____ Date/Time Start Leach: _____ Date/Time End Leach: _____ <small>(extraction time 18±2 hours)</small> | Cont. ID: _____ Analyst: _____ Analyst: _____ | Batch ID: _____ Bal. ID: _____ | |
| Percent Solids Determination Is there obvious liquid? (circle one) Yes No If Yes, proceed with % Solids determination unless there are no obvious solids, then assume <0.5 % Solids. If No, assume 100% Solids. | | | |
| Amount Sample Used: _____ | Filter Wt. (tare): _____ Filter + Sample Wt.: _____ Wt. of Solids: _____ | Container Wt. (tare): _____ Container + Filtrate Wt.: _____ Filtrate Wt.: _____ | Wt. of Filter + Sample after dry: _____ Wt. Dry Solids: _____ |
| % Solids = $\frac{\text{Wt. of Solids}}{\text{Amt. Used}} \times 100$ | | % Dry Solids = $\frac{\text{Wt. of Dry Solids}}{\text{Amt. Used}} \times 100$ | |
| % Solids: _____ | | % Dry Solids: _____ | |
| If % solids or % dry solids < 0.5%, liquid phase is the TCLP extract. Take final pH. If multiphasic, are phases compatible? (circle one) Yes No | | | |
| Size Reduction Obviously < 0.375 inch? (circle one) Yes No Size reduction performed? (circle one) Yes No If Yes, by whom: _____ | | Extraction Fluid Reference #: _____ Fluid pH: _____ (Fluid 1, 4.93±0.05) | |
| Leak Testing ZHE Unit ID: _____ Prior to tumble, did the unit leak when submersed in water? (circle one) Yes No After extraction, did the unit remain pressurized? (circle one) Yes No | | | |
| Extraction Sample Weight Used: _____ Rotator Speed (30±2 rpm): _____ Final Ext. pH: _____ pH Meter ID: _____ | | | |
| Weight of Extraction Fluid: _____ Min/Max Room Temp. (23±2°C): _____ Thermometer Correction Factor: _____ Corrected Min/Max Room Temp.: _____ | | Wt. of Ext. Fluid = $\frac{(20) (\% \text{ Solids}) (\text{Sample Wt.})}{10 \text{ g of solids}}$ | |

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SECTION 18.2: SOP REVISION FORM



SOP REVISION NOTIFICATION FORM FOR:

**THE TOXICITY CHARACTERISTIC LEACHING
PROCEDURE FOR VOLATILE ORGANIC COMPOUNDS**

OLD REVISION: 6

NEW REVISION: 7

SUMMARY OF CHANGES:

- Updated the source for the filters in section 7.2: "Filters, 0.7 micron borosilicate glass fiber filter, 90 mm diameter, ~~MG Thomas Scientific, #F193-12~~ **#XMCL067 or equivalent.**"
- Updated the source for Sodium hydroxide in section 8.2.2: "Sodium hydroxide (NaOH): ~~J.T. Baker #3722-05~~ **EMD #SX0590-3** or equivalent."
- Edited section 10.1.2: "MBLK ~~data~~ that fail to meet the acceptance criteria ~~cause the sample results to be automatically flagged in LIMS turn red at~~ **DataEntry/Review and are automatically flagged** with a "B" **qualifier at reporting.** MBLKs that are below the reporting limit but above the MDL are ~~flagged in LIMS with a "b" qualifier. "b" flagged data is considered as meeting the acceptance criteria.~~
- Edited section 12.6: "Place the extract in the sample storage cooler in the VOA lab and notify the analyst NOTE: If the sample is multi-phasic (i.e. the two phases were not compatible), notify the analyst that they will be analyzing separate phases of the sample ~~and notify the project manager so both phases can be logged in.~~ The final concentration of multi-phasic samples is calculated using the equation in section 13.5. The analyst will fill the VOA vial, leaving zero headspace, for analysis."
- Updated the TCLP Preparation Logsheet in section 18.1.

By initialing below, I certify that I have been *notified* about the approval of a *new revision* to the above mentioned SOP and that I have read, understand, and agree to follow the test procedure as set forth in this new revision.

Initials & Date

Initials & Date

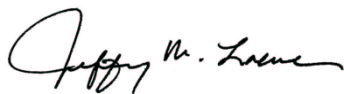
Initials & Date

Form revised 4/5/12

STANDARD OPERATING PROCEDURE FOR PREPARATION OF AQUEOUS SAMPLES USING MANUAL LIQUID-LIQUID EXTRACTION

Revision Author: Christine Robinson

This SOP is effective upon signed approval by the following:



Division Manager

7/13/2012

Date



QA Director

7/13/2012

Date

DISCLAIMER: This SOP has been developed for use at the Microbac Laboratories, Merrillville, Indiana facility. It is intended for use by trained analysts. As written, this SOP may not be specifically applicable to the activities of other organizations.

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2.0 SCOPE AND APPLICATION

- 2.1 This is an extraction procedure for the preparation of samples for Pesticide, Polychlorinated Biphenyl, Total Petroleum Hydrocarbons, Polyaromatic Hydrocarbon, and Semi-Volatile Organic Analytes. This procedure is applicable to the preparation of aqueous and non-aqueous liquid matrix samples.

3.0 SUMMARY

- 3.1 This process involves the isolation and concentration of organic compounds from aqueous samples for a variety of chromatographic techniques.
- 3.2 A measured volume of sample, usually 1 liter, at a specified pH, is serially extracted with methylene chloride using a separatory funnel. The extract is dried, concentrated, and, as necessary, exchanged into a solvent compatible with the cleanup or analytical method used.
- 3.3 This procedure is based on the reference methods listed in section 17 of this document. This procedure contains the following deviations from the reference methods. These deviations are not considered significant deviations from the method.
- 3.3.1 The reference methods were written to use a Snyder column setup for extract concentration. This procedure uses an automated evaporation system (TurboVap) for the concentration of the extracts.
- 3.3.2 The reference method uses 100-ml of Methylene Chloride to extract the sample. This procedure used 60-ml of Methylene Chloride due to the constraints of the 200-ml concentrator tube and to limit out environmental impact by reducing the amount of solvent used in the extraction.
- 3.3.3 EPA Method 604 does a solvent exchange to 2-Propanol prior to analysis. This procedure does not do a solvent exchange for Method 604. The final extracts are in Methylene Chloride. A validation study (found on the S Drive) was performed which showed higher recovery of target analytes on Laboratory Control samples that remained in Methylene Chloride over those that were exchanged to 2-Propanol.

4.0 DEFINITIONS

- 4.1 A list of definitions is in the Quality Assurance Plan. In addition to the terms defined in the QAP, the terms below are specific and critical to this procedure.
- 4.2 Solvent exchange – Adding a different solvent other than the original extraction solvent and evaporating off the original solvent.

5.0 INTERFERENCES

- 5.1 Interferences that co-elute vary considerably from sample to sample.
- 5.2 If the analysis of an extracted sample is prevented due to matrix interferences, further clean-up of the extract may be required.
- 5.3 Phthalate esters can contaminate many types of plasticware and glassware products used in the lab. Plastics, in particular, must be avoided because phthalates are commonly used in plasticizers and are easily extracted from

plastic materials. Phthalate contamination may easily result any time that consistent adherence to the quality control requirements are not practiced.

- 5.4 Soap residue may cause the degradation of certain analytes especially aldrin, heptachlor, and most organophosphorus pesticides. Strict adherence to the Glassware Washing SOP is required.

6.0 **SAFETY**

- 6.1 Consult the current revision of the Chemical Hygiene Plan. Requirements for the use of personal protective equipment (e.g. safety glasses, lab coats, gloves) as well as other area-specific safety requirements (e.g. gas cylinders) and MSDS sheets are addressed in the CHP.

7.0 **EQUIPMENT AND SUPPLIES**

All volumetric glassware used shall be ASTM Class A. Class B glassware must be verified for accuracy on an annual basis and labeled with an appropriate correction.

- 7.1 Autosampler vials and caps, 2 mL
- 7.2 Centrifuge
- 7.3 Disposable pipettes, 1 and 10 mL
- 7.4 Filter paper: Reeve Angel 5202-185 or equivalent
- 7.5 Glass funnels
- 7.6 Graduated cylinders, glass, 100 and 1000 mL
- 7.7 pH paper
- 7.8 Potassium Iodide Starch Test Paper: Fisher #14-860
- 7.9 Teflon separatory funnels, 2 L
- 7.10 Test tubes with caps
- 7.11 TurboVap II concentrator and tubes
- 7.12 VOA vials
- 7.13 Volumetric flasks

8.0 **REAGENTS AND STANDARDS**

- 8.1 All reagents used must be analytical reagent (AR) grade or higher. All standards must be traceable to NIST, when available. Certificates of traceability must be obtained from the manufacturer. All reagents and standards must be documented in the Laboratory-Standards tab in the LIMS. Refer to the requirements in the Labeling of Standards, Reagents, Digestates and Extracts SOP.

8.2 REAGENTS

All stock reagents are stored in the Organics Prep lab unless otherwise noted.

- 8.2.1 Lab pure water: Analyte free water is prepared as described in the Quality Assurance Plan. DI water may be obtained from any of the designated taps throughout the lab.
- 8.2.2 Acetone (C₃H₆O): EMD Omnisolv AX0116-1 or equivalent
- 8.2.3 Acetonitrile (C₂H₃N): Fisher #A9984 or equivalent
- 8.2.4 n-Hexane (C₆H₁₄), 95%: J.T Baker #9262 or equivalent

- 8.2.5 Methanol (CH_4O , also noted as MeOH): EMD Omnisolv MX0488-1 or equivalent
- 8.2.6 Methylene chloride (CH_2Cl_2): Honeywell Burdick & Jackson CS299-200 or equivalent
- 8.2.7 Sodium hydroxide (NaOH): EMD SX0590-3 or equivalent
- 8.2.8 Sodium hydroxide, 10 N NaOH : In a 1 L volumetric flask, dissolve and dilute 400 g NaOH to the mark with DI water.
- 8.2.9 Sodium sulfate, anhydrous (Na_2SO_4): EMD SX0760-20 or equivalent
- 8.2.10 Sodium thiosulfate pentahydrate: Acros #42446-0010, or equivalent (Stored in Wet Chem lab)
- 8.2.11 Sulfuric acid, concentrated (H_2SO_4): EMD SX1244-75 or equivalent

8.3 STANDARDS

All stock standards are stored in the SVOA/GC lab unless otherwise noted. Stock standards are transferred to 2 mL amber screw-top vials with a label after opening. Unless otherwise noted, prepared standards are stored in SVOA/GC lab under appropriate conditions as specified by the manufacturer, and prepared on an as needed basis. Prepared standards are transferred to labeled screw-top jars of various sizes depending on the final volume. The analyst may prepare a larger or smaller volume of spike standard and surrogate standard than those listed below if the volume of the stock standard used is adjusted accordingly.

- 8.3.1 Base-Neutral Stock Spike Standard, 5,000 $\mu\text{g/mL}$ each: Restek #31074. See table in section 20.0 for compound list.
- 8.3.2 Acid Stock Spike Standard, 10,000 $\mu\text{g/mL}$ each: Restek #31061. See table in section 20.0 for compound list.
- 8.3.3 SVOA Working Spike Standard, 100 $\mu\text{g/mL}$ each: In a 50 mL volumetric flask, dilute 1.0 mL of the Base-Neutral Stock Spike Standard and 500 μL of the Acid Stock Spike Standard to the mark with methanol. This prepares a standard containing the base-neutral compounds and the acid compounds at 100 $\mu\text{g/mL}$. **Add 1 mL of this solution to the LCS, MS, and MSD samples.**
- 8.3.4 Method 625 BNA Stock Spike Standard, 400 $\mu\text{g/mL}$ each: For additional analytes for Method 625 the following custom standards are used: (1) Ultra Scientific #CUS-4924; (2) Ultra Scientific # CUS-5946; (3) Ultra Scientific #CUS-3883; (4) Ultra Scientific #CUS-4407; (5) Ultra Scientific #CUS-13706; (6) Ultra Scientific quote # 092911-438 are obtained and used on a rotating basis. See table in section 20.0 for compound list.
- 8.3.5 Method 625 Working Spike Standard, 100 $\mu\text{g/mL}$ each: Using a 100 mL volumetric flask, dilute 25 mL of the Method 625 BNA Stock Spike Standard with an additional 75 mL of methanol bringing the total volume of prepared standard to 100 mL. This prepares a standard containing the Method 625 analytes at 100 $\mu\text{g/mL}$. **Add 1 mL of this solution to the LCS, MS, and MSD samples.**
- 8.3.6 Base-Neutral Stock Surrogate Standard, 5,000 $\mu\text{g/mL}$ each: Supelco #4-7263. Contains Nitrobenzene-d5; p-Terphenyl-d14; 2-Fluorobiphenyl

- 8.3.7 Acid Stock Surrogate Standard, 10,000 µg/mL each: Supelco #4-7260-U. Contains Phenol-d6; 2,4,6-Tribromophenol; 2-Fluorophenol
- 8.3.8 SVOA BNA Stock Surrogate Standard: Ultra ISM-333XC-500 Premade Surrogate with Indicator, or equivalent **Add 1 mL of this solution to all samples.**
- 8.3.8.1 If the premade surrogate standard is not available, the SVOA BNA Stock Surrogate Standard can be prepared by diluting 2.0 mL of the Base-Neutral Stock Surrogate Standard and 1.5 mL of the Acid Stock Surrogate Standard to 100 mL with MeOH. This prepares a standard containing the base-neutral compounds at 100 µg/mL and the acid compounds at 150 µg/mL.
- 8.3.9 TCLP BNA Stock Spike Standard, 2000 µg/mL: Accustandard #TCLP-BNA-PAK. See table in section 20.0 for compound list.
- 8.3.10 TCLP BNA Working Spike Standard, 100 µg/mL: In a 50 mL volumetric flask, dilute 2.5 mL Stock TCLP BNA Spike Standard to the mark with Methanol. **Add 1 mL of this solution to the LCS, MS, and MSD samples.**
- 8.3.11 PNA-IL (GC/MS) Stock Spike Standard, 2000 µg/mL each: Accustandard #Z-014G-R-PAK. See table in section 20.0 for compound list.
- 8.3.12 PNA-IL (GC/MS) Working Spike Standard, 10 µg/mL each: In a 100 mL volumetric flask, dilute 500 µL of the stock PNA-IL spike standard to the mark with Methanol. **Add 1 mL of this solution to the LCS, MS, and MSD samples.**
- 8.3.13 PNA-IL (GC/MS) Working Surrogate Standard, 10 µg/mL each: In a 100 mL volumetric flask, dilute 200 µL of the Stock Base-Neutral Surrogate Standard to the mark with Methanol. **Add 1 mL of this solution to all samples.**
- 8.3.14 Phenol Stock Surrogate Standard, 2000 µg/mL each: Accustandard #M-8040-SS-PAK contains 2-Fluorophenol and 2,4,6-Tribromophenol.
- 8.3.15 Phenol Working Surrogate Standard, 100 µg/mL: In a 20 mL volumetric flask, dilute 1 mL of the stock phenol surrogate standard to the mark with Methanol. **Add 1 mL of this solution to all samples.**
- 8.3.16 Phenol 604 Spike Standard, 2000 µg/mL: Restek #31029
- 8.3.17 Phenol Working Spike Standard, 100 µg/mL: In a 25 mL volumetric flask dilute 1.25 mL of the Phenol 604 Spike Standard to the mark with Methanol. **Add 1 mL of this solution to the LCS, MS and MSD samples.**
- 8.3.18 PCB Stock Spike Standard, 1000 µg/mL each: Restek #32039 contains Aroclor 1016 and Aroclor 1260.
- 8.3.19 PCB Working Spike Standard, 5 µg/mL: In a 50 mL volumetric flask, dilute 250 µL of PCB Stock Spike Standard to the mark with Acetone. **Add 1 mL of this solution to the LCS, MS, and MSD.**
- 8.3.20 Pesticide Stock Spike Standard, 200 µg/mL each: Restek #32291. See the table in section 20.0 for the compound list.

- 8.3.21 Pesticide Working Spike Standard, 0.5 µg/mL each: In a 100 mL volumetric flask, dilute 250 µL of the stock pesticide spike standard to the mark with Acetone. **Add 1 mL of this solution to the LCS, MS, and MSD.**
- 8.3.22 Pest/PCB Stock Surrogate Standard, 200 µg/mL each: Accustandard #CLP-032-R contains DCB and TCMX.
- 8.3.23 Pest/PCB Working Surrogate Standard, 0.2 µg/mL each: In a 200 mL volumetric flask, dilute 200 µL of the stock pest/PCB surrogate standard to the mark with Acetone. **Add 1 mL of this solution to all samples.**
- 8.3.24 ERO/DRO Stock Surrogate Standard, 2000 µg/mL: Accustandard #M-625-04-10X contains Decafluorobiphenyl (DFB).
- 8.3.25 ERO/DRO Working Surrogate Standard, 100 µg/mL: In a 100 mL volumetric flask, dilute 5.0 mL of the stock ERO surrogate standard to the mark with Methylene chloride. Add 1 mL of this solution to all samples.
- 8.3.26 ERO/DRO Stock Spike Standard, 50,000 µg/mL: Ultra Scientific #RGO-616 contains diesel fuel #2.
- 8.3.27 ERO/DRO Working Spike Standard, 1000 µg/mL: In a 100 mL volumetric flask, dilute 2 mL of the stock ERO spike standard to the mark with Methylene Chloride. **Add 1 mL of this solution to LCS, MS & MSD.**

9.0 **SAMPLE COLLECTION, PRESERVATION, AND HOLDING TIMES**

- 9.1 The client or other trained personnel collect samples. Samples received at the laboratory are considered representative unless otherwise noted.
- 9.2 Samples should be collected in an amber glass container. Preservation consists of storage in the range of 0.1 – 6°C. Samples are stored in the coolers located in the sample receipt area. Samples that fail to meet the preservation criteria are noted as such on the Cooler Inspection Report in the Login process.
- 9.3 Samples must be free of chlorine prior to extraction. If chlorine is present, dechlorinate with Na₂S₂O₃ prior to extraction and record actions in the comments column of the Organics Extractions logbook. Samples for analysis of Pesticides/PCBs by EPA Method 608 as well as SW-846 8081/8082 must have a pH in the range of 5 – 9. Use H₂SO₄ or NaOH to adjust as needed and record actions in the comments column of the Organics Extractions logbook. Adjustments are not necessary on 608 samples that are extracted within 3 days of collection. Regardless of chlorine or pH, all samples must be extracted within the maximum allowable hold time of 7 days from collection.

10.0 **QUALITY CONTROL**

- 10.1 An *Initial Demonstration of Capability* study must be performed prior to the initial analysis for each analyst and whenever substantial change has occurred in the procedure or instrument. Refer to the Capability and Detection Limit Studies SOP for details.

- 10.2 A *Method Detection Limit* study must be performed for each new procedure, annually thereafter, and whenever a change in instrument occurs. Refer to the Capability and Detection Limit Studies SOP for details.
- 10.3 A *Method Blank* must be prepared with each batch of samples (maximum 10 samples for method 604 and 608; maximum 20 samples for 8000 methods and method 625).
- 10.3.1 The acceptance criteria are listed in the appropriate test codes in LIMS and are evaluated at analysis.
- 10.4 A *Laboratory Control Sample* must be prepared with each batch of samples (maximum 10 samples for methods 604 and 608; maximum 20 samples for 8000 methods and method 625).
- 10.4.1 The acceptance criteria are listed in the appropriate test codes in LIMS and are evaluated at analysis.
- 10.5 A *Matrix Spike and Matrix Spike Duplicate* sample must be prepared with each batch of samples (maximum 10 samples for method 604 and 608; maximum 20 samples for 8000 methods and method 625). If insufficient sample exists for the preparation of a MS/MSD, a duplicate LCS (i.e. LCSD) should be extracted.
- 10.5.1 The acceptance criteria are listed in the appropriate test codes in LIMS and are evaluated at analysis.

11.0 **CALIBRATION AND STANDARDIZATION**

- 11.1 Perform the required preventative maintenance as necessary. Documentation is retained in the Maintenance Log for the particular piece of equipment.
- 11.2 On a daily basis prior to use, wipe the inside with a clean dry paper towel and check the water level of the Turbo Vap concentrators. Add DI water as needed.
- 11.3 On a daily basis prior to use, verify the settings of the concentrators; Temperature = 29°, Pressure = 18 psi, Time = approximately 30 minutes (nominal) initially.
- 11.4 The thermometers used must be verified in accordance with the Thermometer Calibration SOP.

12.0 **PROCEDURE**

Analytical data is documented and retained using the Organics Extractions Log (copy attached). Analytical data must be maintained in accordance with the document control requirements in the Quality Assurance Plan as well as the Document Control SOP.

- 12.1 Rinse all glassware with acetone.
- 12.2 Triple rinse all glassware with methylene chloride.
- 12.3 Check to ensure sufficient reagent and spike volumes are available for the completion of the entire preparation procedure.
- 12.4 Check for chlorination on Potassium Iodide Starch test paper. If chlorine is present add a stir bar to the jar, and stir the sample while adding small amounts of sodium thiosulfate until chlorine is not present.
- 12.5 Transfer sample into separatory funnel. The MBLK and LCS are prepared with 1000 mL DI water adjusted to the proper pH.

- 12.6 Add surrogate and spike solutions to appropriate samples and record type, lot number, and amount added.
- 12.7 Check the pH with pH paper. If necessary, adjust pH using sulfuric acid or sodium hydroxide. See table in section 20.0 for the required sample pH.
 - 12.7.1 For BNA samples, the pH is first adjusted with sulfuric acid. After completing step 12.14, the pH of the sample will be adjusted with sodium hydroxide and steps 12.7 through 12.14 are repeated.
- 12.8 Using a graduated cylinder, add 60 mL Methylene chloride to separatory funnel. Use approximately 20 mL of the Methylene chloride to rinse the sample jar and pour the rinse into the separatory funnel.
- 12.9 Shake separatory funnel for 2 minutes, venting frequently.
- 12.10 Prepare a funnel with filter paper and a large scoop of sodium sulfate (fills filter approximately half-way).
- 12.11 Rinse filter with approximately 20 mL of Methylene chloride and discard the Methylene chloride. Allow sufficient time for adequate phase separation because the samples may need to be centrifuged into VOA vials to complete the separation. Centrifuge the sample in VOA vials if the emulsion is greater than 1/3 of the solvent layer.
- 12.12 Drain extract from separatory funnel through filter into a concentrator tube. (Occasionally samples will need to be centrifuged in VOA vials to complete the separation).
- 12.13 Repeat the shake/separate steps two more times with fresh volumes of Methylene chloride each time.
- 12.14 Allow the filtration to continue to completion. Rinse filter with methylene chloride adding this to the concentrator tube. The final volume should be less than 200 mL.
 - 12.14.1 For BNA samples, combine the acid and base extracts into one concentrator tube.
- 12.15 Empty contents of separatory funnel into a Class A 1 L graduated cylinder. Record the volume in Extraction Logbook.
- 12.16 Place the concentrator tube into Turbo Vap unit and begin the concentration. The unit will beep when the preset time has expired.
- 12.17 When the unit beeps, check the volume of the sample. Continue evaporation until the extract volume is slightly less than 1 mL (for extractions where the final volume is greater than 1 mL, evaporating down to slightly more than 1 mL, e.g. 1.5 mL, is acceptable). **CAUTION: DO NOT LET EXTRACT GO DRY!**
- 12.18 Perform any "solvent exchange" as needed. See the Extract Conditions table in section 20.0 for the required final solvent.
 - 12.18.1 To "exchange" the solvent, dilute to approximately 10 mL with the new (exchange) solvent then place the sample back into the concentrator and evaporate the extract back down to slightly less than 1 mL (or slightly more if applicable).
- 12.19 Using a 1 mL or 10 mL disposal glass pipette, remove the extract from the concentrator tube and retain the extract in the pipette. Rinse the concentrator tube with a small volume of the appropriate solvent and add this to the volume

already in the pipette. Repeat as needed to obtain the targeted final volume and record the final volume.

12.20 Transfer the extract into an appropriate container (e.g. vials, test tubes, etc.), cap the container and label with the sample I.D., parameter/test, and extraction date.

12.21 Extracts are retained in cold storage (4°C) using the cooler in the GC/SVOA lab. If the instrument analysts do not pick up the extracts on the day they were extracted, place the extracts in the appropriate cooler in a labeled Solo cup for 1 mL final volume extracts.

13.0 CALCULATIONS AND DATA HANDLING

Enter the data into LIMS. Details on the procedure for entering preparation data are in the Preparation Batch Data Entry SOP. Samples prepared beyond the extent of the allowable hold time will be shaded red when selected for a batch in the LIMS. Data associated with a failed hold time must be documented with a CAR form.

14.0 METHOD PERFORMANCE

14.1 Initial Demonstration of Capability study data, Method Detection Limit study data and Performance Testing study data are maintained and available from the QA office.

15.0 INSTRUMENT MAINTENANCE

15.1 Consult the Turbovap Concentrator Operator's Manual for maintenance information and Section 11.0 of this SOP for the normal daily settings. Record all maintenance in the Turbovap maintenance logbook.

16.0 TROUBLESHOOTING

16.1 Consult the Turbovap Concentrator Operator's Manual for troubleshooting information.

17.0 POLLUTION PREVENTION

17.1 The quantity of chemicals purchased should be based on expected usage during their shelf life and the disposal cost of unused material.

17.2 Prepare the minimum amount of reagent and standard necessary.

18.0 WASTE MANAGEMENT

18.1 Refer to the Sample Disposal SOP for guidance on the disposal of any resulting residue, digestate, distillate, extract or standard.

19.0 REFERENCES

19.1 SW-846 Method 3510C

19.2 EPA Methods 604, 608, 625

19.3 Microbac Laboratories Quality Assurance Plan, current revision, all sections

20.0 TABLES, FORMS, CHECKLISTS, AND OTHER ATTACHMENTS

- 20.1 Spike standard compound tables
- 20.2 Extraction Conditions table
- 20.3 Copy of the Organics Prep log
- 20.4 Copy of the SOP Revision Notification form with changes in this revision.

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SECTION 20.1: SPIKE STANDARD COMPOUNDS

| Standard Name | Source | Compounds |
|----------------------------|------------------------------------|--|
| Base-Neutral Stock Spike | Restek #31074 | Acenaphthene • N-Nitrosodi-n-propylamine • Pyrene • 1,2,4-Trichlorobenzene • 1,4-Dichlorobenzene • 2,4-Dinitrotoluene |
| Acid Stock Spike | Restek #31061 | Pentachlorophenol • Phenol • 2-Chlorophenol • 4-Chloro-3-methylphenol • 4-Nitrophenol |
| Method 625 BNA Stock Spike | Ultra Scientific #CUS-4924 | 1,3-dichlorobenzene • 2,4-dinitrophenol • 2,6-dinitrotoluene • 2-methyl-4,6-dinitrophenol • Benz [a] anthracene • Bis (2-chloroethyl) ether • Di-n-octyl phthalate • Fluorene • Benzo [b] fluoranthene • Hexachlorobutadiene |
| Method 625 BNA Stock Spike | Ultra Scientific #CUS-5946 | 2-chloronaphthalene • 3,3'-dichlorobenzidine • 4-chlorophenyl phenyl ether • Benzo[g,h,i]perylene • Benzo[k]fluoranthene • Bis(2-chloroisopropyl) ether • Bis(2-ethylhexyl) phthalate • Dibenz[ah]anthracene • Diethyl phthalate • Dimethyl phthalate • Indeno[1,2,3-cd]pyrene |
| Method 625 BNA Stock Spike | Ultra Scientific #CUS-3883 | Acenaphthylene • Anthracene • Bis(2-chloroethoxy)methane • Chrysene • Di-n-butyl phthalate • Hexachlorobenzene • Naphthalene • 2,4-dichlorophenol • 2,4,6-trichlorophenol • 2-nitrophenol |
| Method 625 BNA Stock Spike | Ultra Scientific #CUS-4407 | 1,2-dichlorobenzene • 2,4-dimethylphenol • 4-bromophenol phenyl ether • Benzo[a]pyrene • Butyl benzyl phthalate • Fluoranthene • Hexachloroethane • Isophorone • Nitrobenzene • Phenanthrene |
| Method 625 BNA Stock Spike | Ultra Scientific #CUS-13706 | N-Nitrosodimethylamine • 3-Methylphenol • 4-Methylphenol • Benzoic acid • 4-Chloroaniline • 2,4,5-Trichlorophenol • 2-Nitroaniline • Dibenzofuran • 4-Nitroaniline • 1,2-Diphenylhydrazine • alpha-Terpineol • Benzyl alcohol |
| Method 625 BNA Stock Spike | Ultra Scientific Quote# 092911-438 | Pyridine • Aniline • 2-Methylphenol • 2,6-Dichlorophenol • 2-Methylnaphthalene • Hexachlorocyclopentadiene • 3-Nitroaniline • N-Nitrosodiphenylamine • Carbazole • Biphenyl |
| SVOA BNA Stock Surrogate | Ultra #ISM-333XC-500 | Terphenyl-d14 • Phenol-d5 • Nitrobenzene-d5 • 2-Fluorophenol • 2-Fluorobiphenyl • 2-Chlorophenol-d4 • 1,4,6-Tribromophenol • 1,2-Dichlorobenzene-d4 |
| TCLP BNA Stock Spike | Accustandard #TCLP-BNA-PAK | o-Cresol • m-Cresol • p-Cresol • 1,4-Dichlorobenzene • 2,4-Dinitrotoluene • Hexachlorobenzene • Hexachlorobutadiene • Hexachloroethane • Nitrobenzene • Pentachlorophenol • Pyridine • 2,4,5-Trichlorophenol • 2,4,6-Trichlorophenol |
| PNA-IL Stock Spike | Accustandard #Z-014G-R-PAK | Naphthalene • Acenaphthene • Acenaphthylene • Fluorene • Pyrene • Benzo(a)anthracene • Chrysene • Carbazole • Phenanthrene • Benzo(b)fluoranthene • Benzo(k)fluoranthene • Benzo(a)pyrene • Dibenz (a,h)anthracene • Benzo(g,h,i)perylene • Indeno(1,2,3-cd)pyrene • Fluoranthene • Anthracene |

| Standard Name | Source | Compounds |
|------------------------|---------------|--|
| Pesticide Stock Spike | Restek #32039 | Aldrin • Alpha-BHC • Alpha-Chlordane • Beta-BHC • Delta-BHC • Dieldrin • Endosulfan I • Endosulfan II • Endosulfan sulfate • Endrin • Endrin aldehyde • Endrin ketone • Gamma-BHC • Gamma- Chlordane • Heptachlor • Heptachlor epoxide • Methoxychlor • 4,4'-DDD • 4,4'-DDE • 4,4'-DDT |
| Phenol 604 Stock Spike | Restek #31029 | Phenol • Pentachlorophenol • 4-Nitrophenol • 4-Chloro-3-methylphenol • 4,6- Dinitro-2-methylphenol • 2-Nitrophenol • 2-Chlorophenol • 2,4- Dinitrophenol • 2,4-Dimethylphenol • 2,4-Dichlorophenol • 2,4,6-Trichlorophenol |

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SECTION 20.2: EXTRACTIONS CONDITIONS TABLE

| Determinative Method and Prep Code | Analyte Group | Initial Extraction pH | Secondary Extraction pH | Final Solvent for Analysis | Final Solvent for Cleanup | Final Vol., mL |
|---|-------------------------|-----------------------|-------------------------|----------------------------|---------------------------|--|
| 608 608PW (<3 days from collection) | PCBs/ Pest | <u>As received</u> | None | Hexane | Hexane | 10 |
| 608 608PW (>3 days from collection) | PCBs/ Pest | <u>5-9</u> | None | Hexane | Hexane | 10 |
| 8015B 3510_D | Diesel Range Organics | As received | None | Methylene Chloride | --- | 1/100 of the initial volume rounded down to the whole mL |
| 8015B 3510_ERO | Extended Range Organics | As received | None | Methylene Chloride | --- | 10 |
| 604 604PW | Phenols | ≤ 2 | None | Methylene Chloride | --- | 1 |
| 8081A 3510_P | Pest | 5 – 9 | None | Hexane | Hexane | 10 |
| 8082 3510_PCB | PCBs | 5 – 9 | None | Hexane | Hexane | 10 |
| 8270C¹/625 3510_B/625PW | SVOA (BNA) | <2 | >11 | Methylene Chloride | --- | 1 |
| PAH-MS-SIM-W 3510_B-SIM | PNA-IL (GC/MS) | >11 | None | Methylene Chloride | --- | 1 |

1 = Extraction pH sequence may be reversed to better separate the acid and neutral components.
Excessive pH adjustments may result in the loss of some analytes.

Microbac Laboratories - Chicagoland Division
ORGANICS EXTRACTIONS LOG

Pipette Lot Number _____

revision: _f_9/2010

SECTION 20.4: SOP REVISION FORM

SOP REVISION NOTIFICATION FORM FOR:
**PREPARATION OF AQUEOUS SAMPLES USING
 MANUAL LIQUID-LIQUID EXTRACTION**
 OLD REVISION: 7
 NEW REVISION: 8

SUMMARY OF CHANGES:

1. Added effective date to the header.
2. Changed "Revision author" to Christine Robinson.
3. Changed "Lab Director" to "Division Manager".
4. Edited section 2.1: "This is an extraction procedure for the preparation of samples for Pesticide, Polychlorinated Biphenyl, Total Petroleum Hydrocarbons, Polyaromatic Hydrocarbon, and Semi-Volatile Organic Analytes. This procedure is applicable to the preparation of aqueous and non-aqueous liquid matrix samples."
5. Added section 3.3.2: "The reference method uses 100-ml of Methylene Chloride to extract the sample. This procedure used 60-ml of Methylene Chloride due to the constraints of the 200-ml concentrator tube and to limit out environmental impact by reducing the amount of solvent used in the extraction."
6. Added section 3.3.3: "EPA Method 604 does a solvent exchange to 2-Propanol prior to analysis. This procedure does not do a solvent exchange for Method 604. The final extracts are in Methylene Chloride. A validation study (found on the S Drive) was performed which showed higher recovery of target analytes on Laboratory Control samples that remained in Methylene Chloride over those that were exchanged to 2-Propanol."
7. Section 7.0: Updated the sources for supplies as necessary.
8. Added the following equipment to the list in section 7: [7.13] Volumetric flask
9. Section 8.0: Updated the sources for reagents as necessary.
10. Edited Section 8.1: "All reagents used must be analytical reagent (AR) grade or higher. All standards must be traceable to NIST, when available. Certificates of traceability must be obtained from the manufacturer. All reagents and standards must be documented in the appropriate preparation logbook Laboratory-Standards tab in the LIMS. Refer to the requirements in the Labeling of Standards, Reagents, Digestates and Extracts SOP."
11. Added the following to the list of reagents: [8.2.7] Sodium hydroxide (NaOH): EMD SX0590-3 or equivalent; [8.2.8] Sodium hydroxide, 10 N NaOH: In a 1 L volumetric flask, dissolve and dilute 400 g NaOH to the mark with DI water. [8.2.10] Sodium thiosulfate pentahydrate: Acros #42446-0010, or equivalent (Stored in Wet Chem lab) [8.2.11] Sulfuric acid, concentrated (H₂SO₄): EMD SX1244-75 or equivalent
12. Edited Section 8.3: "All stock standards are stored in the SVOA/GC lab unless otherwise noted. Stock standards are transferred to 2 mL amber screw-top vials with a label after opening. Unless otherwise noted, prepared standards are stored in SVOA/GC lab under appropriate conditions as specified by the manufacturer, and prepared on an as needed basis. Prepared standards are transferred to labeled screw-top jars of various sizes depending on the final volume. The analyst may prepare a ~~higher~~ larger or smaller volume of spike standard and surrogate standard than those listed below if the volume of the stock standard used is adjusted accordingly."



SOP REVISION NOTIFICATION FORM FOR:

**PREPARATION OF AQUEOUS SAMPLES USING
MANUAL LIQUID-LIQUID EXTRACTION**

OLD REVISION: 7

NEW REVISION: 8

13. Section 8.3: Renamed standards such that the name states the type of analyte and then the use of the analyte. For instance, "Stock Base-Neutral Surrogate Standard" is now called "Base-Neutral Stock Surrogate Standard"
14. Edited section 8.3.8 by moving the prep to section 8.3.8.1 and adding the stock source to 8.3.8: "[8.3.8] SVOA Surrogate **BNA Stock Standard: Ultra ISM-333XC-500 Premade Surrogate with Indicator, or equivalent** Add 1 mL of this solution to all samples. [8.3.8.1] If the **premade stock surrogate** standard is not available, the SVOA Surrogate Standard can be prepared by diluting 2.0 mL of the stock base-neutral surrogate standard and 1.5 mL of the stock acid surrogate standard to 100 mL with MeOH. This prepares a standard containing the base-neutral compounds at 100 µg/mL and the acid compounds at 150 µg/mL.
15. Deleted what had been sections 8.3.14 through 8.3.17. These sections described the HPLC PNA standards which are no longer used.
16. Edited section 8.3.15: "Phenol Working Surrogate Standard, 100 µg/mL: In a ~~25~~ **20** mL volumetric flask, dilute ~~4.25~~ **1** mL of the stock phenol surrogate standard to the mark with Methanol. Add 1 mL of this solution to all samples.
17. Added section 8.3.16: "Phenol **604 Spike Standard, 2000 ug/mL: Restek #31029**"
18. Added section 8.3.17: "Phenol Working Spike Standard, 100 µg/mL: In a **25** mL volumetric flask dilute **1.25** mL of the Phenol **604 Spike Standard** to the mark with Methanol. Add 1 mL of this solution to the LCS, MS and MSD samples."
19. Edited section 8.3.18: "PCB Stock Spike Standard, 1000 µg/mL each: ~~Supelco #4-8097 Restek #32039~~ contains Aroclor 1016 and ~~Supelco #4-4809~~ contains Aroclor 1260."
20. Edited section 8.3.19: "PCB Working Spike Standard, 5 µg/mL: In a ~~400~~ **50** mL volumetric flask, dilute ~~500~~ **250** µL of each ~~Aroclor 1016 and Aroclor 1260~~ PCB Stock Spike Standard to the mark with Acetone. Add 1 mL of this solution to the LCS, MS, and MSD."
21. Edited section 8.3.20: "Pesticide Stock Spike Standard, 2000 µg/mL each: ~~Supelco #4-8943 Restek #32291~~. See the table in section ~~2048.0~~ for the compound list."
22. Edited section 8.3.21: "Pesticide Working Spike Standard, 0.5 µg/mL each: In a 100 mL volumetric flask, dilute 250 µL of the stock pesticide spike standard to the mark with Acetone. Add 1 mL of this solution to the LCS, MS, and MSD."
23. Edited section 8.3.24: "ERO/DRO Stock Surrogate Standard, 2000 µg/mL: Accustandard ~~#M-625-04-10X~~ contains Decafluorobiphenyl (DFB).
24. Edited section 8.3.25: "ERO/DRO Working Surrogate Standard, 100 µg/mL: In a ~~50~~ **100** mL volumetric flask, dilute ~~2.5~~ **5.0** mL of the stock ERO surrogate standard to the mark with ~~Methylene chloride~~ **Methanol**. Add 1 mL of this solution to all samples."
25. Edited section 8.3.26: "ERO/DRO Stock Spike Standard, 50,000 µg/mL: Ultra Scientific ~~#RGO-616~~ contains diesel fuel #2."



SOP REVISION NOTIFICATION FORM FOR:

**PREPARATION OF AQUEOUS SAMPLES USING
MANUAL LIQUID-LIQUID EXTRACTION**

OLD REVISION: 7

NEW REVISION: 8

26. Edited section 8.3.27: "ERO/DRO Working Spike Standard, 1000 µg/mL: In a ~~50~~ 100 mL volumetric flask, dilute 4 2 mL of the stock ERO spike standard to the mark with Methylene Chloride-Methanol. Add 1 mL of this solution to LCS, MS & MSD."
27. Deleted what had been sections 8.3.32 through 8.3.34. These sections described the Full List BNA standards which are no longer used.
28. Edited section 10.3: "A *Method Blank* must be prepared with each batch of samples (maximum 10 samples for method 604; and 608 and 640; maximum 20 samples for 8000 methods and method 625)."
29. Edited section 10.4: "A *Laboratory Control Sample* must be prepared with each batch of samples (maximum 10 samples for methods 604 and 608 and 640; maximum 20 samples for 8000 methods and method 625)."
30. Edited section 10.5: "A *Matrix Spike and Matrix Spike Duplicate* sample must be prepared with each batch group of samples (maximum 10 samples for method 604 and 608; maximum 20 samples for 8000 methods and method 625). ~~of maximum 10 samples at a minimum of one per day extracted for the 600-series methods with the exception of method 625, and with each group of maximum 20 samples at a minimum of one per day extracted for the 8000-series methods and method 625.~~ If insufficient sample exists for the preparation of a MS/MSD, a duplicate LCS (i.e. LCSd) should be extracted. "
31. Edited section 12.7.1: "For BNA samples, the pH is first adjusted with sulfuric acid. After completing step 12.14, the pH of the sample will be adjusted with sodium hydroxide and steps ~~12.6-12.7~~ 12.7 through 12.14 are repeated."
32. Edited section 12.8: "Using a graduated cylinder, add 60 mL Methylene chloride to separatory funnel. Use approximately 20 mL of the Methylene chloride to rinse the sample jar and pour the rinse into the separatory funnel."
33. Edited section 12.10: "Prepare a funnel with filter paper and a ~~small~~ large scoop of sodium sulfate (fills filter approximately half-way) then rinse filter with Methylene chloride and discard the rinsing."
34. Edited section 12.11: "Rinse filter with approximately 20 mL of Methylene chloride and discard the Methylene chloride. Allow sufficient time for adequate phase separation because the samples may need to be centrifuged into VOA vials to complete the separation. Centrifuge the sample in VOA vials if the emulsion is greater than 1/3 of the solvent layer."
35. Edited section 13.1: "Enter the data into LIMS. Details on the procedure for entering preparation data are in the Preparation Batch Data Entry SOP. Samples prepared beyond the extent of the allowable hold time will be shaded red when selected for a batch in the LIMS. ~~automatically show a message similar to "The prep Hold Time was exceeded by 4 days" in the comments field of the Prep Batch screen and report.~~ Data associated with a failed hold time must be documented with a CAR form."
36. Added Section 15.0: "INSTRUMENT MAINTENANCE"



SOP REVISION NOTIFICATION FORM FOR:

**PREPARATION OF AQUEOUS SAMPLES USING
MANUAL LIQUID-LIQUID EXTRACTION**

OLD REVISION: 7

NEW REVISION: 8

37. Added Section 15.1: "Consult the Turbovap Concentrator Operator's Manual for maintenance information and Section 11.0 of this SOP for the normal daily settings. Record all maintenance in the Turbovap maintenance logbook."
38. Added Section 16.0: "TROUBLESHOOTING"
39. Added Section 16.1: Consult the Turbovap Concentrator Operator's Manual for troubleshooting information."
40. Edited section 19.2: "EPA Methods 604, 608, 640, 625"
41. Edited section 20.1: Added Phenol Spike compound list as well as two new custom standards for 625 spike.
42. Edited section 20.2: Added DRO directions to the table. Deleted the HPLC directions from the table.
43. Edited section 20.2 to update prep codes to Element codes.
44. Updated section 20.3: Added the newest version of the Organics Prep Log.

By initialing below, I certify that I have been *notified* about the approval of a *new revision* to the above mentioned SOP and that I have read, understand, and agree to follow the test procedure as set forth in this new revision.

Initials & Date

Initials & Date

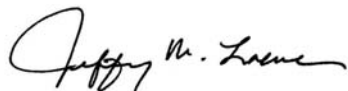
Initials & Date

Form revised 4/5/12

STANDARD OPERATING PROCEDURE FOR PREPARATION OF NON-AQUEOUS SAMPLES USING SONICATION

Revision Author: Donna Ruukonen

This SOP is effective upon signed approval by the following:



1/27/2012

Division Manager

Date



1/30/2012

QA Director

Date

DISCLAIMER: This SOP has been developed for use at the Microbac Laboratories, Merrillville, Indiana facility. It is intended for use by trained analysts. As written, this SOP may not be specifically applicable to the activities of other organizations.

1.0 TABLE OF CONTENTS

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2.0 SCOPE AND APPLICATION

- 2.1 This procedure is for the preparation of samples for Pesticide, Polychlorinated Biphenyl, Polyaromatic Hydrocarbon, and Semi-Volatile Organic Analytes using ultrasonic extraction. This procedure is applicable to the preparation of solid matrix samples.

3.0 SUMMARY

- 3.1 This process involves the isolation and concentration of organic compounds from non-aqueous samples for a variety of chromatographic techniques. The sonication process is used to ensure intimate contact of the sample matrix with the extraction solvent.
- 3.2 A measured amount of sample, usually 30 grams, is mixed with a drying agent to form a free-flowing mixture. This mixture is then serially extracted with an organic solvent using a sonicator. The extract is dried, concentrated, and, as necessary, exchanged into a solvent compatible with the cleanup or analytical method used.
- 3.3 Methylene chloride is used as the extraction solvent for this procedure. Method 3550B section 5.4 allows for the use of a variety of "solvent systems" provided it gives optimum, reproducible recovery of the analytes of interest in the sample matrix. Table 1 in Method 3550B provides data using Methylene chloride alone (solvent system A) indicating comparable results to other solvents or solvent mixtures.
- 3.4 This procedure is based on the reference methods listed in section 17 of this document. This procedure contains the following deviations from the reference methods. These deviations are not considered significant deviations from the method.
- 3.4.1 Section 7.3.1.2 of Method 3550B states to use 60 g of Na_2SO_4 to dry the samples. This procedure does not specify a minimum amount of Na_2SO_4 as the intention is to create a free flowing powder from the original sample. In an effort towards waste minimization, the minimum amount of reagent necessary is used.
- 3.4.2 Section 7.3.1.6 of Method 3550B states to use 100 mL of solvent for each serial extraction. This procedure uses 60 mL for each serial extraction. This volume is largely based on the volume of the concentrator tubes used in our evaporation equipment. Method 3550B assumes that a pre-concentration step is performed so to provide a solvent volume capable of fitting in the concentrator tubes of a nitrogen blowdown evaporation system.
- 3.4.3 Section 7.3.5 of Method 3550B states to apply a vacuum to the filtration of the extract. To avoid the potential of drying out the filter, this procedure omits the application of the vacuum filtration.

4.0 DEFINITIONS

- 4.1 A list of definitions is in the Quality Assurance Plan. In addition to the terms defined in the QAP, the terms below are specific and critical to this procedure.
- 4.2 Solvent exchange – Adding a different solvent other than the original extraction solvent after evaporating off the original solvent. This is performed so to provide a solvent matrix compatible with the analytes and analytical system employed.

5.0 INTERFERENCES

- 5.1 Interferences that co-elute vary considerably from sample to sample.
- 5.2 If the analysis of an extracted sample is prevented due to matrix interferences, further clean-up of the extract may be required.
- 5.3 Phthalate esters can contaminate many types of plasticware and glassware products used in the lab. Plastics, in particular, must be avoided because phthalates are commonly used in plasticizers and are easily extracted from plastic materials. Phthalate contamination may easily result any time that consistent adherence to the quality control requirements are not practiced.
- 5.4 Soap residue may cause the degradation of certain analytes especially Aldrin, heptachlor, and most organophosphorus pesticides. Strict adherence to the Glassware Washing and Preparation SOP is required.

6.0 SAFETY

- 6.1 Consult the current revision of the Chemical Hygiene Plan. Requirements for the use of personal protective equipment (e.g. safety glasses, lab coats, gloves) as well as other area-specific safety requirements (e.g. gas cylinders) and MSDS sheets are addressed in the CHP.

7.0 EQUIPMENT AND SUPPLIES

- 7.1 The following is a list of materials needed to perform the steps of this procedure as written. See the reference method(s) for equipment and supply specifications.
- 7.2 All volumetric glassware used shall be ASTM Class A. Class B glassware must be verified for accuracy on an annual basis and labeled with an appropriate correction.
- 7.3 Bottle-top dispenser (Fisher): 30 mL
- 7.4 Disposable pipettes: 1 and 10 mL
- 7.5 Filter paper (Whatman No. 41 or equivalent; Fisher P8 is equivalent)
- 7.6 Glass beakers: 250 mL

- 7.7 Glass funnels
- 7.8 Graduated cylinders, glass, 100 and 1000 mL
- 7.9 Screw-top jars: 60 mL, 120 mL, 250 mL
- 7.10 Screw-top vials with tops: 2mL
- 7.11 Sonicator (Fisher Scientific model 550/500 Sonic Dismembrator with a $\frac{3}{4}$ inch horn, or equivalent)
- 7.12 Test tubes with caps
- 7.13 Tongue depressors
- 7.14 Top loading balance (Denver XL-500, or equivalent)
- 7.15 Turbo Vap II concentrator and tubes

8.0 REAGENTS AND STANDARDS

- 8.1 All reagents used must be analytical reagent (AR) grade or higher. All standards must be traceable to NIST, when available. Certificates of traceability must be obtained from the manufacturer. All reagents and standards must be documented in the appropriate preparation logbook. Refer to the requirements in the Labeling of Standards, Reagents, Digestates and Extracts SOP.
- 8.2 Reagents
 - 8.2.1 All stock reagents are stored in the Organics Prep lab unless otherwise noted.
 - 8.2.2 Lab pure water (DI water): Analyte free water is prepared as described in the Quality Assurance Plan. DI water may be obtained from any of the designated taps throughout the lab.
 - 8.2.3 Acetone (C_3H_6O): OmniSolv #AX0116-1, or equivalent
 - 8.2.4 Acetonitrile (C_2H_3N): Fisher #A9984, or equivalent
 - 8.2.5 n-Hexane, 95% (C_6H_{14}): Honeywell Burdick & Jackson #213-4, or equivalent
 - 8.2.6 Methanol (CH_4O , also noted as MeOH): OmniSolv #MX0488-1, or equivalent
 - 8.2.7 Methylene chloride (CH_2Cl_2): Honeywell Burdick & Jackson #CS299-200, or equivalent
 - 8.2.8 Sodium sulfate (Na_2SO_4): EMD #SX0760-20 or equivalent

8.3 Standards

- 8.3.1 All stock standards are stored in the SVOA/GC lab unless otherwise noted. Stock standards are transferred to 2 mL amber screw-top vials with a label after opening. Unless otherwise noted, prepared standards are stored in SVOA/GC lab under appropriate conditions as specified by the manufacturer, and prepared on an as needed basis. Prepared standards are transferred to labeled screw-top jars of various sizes depending on the final volume. The analyst may prepare a higher volume of spike standard and surrogate standard than those listed below if the volume of the stock standard used is adjusted accordingly.
- 8.3.2 Stock Base-Neutral Spike Standard, 5,000 µg/mL each: Restek #31074. See table in section 18.1 for compound list.
- 8.3.3 Stock Acid Spike Standard, 10,000 µg/mL each: Restek #31061. See table in section 18.1 for compound list.
- 8.3.4 SVOA Spike, 100 µg/mL: In a 50 mL volumetric flask, dilute 1.0 mL of the Stock Base-Neutral Spike Standard and 500 µL of the Stock Acid Spike Standard to the mark with MeOH. This prepares a standard containing the base-neutral compounds and the acid compounds at 100 µg/mL. **Add 1 mL of this solution to the LCS, MS, and MSD samples.**
- 8.3.5 Stock Method 625 BNA Spike Standard, 400 µg/mL: For additional analytes for Method 625 the following custom standards are used on a rotating basis: (1) Ultra Scientific #CUS-4924; (2) Ultra Scientific # CUS-5946; (3) Ultra Scientific #CUS-3883; (4) Ultra Scientific #CUS-4407; (5) Ultra Scientific #CUS-13706; (6) Ultra Scientific Quote Number 092911-438. See table in section 18.1 for compound list.
- 8.3.6 Method 625 Spike, 100µg/mL: Using a 100-ml volumetric flask, dilute 25-ml of the Stock Method 625 BNA Spike Standard with an additional 75-ml of Methanol bringing the total volume of prepared standard to 100-ml. This prepares a standard containing the Method 625 analytes at 100 ug/ml. **Add 1-ml of this solution to the LCS, MS, and MSD samples.**
- 8.3.7 PNA-IL Stock Base-Neutral Surrogate Standard, 5,000 µg/mL each: Supelco #4-7263. See table in section 18.1 for compound list.
- 8.3.8 PNA-IL Surrogate Standard, 10 µg/mL: In a 100 mL volumetric flask, dilute 400 µL of the PNA-IL Stock Base-Neutral Surrogate Standard to the mark with MeOH. Transfer this solution into a 250 mL screw-top jar. Fill the same volumetric flask to the mark with MeOH and transfer it into the same 250 mL screw-top jar. **Add 1 mL of this solution to all samples.**
- 8.3.9 Semi-volatile Surrogate Standard Mixture with Indicator: BNA Custom Standard with Indicator: Ultra #ISM-333XC-500.

- 8.3.10 Stock PNA-IL Spike Standard, 2000 µg/mL each: Accustandard #Z-014G-R-PAK. See table in section 18.1 for compound list.
- 8.3.11 PNA-IL Spike Standard, 10 µg/mL each: In a 100 mL volumetric flask, dilute 500 µL of the Stock PNA-IL Spike Standard to the mark with MeOH. **Add 1 mL of this solution to the LCS, MS, and MSD samples.**
- 8.3.12 Stock PCB Spike Standard, 1000 µg/mL each of Aroclor 1016 and 1260: Restek #32039.
- 8.3.13 PCB Spike Standard, 5 µg/mL: In a 100 mL volumetric flask, dilute 500 µL of Aroclor 1016 and Aroclor 1260 Stock PCB Spike Standard to the mark with Acetone. **Add 1 mL of this solution to the LCS, MS, and MSD.**
- 8.3.14 Stock Pesticide Spike Standard, 200 µg/mL each: Restek #32291. See the table in section 18.1 for the compound list.
- 8.3.15 Pesticide Spike Standard, 0.5 µg/mL each: In a 50 mL volumetric flask, dilute 125 µL of the Stock Pesticide Spike Standard to the mark with Acetone. **Add 1 mL of this solution to the LCS, MS, and MSD.**
- 8.3.16 Stock Pest/PCB Surrogate Standard, 200 µg/mL each: Accustandard #CLP-032-R contains DCB and TCMX.
- 8.3.17 Pest/PCB Surrogate Standard, 0.2 µg/mL each: In a 100 mL volumetric flask, dilute 200 µL of the Stock Pest/PCB Surrogate Standard to the mark with Acetone. Transfer this solution into a 250 mL screw-top jar. Fill the same volumetric flask to the mark with Acetone and transfer it into the same 250 mL screw-top jar. **Add 1 mL of this solution to all samples.**

9.0 SAMPLE COLLECTION, PRESERVATION, AND HOLDING TIMES

- 9.1 The client or other trained personnel collect samples. Samples received at the laboratory are considered representative unless otherwise noted.
- 9.2 Samples should be collected in a clean glass container. Preservation consists of storage in the range of 0.1-6.0°C. Samples are stored in the coolers located in the sample receipt area. Samples that fail to meet the preservation criteria are noted as such on the Cooler Inspection Report in the Login process.
- 9.3 Analysis must be performed within the maximum allowable hold time of 14 days from collection.

10.0 QUALITY CONTROL

- 10.1 An *Initial Demonstration of Capability* study must be performed prior to the initial analysis for each analyst and whenever substantial change has occurred in the procedure or instrument. Refer to the Capability and Detection Limit Studies SOP for details.

- 10.2 A *Method Detection Limit* study must be performed for each new procedure, annually thereafter, and whenever a change in instrument occurs. Refer to the Capability and Detection Limit Studies SOP for details.
- 10.3 A *Method Blank* must be prepared and analyzed with each batch of maximum 20 samples and at a minimum of one per day.
- 10.3.1 Method blanks are prepared using 30 g Na₂SO₄.
- 10.4 A *Laboratory Control Sample* must be prepared and analyzed with each batch of maximum 20 samples and at a minimum of one per day.
- 10.4.1 Lab Control Samples are prepared using 30 g Na₂SO₄ and the applicable spike standards.
- 10.5 A *Matrix Spike and Matrix Spike Duplicate* sample must be prepared and analyzed with each batch of maximum 20 samples per matrix and at a minimum of one per day.
- 10.5.1 The acceptance criteria are listed in the applicable analytical procedure.

11.0 CALIBRATION AND STANDARDIZATION

- 11.1 Perform the required preventative maintenance as necessary. Documentation is retained in the Maintenance Log for the particular piece of equipment.
- 11.2 On a daily basis prior to use, verify (or calibrate if needed) the balance.
- 11.3 On a daily basis prior to use, wipe the inside with a clean dry paper towel and check the water level of the Turbo Vap concentrators. Add DI water as needed.
- 11.4 On a daily basis prior to use, verify the settings of the concentrators; Temperature = 29°C, Pressure = 18 psi, Time = approximately 30 minutes (nominal) initially.
- 11.5 On a weekly basis, tune the sonicator units on Sonicator #2. The procedure for this is found in the manufacturer's user's manual. Sonicator #1 has factory set tuning with no user-accessible adjustments.

12.0 PROCEDURE

- 12.1 Analytical data is documented and retained using the Organics Extractions Log (copy attached).
- 12.2 As noted in Method 3550B, ultrasonic extraction is not as rigorous a method as other extraction methods for soils/solids. Therefore, it is critical that the method be followed explicitly (including the manufacturer's instructions) to achieve the maximum extraction efficiency. At a minimum, successful use of this technique requires that:

- The extraction device must have a minimum of 300 watts of power and be equipped with appropriate size disrupter horns.
 - The horn must be properly maintained, including tuning according to the manufacturer's instructions prior to use, and inspection of the horn tip for excessive wear.
 - The samples must be properly prepared by thorough mixing with sodium sulfate so that they form a free-flowing mixture prior to the addition of the solvent.
 - The extraction horns used for "low concentration" and "high concentration" protocols are not interchangeable. Results indicate that the use of the 3/4" horn is inappropriate for the high concentration method, particularly for extraction of very nonpolar organic compounds such as PCBs, which are strongly adsorbed to the soil matrix.
 - Three extractions are performed with the appropriate solvent, the extraction is performed in the specified pulse mode, and the horn tip is positioned just below the surface of the solvent yet above the sample.
 - Very active mixing of the sample and the solvent must occur when the ultrasonic pulse is activated. The analyst must observe such mixing at some point during the extraction process.
- 12.3 Ensure that all glassware has been triple-rinsed with acetone and methylene chloride.
- 12.4 Check to ensure sufficient reagent and spike volumes are available for the completion of the entire preparation procedure.
- 12.5 Transfer 30.0 ± 0.1 g of a well-mixed sample into beaker and record the weight in the Organics Extraction Logbook.
- 12.6 Add enough sodium sulfate to the beaker to dry the sample, and mix using the disposable tongue depressor. Nonporous or wet samples (gummy or clay type) that do not have a free flowing sandy texture must be mixed with 60 g of anhydrous sodium sulfate, (or three large scoops). If required, more sodium sulfate may be added.
- 12.7 After addition of sodium sulfate, mix well using wooden, disposable depressor until the sample is free flowing.
- 12.8 Add surrogates and spikes to appropriate samples and record type, lot number, and amount added in the Organics Extraction Logbook.
- 12.9 Using a graduated cylinder or bottle-top dispenser, add 60 mL methylene chloride to the beaker.
- 12.10 Wipe the sonicator horn with methylene chloride.
- 12.11 Position the sonicator horn into the sample so the tip of the horn is approximately $\frac{1}{2}$ " below the surface of the solvent but above the sediment of the sample itself. For optimum performance, the Power output of the sonicator unit should be set at

10 and the Cycle set for 50% On / 50% Off (default at 1.0 sec for each). Sonicate the sample for 3 minutes.

- 12.12 Prepare a funnel with filter paper and a small scoop of sodium sulfate (fills filter approximately half-way) then rinse filter with methylene chloride and discard the rinsing.
- 12.13 Pour extract from the beaker through a filter into a concentrator tube.
- 12.14 Repeat the solvent addition, sonication, and filtration steps two more times (total of three extractions) adding the extract from each into the same concentrator tube. After the last extraction transfer the entire contents of the beaker (i.e. extract and sample) into the funnel. Rinse the beaker with methylene chloride until all of the sediment is transferred.
- 12.15 Allow the filtration to continue to completion. Rinse filter with methylene chloride adding this to the concentrator tube. The final volume should be less than 200 mL.
- 12.16 Place the concentrator tube into Turbo Vap unit and begin the concentration. The unit will beep when the preset time has expired.
- 12.17 When the unit beeps, check the volume of the sample. Continue evaporation until the extract volume is slightly less than 1 mL (for extractions where the final volume is greater than 1 mL, evaporating down to slightly more than 1 mL, e.g. 1.5 mL, is acceptable). **CAUTION: DO NOT LET EXTRACT GO DRY!**
- 12.18 Perform any "solvent exchange" as needed. See the Extract Conditions table in section 18.2 for the required final solvent.
 - 12.18.1 To "exchange" the solvent, dilute to approximately 10 mL with the new (exchange) solvent then place the sample back into the concentrator and evaporate the extract back down to slightly less than 1 mL (or slightly more if applicable).
- 12.19 Using a 1 mL or 10 mL disposal glass pipette, remove the extract from the concentrator tube and retain the extract in the pipette. Rinse the concentrator tube with a small volume of the appropriate solvent and add this to the volume already in the pipette. Repeat as needed to obtain the targeted final volume and record the final volume in the Organic Extraction Logbook.
- 12.20 Transfer the extract into an appropriate container (e.g. vials, test tubes, etc.), cap the container, and label with the sample I.D., parameter/test, and extraction date.
- 12.21 Extracts are retained in cold storage (4°C) using the cooler in the GC/SVOA lab. If the instrument analysts do not pick up the extracts on the day they were extracted, place the extracts in a labeled solo cup and put into the SVOA extract freezer.

13.0 CALCULATIONS AND DATA HANDLING

- 13.1 Enter the data into LIMS. Details on the procedure for entering preparation data are in the Preparation Batch Data Entry SOP. Samples prepared beyond the extent of the allowable hold time will be automatically identified with a red font in LIMS when samples are added to the Batch Sheet. Data associated with a failed hold time must be documented with a CAR form.

14.0 METHOD PERFORMANCE

- 14.1 Initial Demonstration of Capability study data, Method Detection Limit study data and Performance Testing study data are maintained and available from the QA office.

15.0 POLLUTION PREVENTION

- 15.1 The quantity of chemicals purchased should be based on expected usage during their shelf life and the disposal cost of unused material.
- 15.2 Prepare and use the minimum amount of reagent and standard necessary.

16.0 WASTE MANAGEMENT

- 16.1 Refer to the Sample Disposal SOP for guidance on the disposal of any resulting residue, digestate, distillate, extract or standard.

17.0 REFERENCES

- 17.1 SW-846 Method 3550B
- 17.2 Microbac Laboratories Quality Assurance Plan, current revision, all sections

18.0 TABLES, FORMS, CHECKLISTS, AND OTHER ATTACHMENTS

- 18.1 Spike standard compound tables (2 pages)
- 18.2 Extraction Conditions table (1 page)
- 18.3 Copy of the Organics Extraction Log (1 page)
- 18.4 Copy of the SOP Revision Notification Form denoting changes to this revision. (2 pages)

SECTION 18.1: SPIKE STANDARD COMPOUND TABLES

| Stock Base-Neutral Spike Standard Compounds (Restek #31074) | | |
|---|----------------------------------|---|
| Acenaphthene N-Nitrosodi-n-propylamine | Pyrene 1,2,4-Trichlorobenzene | 1,4-Dichlorobenzene 2,4-Dinitrotoluene |

| Stock Acid Spike Standard Compounds (Restek #31061) | | |
|---|---|---------------|
| Pentachlorophenol Phenol | 2-Chlorophenol 4-Chloro-3-methylphenol | 4-Nitrophenol |

| Stock PNA-IL Surrogate Standard Compounds (Supelco #4-7263) | | |
|---|-----------------|-----------------|
| 2-Fluorobiphenyl | Nitrobenzene-d5 | p-Terphenyl-d14 |

| Stock PNA-IL Spike Standard Compounds (Accustandard #Z-014G-R-PAK) | | |
|---|---|--|
| Naphthalene Acenaphthene Acenaphthylene Fluorene Pyrene Benzo(a)anthracene | Chrysene Carbazole Phenanthrene Benzo(b)fluoranthene Benzo(k)fluoranthene Benzo(a)pyrene | Dibenzo(a,h)anthracene Benzo(g,h,i)perylene Indeno(1,2,3-cd)pyrene Fluoranthene Anthracene |

| Method 625 Custom Standard (Ultra Scientific # CUS-4924) | | |
|--|--|---|
| 1,3-dichlorobenzene 2,4-dinitrophenol 2,6-dinitrotoluene 2-methyl-4,6-dinitrophenol | Benz [a] anthracene Bis (2-chloroethyl) ether Di-n-octyl phthalate | Fluorene Benzo [b] fluoranthene Hexachlorobutadiene |
| Matrix: Methanol | | |

| Method 625 Custom Standard (Ultra Scientific # CUS-5946) | | |
|--|---|---|
| 2-chloronaphthalene 3,3'-dichlorobenzidine 4-chlorophenyl phenyl ether Benzo[ghi]perylene | Benzo[k]fluoranthene Bis(2-chloroisopropyl) ether Bis(2-ethylhexyl) phthalate Dibenz[ah]anthracene | Diethyl phthalate Dimethyl phthalate Indeno[1,2,3-cd]pyrene |
| Matrix: Methanol/Methylene Chloride (1:1) | | |

| Method 625 Custom Standard (Ultra Scientific #CUS-3883) | | |
|--|--|--|
| Acenaphthylene Anthracene Bis(2-chloroethoxy)methane Chrysene | Di-n-butyl phthalate Hexachlorobenzene Naphthalene | 2,4-dichlorophenol 2,4,6-trichlorophenol 2-nitrophenol |
| Matrix: Methanol/Methylene Chloride (1:1) | | |

| Method 625 Custom Standard (Ultra Scientific #CUS-4407) | | |
|---|--|--|
| 1,2-dichlorobenzene 2,4-dimethylphenol 4-bromophenol phenyl ether Benzo[a]pyrene | Butyl benzyl phthalate Fluoranthene Hexachloroethane | Nitrobenzene Isophorone Phenanthrene |
| Matrix: Methanol | | |

| Method 625 Custom Standard (Ultra Scientific #CUS-13706) | | |
|--|--|--|
| N-nitrosodimethylamine 3-methylphenol 4-methylphenol Benzoic acid | 4-chloroaniline 2,4,5-trichlorophenol 2-nitroaniline dibenzofuran | 4-nitroaniline 1,2-diphenylhydrazine Alpha-terpineol Benzyl alcohol |
| Matrix: Methanol/Methylene Chloride (1:1) | | |

| Method 625 Custom Standard (Ultra Scientific Quote Number: 092911-438) | | |
|--|--|---|
| Pyridine Aniline 2-methylphenol 2,6-dichlorophenol | 2-methylnaphthalene Hexachlorocyclopentadiene 3-nitroaniline | N-nitrosodiphenylamine Carbazole Biphenyl |
| Matrix: Methanol/Methylene Chloride (1:1) | | |

| Pesticide Spike Standard Compounds (Restek #32291) | | |
|---|--|--|
| Aldrin Alpha-BHC Alpha-Chlordane Beta-BHC Delta-BHC Dieldrin Endosulfan I | Endosulfan II Endosulfan sulfate Endrin Endrin aldehyde Endrin ketone Gamma-BHC | Heptachlor Heptachlor epoxide Methoxychlor 4,4'-DDD 4,4'-DDE 4,4'-DDT |
| Matrix: Hexane/Toluene (1:1) | | |

SECTION 18.2: EXTRACTIONS CONDITIONS TABLE

| Extraction Conditions | | | |
|------------------------------|----------------------------|---------------------------|-------------------|
| Analyte Group | Final Solvent for Analysis | Final Solvent for Cleanup | Final Volume (mL) |
| Pesticides | Hexane | Hexane | 10 |
| PCBs | Hexane | Hexane | 10 |
| SVOA (BNA) | Methylene Chloride | --- | 1 |
| PAH (HPLC) | Acetonitrile | --- | 1 |
| PAH (PNA) and PNA-IL (GC/MS) | Methylene Chloride | --- | 1 |

Microbac Laboratories - Chicagoland Division
ORGANICS EXTRACTIONS LOG

Date & Time: _____ Analyst: _____ Batch #: _____

Analyte: _____ Matrix: _____ Peer Review: _____

Liquid Reagents: Lot #: Solid Reagents: Lot #:

All 8081/82 waters and 608 waters >3 days from collection, pH = 5-9

Yes[] No [] If "No" explain in Comments.


Pipette Lot Number _____

[illegible]

* Mark as "Y", "N" or "N/A". If chlorine is present dechlorinate with Na₂S₂O₃ and note in Comments.

revision: _f_9/2010


SECTION 18.4: SOP REVISION FORM

| | |
|---|---|
|  | SOP REVISION NOTIFICATION FORM FOR: |
| | PREPARATION OF NON-AQUEOUS SAMPLES USING SONICATION |
| | OLD REVISION: 7 |
| | NEW REVISION: 8 |

CHICAGOLAND

SUMMARY OF CHANGES:

1. Edited section 3.2: "A measured amount of sample, usually 30 grams, is mixed with a drying agent to form a free flowing **powder mixture**. This mixture is then serially extracted with an organic solvent using a sonicator. The extract is dried, concentrated, and, as necessary, exchanged into a solvent compatible with the cleanup or analytical method used."
2. Added to Section 7.11: "Sonicator (Fisher Scientific model 550/500 Sonic Dismembrator with a ¾ inch horn, or equivalent)."
3. Section 8.2: Updated the sources for acetone, n-hexane, methanol, methylene chloride, and sodium sulfate.
4. Deleted what had been section 8.3.5, 8.3.6, 8.3.10, 8.3.11, 8.3.12, and 8.3.13: "[8.3.5] Stock Acid Surrogate Standard, 10,000 µg/mL each: Supelco #4-7260 U. See table in section 18.0 for compound list. [8.3.6] SVOA Surrogate Standard: In a 100 mL volumetric flask, dilute 2.0 mL of the stock base neutral surrogate standard and 1.5 mL of the stock acid surrogate standard to the mark with MeOH. This prepares a standard containing the base neutral compounds at 100 µg/mL and the acid compounds at 150 µg/mL. Add 1 mL of this solution to all samples. [8.3.10] Stock HPLC PNA Spike Standard, Varied concentrations ranging (20-100ug/ml): TCL PAH Mix, Supelco #4-9456. An additional Stock Standard is obtained as per the Client Specifications, but not required; 2-Methylnaphthalene, 100ug/ml: Ultra #SV-200. See table in section 18.0 for compound list. [8.3.11] HPLC PNA Spike Standard, Varied concentrations ranging (0.8-40ug/ml): In a 50 ml volumetric flask, dilute 2.0 ml Stock HPLC PNA Spike Standard and, if applicable, (5ug/ml), 2.5 ml of Stock 2-Methylnaphthalene Spike Standard to the mark with Acetonitrile. **Add 1 ml of this solution to the LCS, MS, and MSD samples.** [8.3.12] Stock HPLC PNA Surrogate Standard, 2000 ug/ml: Accustandard #M-625-04-10X contains Decafluorobiphenyl (DFB). [8.3.13] HPLC PNA Surrogate Standard, 50 ug/ml: In a 50 ml volumetric flask, dilute 1.25 ml of the stock HPLC PNA surrogate standard to the mark with Acetonitrile. **Add 1 ml of this solution to all samples.**"
5. Added section 8.3.5 to the standards: "Stock Method 625 BNA Spike Standard, 400 µg/mL: For additional analytes for Method 625 the following custom standards are used on a rotating basis: (1) Ultra Scientific #CUS-4924; (2) Ultra Scientific # CUS-5946; (3) Ultra Scientific #CUS-3883; (4) Ultra Scientific #CUS-4407; (5) Ultra Scientific #CUS-13706; (6) Ultra Scientific Quote Number 092911-438. See table in section 18.0 for compound list."
6. Added section 8.3.6 to the standards: "Method 625 Spike, 100ug/ml: Using a 100-ml volumetric flask, dilute 25-ml of the Stock Method 625 BNA Spike Standard with an additional 75-ml of Methanol bringing the total volume of prepared standard to 100-ml. This prepares a standard containing the Method 625 analytes at 100 ug/ml. Add 1-ml of this solution to the LCS, MS, and MSD samples."
7. Edited section 8.3.7: "PNA-IL Stock Base-Neutral Surrogate Standard, 5,000 µg/mL each: Supelco #4-7263. See table in section 18.1 for compound list."
8. Added section 8.3.9 to the standards: "Semi-volatile Surrogate Standard Mixture with Indicator: BNA Custom Standard with Indicator: Ultra #SM-333XC-500."

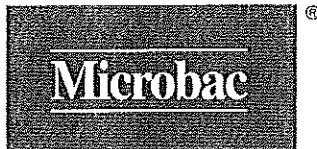
| | | |
|--|--|---|
|  CHICAGOLAND | SOP REVISION NOTIFICATION FORM FOR: | |
| | PREPARATION OF NON-AQUEOUS SAMPLES USING SONICATION | |
| | OLD REVISION: | 7 |
| | NEW REVISION: | 8 |

9. Edited section 8.3.12: "Stock PCB Spike Standard, 1000 µg/mL each ~~Supelco # 4-8997 contains Aroclor 1016 and Supelco #4-4809 contains Aroclor 1260~~ of Aroclor 1016 and 1260: Restek #32039"
10. Edited section 8.3.14: "Stock Pesticide Spike Standard, ~~2000~~ 200 µg/mL each: ~~Supelco #4-8943~~ Restek #32291. See the table in section 18.0 for the compound list."
11. Edited section 8.3.15: "Pesticide Spike Standard, 0.5 µg/mL each: In a ~~400~~ 50 mL volumetric flask, dilute ~~25~~ 125 µL of the Stock Pesticide Spike Standard to the mark with Acetone. **Add 1 mL of this solution to the LCS, MS, and MSD.**"
12. Edited a bullet in section 12.2: "The samples must be properly prepared by thorough mixing with sodium sulfate so that they form a free-flowing ~~powder~~ mixture prior to the addition of the solvent."
13. Deleted a bullet in section 12.2: "This procedure ~~is~~ employs the "low concentration" protocol."
14. Added to section 12.6: "Add enough sodium sulfate to beaker to dry the sample, and mix using the disposable torque depressor. **Nonporous or wet samples (gummy or clay type) that do not have a free flowing sandy texture must be mixed with 60 g of anhydrous sodium sulfate, (or three large scoops). If required, more sodium sulfate may be added.**
15. Added Section 12.7: "**After addition of sodium sulfate, mix well using wooden, disposable depressor until the sample is free flowing.**"
16. Edited section 13.1: "Enter the data into LIMS. Details on the procedure for entering preparation data are in the Preparation Batch Data Entry SOP. Samples prepared beyond the extent of the allowable hold time will ~~automatically show a message similar to "The prep HoldTime was exceeded by 1 days" in the comments field of the Prep Batch screen and report~~ be automatically identified with a red font in LIMS when samples are added to the Batch Sheet. Data associated with a failed hold time must be documented with a CAR form."
17. Edited the Spike Standard Compound Tables in section 18.1 to reflect the changes made in section 8.3 as stated above in this document.
18. Updated the Organics Extraction Log in section 18.3.

By initialing below, I certify that I have been *notified* about the approval of a *new revision* to the above mentioned SOP and that I have read, understand, and agree to follow the test procedure as set forth in this new revision.

| Initials & Date | Initials & Date | Initials & Date |
|-----------------|-----------------|-----------------|
| _____ | _____ | _____ |
| _____ | _____ | _____ |
| _____ | _____ | _____ |
| _____ | _____ | _____ |

Form revised 2/27/12



Microbac Laboratories, Inc.
SOP Revision Request Form

Request

Date: 10/10/2011

Requested by: Donna Ruokonen

SOP ID: 608-8081(6) SOP, 608-8082(6) SOP, 624-8260(6), 625-8270(11), 8151A(1), EXP8330(0), GRO-DRO-TPH(0), TO-11(0), TO15(5), and TPH-ERO(1),

Requested Change(s):
Section 10 for surrogates add the following:

-In the instances where samples are reported from multiple runs, the surrogate(s) must be reported from the first reported run or the least diluted if multiple dilutions exist.

Approval

Unit Supervisor: [Signature]

Date: 10-10-2011

QA Manager: [Signature]

Date: 10-10-2011

Notification

I am aware of these change(s) and I will implement them the next time I perform this procedure.

Initials & Date

Initials & Date

Initials & Date

Distribution

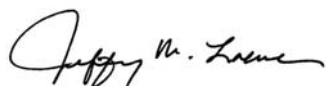
Upon approval and notification, make sufficient copies to be attached to the controlled copies of the SOP, which have been distributed in the lab. Attach this original form to the original SOP maintained in the QA office. Attach the copies of this form to the copies available in the lab. Notify all appropriate personnel of the change approved by this form. The related SOP should be revised ASAP but in no longer than 3 months from the date of this approval.

Upon signed approval and attachment to the above listed SOP, the changes listed are considered required elements of the SOP.

**STANDARD OPERATING PROCEDURE FOR
DETERMINATION OF VOLATILE ORGANIC COMPOUNDS (VOCs)
IN AIR COLLECTED IN SPECIALLY-PREPARED CANISTERS
AND ANALYZED BY GC/MS**

Revision Author: Brian Mills

This SOP is effective upon signed approval by the following:



12/22/2010

Laboratory Director

Date



12/22/2010

QA Director

Date

DISCLAIMER: This SOP has been developed for use at the Microbac Laboratories, Merrillville, Indiana facility. It is intended for use by trained analysts. As written, this SOP may not be specifically applicable to the activities of other organizations.

1.0 TABLE OF CONTENTS

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2.0 SCOPE AND APPLICATION

- 2.1 This is a GC/MS procedure for the determination of Volatile Organic Compounds. This procedure is applicable to the analysis of air matrix samples collected in Summa canisters or Tedlar bags. The applicable analytes, detection limits and routine reporting limits (PQL) are listed at the Limits tab of the applicable test codes in LIMS. For low level reporting limits, GC/MS SIM analysis may be applied.
- 2.2 This method applies to ambient concentrations of VOCs above 0.5 ppbv for regular GC/MS analysis using scan mode and between 0.05 and 0.5 ppbv using SIM. These low detection levels may be attained by concentrating up to one liter of a sample volume. This method applies under most conditions encountered in sampling of ambient air into canisters. However, the composition of a gas mixture in a canister, under unique or unusual conditions, will change so that the sample is known not to be a true representation of the ambient air from which it was taken. For example, low humidity conditions in the sample may lead to losses of certain VOCs on the canister walls, losses that would not happen if the humidity were higher. If the canister is pressurized, then condensation of water from high humidity samples may cause fractional losses of water-soluble compounds. Since the canister surface area is limited, all gases are in competition for the available active sites. Hence absolute storage stability cannot be assigned to a specific gas.

3.0 SUMMARY

- 3.1 The atmosphere is sampled by introduction of air into a specially-prepared stainless steel canister. Both sub-atmospheric pressure and pressurized sampling modes use an initially evacuated canister. A pump-ventilated sampling line is used during sample collection with most commercially available samplers. Pressurized sampling requires an additional pump to provide positive pressure to the sample canister. A sample of air is drawn through a sampling train comprised of components that regulate the rate and duration of sampling into the pre-evacuated and passivated canister.
- 3.2 To analyze the sample, a known volume of sample is directed from the canister through a solid multisorbent concentrator. A portion of the water vapor in the sample breaks through the concentrator during sampling, to a degree depending on the multisorbent composition, duration of sampling, and other factors. Water content of the sample can be further reduced by dry purging the concentrator with helium while retaining target compounds. After the concentration and drying steps are completed, the VOCs are thermally desorbed, entrained in a carrier gas stream, and then focused in a small volume by trapping on a reduced temperature trap or small volume multisorbent trap. The sample is then released by thermal desorption and carried onto a gas chromatographic column for separation. As a simple alternative to the multisorbent/dry purge water management technique, the amount of water vapor in the sample can be reduced below any threshold for affecting the proper operation of the analytical system by reducing the sample size. For example, a small sample can be concentrated on a cold trap and released directly to the gas chromatographic column. The reduction in sample volume may require an enhancement of detector sensitivity. Other water management approaches are also acceptable

provided their usage does not compromise the attainment of the performance criteria listed in Section 11 of the reference method. One alternative for drying the sample is to separate VOCs from condensate on a low temperature trap by heating and purging the trap.

- 3.3 The analytical strategy for the EPA Compendium Method TO-15 involves using a high resolution gas chromatograph (GC) coupled to a mass spectrometer. If the mass spectrometer is a linear quadrupole system, it is operated either by continuously scanning a wide range of mass to charge ratios (SCAN mode) or by monitoring select ion monitoring mode (SIM) of compounds on the target list. If the mass spectrometer is based on a standard ion trap design, only a scanning mode is used (note however, that the Selected Ion Storage (SIS) mode for the ion trap has features of the SIM mode). Mass spectra for individual peaks in the total ion chromatogram are examined with respect to the fragmentation pattern of ions corresponding to various VOCs including the intensity of primary and secondary ions. The fragmentation pattern is compared with stored spectra taken under similar conditions in order to identify the compound. For any given compound, the intensity of the primary fragment is compared with the system response to the primary fragment for known amounts of the compound. This establishes the compound concentration that exists in the sample. Mass spectrometry is considered a more definitive identification technique than single specific detectors such as flame ionization detector (FID), electron capture detector (ECD), photoionization detector (PID), or a multi-detector arrangement of these. The use of both gas chromatographic retention time and the generally unique mass fragmentation patterns reduce the chances for misidentification. If the technique is supported by a comprehensive mass spectral database and a knowledgeable operator, then the correct identification and quantification of VOCs is further enhanced.
- 3.4 A whole air sample is cryogenically trapped in order to remove the matrix and concentrate the analytes. The sample is then thermally desorbed onto a cryofocusing unit, which maintains a tight band of analytes as they are transferred onto a capillary column. The compounds are separated by gas chromatography and detected with a mass spectrometer. Identification of target analytes is accomplished by comparing their mass spectra and retention times with spectra and retention times of standards contained in a database. Quantification is accomplished by comparing the response of major ion (for a particular analyte) relative to an internal standard. This response ratio is then compared to ratios of standards collected under the same conditions.
- 3.5 This procedure is based on the reference methods listed in section 17 of this document. This procedure contains the following deviations from the reference methods. These are not considered significant deviations.
- 3.5.1 Section 10.4.3 of the reference method requires the use of 50 ng BFB for the instrument tune. This procedure uses a lesser amount due to the concentration available in the standard used.

4.0 DEFINITIONS

- 4.1 A list of definitions is in the Quality Assurance Plan. In addition to the terms defined in the QAP, the terms below, are specific and critical to this procedure.

- 4.2 Analytical sequence – time frame in which samples are analyzed. An analytical sequence is limited to 24 hours and 20 environmental samples, whichever is less. An analytical sequence must contain an instrument performance check (Tune standard), appropriate calibration or verification standards, and a method blank.
- 4.3 Summa canister - a stainless steel canister for the collection of ambient air samples.

5.0 INTERFERENCES

- 5.1 Analytes that are not separated chromatographically, but which have different mass-spectra and non-interfering quantitation ions can be identified in the same calibration mixture or air sample. Analytes that have very similar mass spectra cannot be individually identified and measured in the same calibration mixture or air sample unless they have different retention times. Co-eluting compounds with very similar mass spectra—typically many structural isomers—must be reported as an isomeric group or pair. For example, m-Xylene and p-Xylene cannot be separated under the listed chromatographic conditions while 1,3-dichlorobenzene and 1,4-dichlorobenzene are successfully separated.
- 5.2 Interferences to this method result mostly from contamination of sampling containers and gases used in the analysis. Attention to the following details will help minimize the possibility of contamination issues:
- 5.2.1 Canisters must be stored in a contaminant-free location and should be capped tightly during shipment to prevent leakage and minimize any compromise of the sample.
- 5.2.2 Impurities in the calibration dilution gas and carrier gas, organic outgassing from the system components ahead of the trap, and solvent vapors in the laboratory account for the majority of contamination of problems. The analytical system is demonstrated to be free of contamination on a daily basis by the analysis of humidified zero-air blanks (daily blanks).
- 5.2.3 Significant contamination of the analytical system may occur whenever samples containing high concentrations of VOCs are analyzed. This in turn can result in carryover contamination in subsequent analyses. Whenever a high concentration sample is encountered (>25 ppbv of a trace species), it should be followed by an analysis of humid zero air to check for carry-over contamination.

6.0 SAFETY

- 6.1 Consult the current revision of the Chemical Hygiene Plan. Requirements for the use of personal protective equipment (e.g. safety glasses, lab coats, gloves) as well as other area-specific safety requirements (e.g. gas cylinders) and MSDS sheets are addressed in the CHP. NOTE that this procedure uses liquid nitrogen, thus the CHP section on Cryogenics is of critical importance.

7.0 EQUIPMENT AND SUPPLIES

- 7.1 The following is a list of materials needed to perform the steps of this procedure as written. See the reference method(s) for equipment and supply specifications.
- 7.2 All volumetric glassware used shall be ASTM Class A. Class B glassware must be verified for accuracy on an annual basis and labeled with an appropriate correction.
- 7.3 Entech 7100A Automated Preconcentrator (cryogenic sample concentrator)
- 7.4 Traps (ordered through Entech)
- 7.5 Empty Trap, cryomodule
- 7.6 1/8 Tenax Trap, cryomodule
- 7.7 Focusing trap
- 7.8 HP 5890 series II+ Gas Chromatograph
- 7.9 Column Restek RTX-1 60m, 0.32 mm ID, 1.0 μ m
- 7.10 HP5972 Mass Selective Detector
- 7.11 HP59822B Ionization Gauge Controller
- 7.12 Data station
- 7.13 Chemstation software version G1701BA
- 7.14 Entech software version 3.66
- 7.15 Entech 4600A Dynamic Diluter
- 7.16 Entech 3100A Can Cleaning System
- 7.17 Summa canisters, 1 L & 6 L volume: Leak-free stainless steel pressure vessels with valve and specially prepared interior surfaces
- 7.18 Tedlar bags or equivalent
- 7.19 Porters model 201 Mass Flow Controller
- 7.20 Vacuum pumps
- 7.21 UHP Helium – column carrier gas
- 7.22 Liquid Nitrogen – cryogen gas
- 7.23 Zero Grade Nitrogen – concentrator gas
- 7.24 Digital timer: Fisher 15-077-965 or equivalent

7.25 Critical Orifices

7.26 Pressure Gauge: Matheson 63-3704A or equivalent

7.26.1 NOTE: The status of the Canisters, goosenecks, regulators/gauges and orifices is maintained using the SQL database, TO15SQL, which is located on CHIWSA (example attached). It can be accessed by using an shortcut icon, on the computer system controlling the GC/MS.

8.0 REAGENTS AND STANDARDS

8.1 All standards must be traceable to NIST, when available. Certificates of traceability must be obtained from the manufacturer. All standards must be documented in the appropriate preparation logbook. Refer to the requirements in the Labeling of Standards, Reagents, Digestates and Extracts SOP.

8.2 Reagents

8.2.1 Lab pure water (DI water): Analyte free water is prepared as described in the Quality Assurance Plan. DI water may be obtained from any of the designated taps throughout the lab.

8.3 Standards (SCAN Mode)

8.3.1 All stock standards are stored in the pressurized cylinders, located in the GC/MS TO-15 room unless otherwise noted. Unless otherwise noted, prepared standards are stored in 6 Liter summa canisters, and prepared on an as needed basis.

8.3.2 Stock Calibration Standard, 100 ppbv: Restek catalog #34437 or equivalent. See section 18 for a list of analytes.

8.3.3 Working Calibration Standard, 10 ppbv or 20 ppbv. Prepare the working standard as follows. Prepare a new standard every 30 days or sooner as needed.

8.3.3.1 Using the 4600a's dynamic dilution feature Make sure the Stock calibration standard is connected to one of the three MFC ports usually Module 2.

8.3.3.2 Several of the routine standards are saved in the software. On the main screen of the 3600 software select open and then select TO1510ppb. This will load of the following two parameters for the calibration standard: (1) 450 mL/min of zero air (module 1) and 50 mL/min of standard (module 2) for 10 ppbv std; (2) 200 mL/min of zero air (module 1) and 50 mL/min of standard (module 2) for 20 ppbv std.

8.3.3.3 $C_f = C_i (f_i / f_t) = C_i (f_i / f_s + f_d)$

Where:

C_i = Initial concentration of standard

C_f = Final concentration desired

f_i = Flow rate from specific analyte cylinder

f_d = Flow rate of dilution gas

f_s = Sum of flow rates from all standard channels 2-6

f_t = Total flow rate

8.3.3.4 Check level of water in humidifier on back of diluter. The level should be 25-50% full.

8.3.3.5 Select "GO" and allow the standard to be generated for about 5 minutes to create equilibrium. Then open the front green Swagelok valve slightly to flush the lines. At this time remember to add 50 µL of DI water to the top of the Summa can before attachment. Connect the canister, to the flushing line and close the green Swagelok valve.

8.3.3.6 Temporarily open the canister valve to evacuate the line, then close it. Verify for 30 seconds that the vacuum in the outlet line remains constant between 0-2 psi as displayed in the 4600A application software. If it does not, there is a leak in the can connection. Correct the leak and repeat the leak test prior to filling the can.

8.3.3.7 When the canister is opened the internal pressure will be displayed on the screen. When the canister is about 29.4 psi (2 atm) close the canister valve, and green Swagelok valve, then select "STOP".

8.3.3.8 To clear the mixing chamber, enter 1000 mL/min for module 1 and 0 mL/min all other MFC and select "Go". Allowing this to run for 5-10 minutes will insure a clean chamber for the next standard.

8.3.3.9 Prepare a new standard every 30 days or sooner as needed.

8.3.4 Stock Cumene /Naphthalene Calibration Standard, 100 ppbv: Restek catalog #561647 or equivalent.

8.3.5 Working Cumene/Naphthalene Calibration Standard, 10 ppbv: Prepare this working standard as detailed above for the working calibration standard.

8.3.6 Stock Internal Standard / Surrogate Standard, 1000 ppbv: Restek catalog # 34408 or equivalent. This standard contains the following analytes:

- Bromochloromethane (I.S.)
- 1,4-Difluorobenzene (I.S.)
- Chlorobenzene-d5 (I.S.)
- 4-Bromofluorobenzene (SURR)

8.3.7 Working I.S. / SURR Standard, 100 ppbv: Prepare the working I.S. / SURR standard by following the steps (above) for the 10 ppbv working calibration standard with the following listed exceptions. Prepare a new standard every 30 days or sooner as needed.

- 8.3.7.1 Only internal standard will be attached to MFC module 3 On the main screen of the 4600 software select open and then select IS100ppb. This will load the following parameters for the calibration standard: 450 mL/min of zero air (module 1) and 50 mL/min of standard (module 3).
- 8.3.8 Stock Verification Standards, 100 ppbv: Restek catalog #344421 TO-14A calibration mix or equivalent and Restek catalog # 34435 TO-15 Subset 25 compound Mix. These standards must be of a source/lot that is different than that used for instrument calibration.
- 8.3.9 Working Verification Standard, 10 ppbv or 20 ppbv: Prepare the working standard by following the steps (above) for the 10 ppbv or 20 ppbv working calibration standard. Prepare a new standard every 30 days or sooner as needed.
- 8.3.9.1 On the main screen of the 4600 software select open and then select spike10 ppb or spike 20 ppb. This will load the following parameters for the verification standard: 400 mL/min of zero air (module 1), 50 mL/min of standard (module 2) and 50 mL/min of standard (module 4) for 10 ppbv and 150 mL/min of zero air (module 1), 50 mL/min of standard (module 2) and 50 mL/min of standard (module 4) for 20 ppbv.
- 8.4 Standards (SIM Mode)
- 8.4.1 2.5 ppbv Intermediate Standard: Dilute 1000 ppb Stock Standard 400:1. Flow 3990 sccm of zero air and 10 sccm of 1000 ppb std to 2 ATM using the 4600 diluter.
- 8.4.2 0.25 ppbv Working Standard: Dilute 2.5 ppb Standard 10:1. Flow 100 sccm of 2.5 ppb std for 6 minutes into an evacuated 6L canister (= 600cc) using MFC. Pressurize to 1ATM using the 4600 diluter.
- 8.4.3 1.0 ppbv ISTD/SURR standard in Can: Dilute 1000 ppb Stock Standard 250:1. Flow 2490 sccm of zero air and 10 sccm of std to 7.5 ATM using the 4600 diluter. Dilute to 30 ATM using the 4600 diluter. ISTD can is 1.0 ppb in the can, and 0.1 ppb on column.

9.0 SAMPLE COLLECTION, PRESERVATION, AND HOLDING TIMES

- 9.1 The client or other trained personnel collect samples. Samples received at the laboratory are considered representative unless otherwise noted.
- 9.2 Samples should be collected in a summa canister. The canisters are appropriately cleaned and prepared by the laboratory. The canisters are evacuated to a vacuum of -29 in Hg. No chemical preservation is applicable. Samples are stored at ambient temperature and pressure in the VOA lab.
- 9.3 Analysis must be performed within the maximum allowable hold time of 30 days from collection.

10.0 **QUALITY CONTROL**

10.1 An *Initial Demonstration of Capability* study must be performed prior to the initial analysis for each analyst and whenever substantial change has occurred in the procedure or instrument. This is accomplished by meeting the replicate precision and audit accuracy criteria as described in sections 11.3 and 11.4 of the reference method.

10.2 QC samples must be run on same instrument and on the same analytical sequence as the samples.

10.2.1 **Replicate Precision**

10.2.1.1 The measure of replicate precision used for this program is the absolute value of the difference between replicate measurements of the sample divided by the average value and expressed as a percentage as follows:

$$\text{Percent Difference} = \frac{|A-B|}{C} \times 100$$

Where:

A = First measurement value

B = Second measurement value

C = Average of the two values

10.2.2 **Audit Accuracy**

10.2.2.1 A measure of analytical accuracy is the degree of agreement with audit standards. Audit accuracy is defined as the difference between the nominal concentration of the audit compound and the measured value divided by the audit value and expressed as a percentage, as illustrated in the following equation:

$$\text{Audit Accuracy, \%} = \frac{\text{Spiked value} - \text{Observed value}}{\text{Spiked value}} \times 100$$

10.3 A *Method Detection Limit* study must be performed for each new procedure, annually thereafter, and whenever a change in instrument occurs. Refer to the Capability and Detection Limit Studies SOP for details. An MDL qualitative verification QC (LOQ), sample must follow the MDL study on each instrument at 1-4X the MDL concentration and quarterly thereafter.

- 10.4 A *BFB Tune Check* must be performed at the beginning of each 24 hour sequence. The analysis is performed by trapping BFB using the sample pre-concentration parameters. The working tune solution (which contains 4-Bromofluorobenzene) must meet the following ion abundance criteria. If the criteria are not met, reanalyze. An acceptable tune must be obtained before samples can be analyzed.

| BFB (4-BROMOFLUOROBENZENE) KEY IONS and ION ABUNDANCE CRITERIA | |
|---|------------------------------------|
| Mass | Ion Abundance Criteria |
| 50 | 8 – 40% of mass 95 |
| 75 | 30 – 66 % of mass 95 |
| 95 | Base peak, 100% relative abundance |
| 96 | 5 – 9 % of mass 95 |
| 173 | < 2 % of mass 174 |
| 174 | 50 – 120 % of mass 95 |
| 175 | 4 – 9 % of mass 174 |
| 176 | 93 – 101 % of mass 174 |
| 177 | 5 – 9 % of mass 176 |

- 10.5 *Internal Standards* (I.S.) must be added to all standards, QC samples and environmental samples.
- 10.5.1 Acceptance criteria for all standards and samples (i.e. ICAL, QC and environmental) are:
- Area at each level within $\pm 40\%$ of mean area
 - RT ± 20 seconds (0.33 minutes) of the mean retention time over the calibration range for each internal standard
- 10.5.2 These criteria are evaluated by the data system and printed on the Continuing Calibration Report. Failures in the CCV are automatically flagged on this report.
- 10.5.2.1 If the acceptance criteria are not met for the CCV, perform instrument maintenance then reanalyze the CCV or perform a new initial calibration.
- 10.6 *Surrogate* (SURR) compounds are added to all quality control samples, blanks, and samples.
- 10.6.1 Acceptance criteria are the nominal limits listed in the appropriate test code in LIMS. These limits are based on the Audit Accuracy criteria in the reference method.
- 10.6.2 Surrogate standards that fail to meet the acceptance criteria are automatically flagged red at Data Entry/Review in LIMS and with an “S” qualifier at reporting.
- 10.6.3 The reporting of data associated with failing surrogate standards must be documented with a CAR form.

- 10.6.4 If the acceptance criteria are not met, reanalyze the sample. If insufficient sample is available for reanalysis, report the original result with a Case Narrative to the client. If reanalysis fails to meet the acceptance criteria the original results should be reported with a Case Narrative to the client. If reanalysis does meet the acceptance criteria but was performed beyond the maximum hold time both sets of results should be reported to the client with an appropriate Case Narrative.
- 10.7 An *Initial Calibration Verification* (ICV) standard must be analyzed after the initial linearity (ICAL) and prior to the analysis of environmental samples. This is the analysis of a second source standard.
- 10.7.1 Acceptance criteria are a response <30% deviation from the initial calibration standard. NOTE: if linear regression was used to assess the linearity of the initial calibration, percent drift is used in place of percent deviation. Two compounds are allowed above the 30% deviation but must be less than 40%. If the acceptance criteria are not met, reanalyze. If the acceptance criteria are not met, reanalyze. If reanalysis fails to meet the acceptance criteria, stop analysis, make new standards, recalibrate or report data with an appropriate qualifier.
- 10.7.2 ICV standards that fail to meet the acceptance criteria are automatically flagged red in Data Entry/Review in LIMS and with an "S" qualifier at reporting.
- 10.7.3 The reporting of data associated with a failed ICV must be documented with a CAR form. If the failure is considered to have a significant affect on the data, client notification is required using the Case Narrative of the report.
- 10.7.4 Samples associated with an ICV that fails with a positive bias can be reported without narration if the sample concentration is below the reporting limit.
- 10.8 A *Calibration Verification Standard* is a mid-level calibration source standard and must be analyzed at the beginning of each analytical sequence following an acceptable instrument tune. The 24 hour analytical sequence begins with the injection of BFB, continues through the analysis of the CCV, samples and QC samples. The tune is evaluated as a component of the CCV.
- 10.8.1 Acceptance criteria are a response <30% deviation from the initial calibration standard. NOTE: if linear regression was used to assess the linearity of the initial calibration, percent drift is used in place of percent deviation. Two compounds are allowed above the 30% deviation but must be less than 40%. If the acceptance criteria are not met, reanalyze. If reanalysis fails to meet the acceptance criteria, stop analysis and recalibrate or report data with an appropriate qualifier.
- 10.8.2 The reporting of data associated with a failed CCV must be documented with a CAR form. If the failure is considered to have a significant affect on the data, client notification is required using the Case Narrative of the report.
- 10.8.3 Samples associated with a CCV that fails with positive bias can be reported without narration if the sample concentration is below the reporting limit.

- 10.9 A *Method Blank* (MBLK) must be prepared and analyzed with each analytical sequence. A MBLK is an unused, certified canister that has not left the building. The blank canister must be filled with humidified, ultra-pure zero air and processed using the same procedural steps as an environmental sample.
- 10.9.1 The acceptance criteria are the Internal Standard criteria as well as < PQL for the target analytes. If the acceptance criteria are not met, reanalyze. If reanalysis fails to meet the acceptance criteria, stop analysis and recalibrate or report data with an appropriate qualifier.
- 10.9.2 MBLKs that fail to meet the acceptance criteria cause the sample results to be automatically flagged red in LIMS and associated samples are flagged with a "B" qualifier.
- 10.9.3 The reporting of data associated with a failed control sample must be documented with a CAR form. If the failure is considered to have a significant effect on the data, client notification is required using the Case Narrative of the report.
- 10.9.4 Samples associated with a MBLK that fails with positive bias can be reported without narration if the sample concentration is < PQL or greater than 10-times the blank contamination.
- 10.10 A *Laboratory Control Sample* is a second source standard that must be prepared and analyzed with each analytical sequence.
- 10.10.1 Acceptance criteria are a response <30% deviation from the initial calibration standard. NOTE: if linear regression was used to assess the linearity of the initial calibration, percent drift is used in place of percent deviation. Two compounds are allowed above the 30% deviation but must be less than 40%. If the acceptance criteria are not met, reanalyze. If reanalysis fails to meet the acceptance criteria, stop analysis and recalibrate or report data with an appropriate qualifier.
- 10.10.2 LCSs that fail to meet the acceptance criteria are automatically flagged red in LIMS at Data Entry/Review with an "S" qualifier at reporting.
- 10.10.3 The reporting of data associated with a failed LCS must be documented with a CAR form. If the failure is considered to have a significant effect on the data, client notification is required using the Case Narrative of the report.
- 10.10.4 Samples associated with a LCS that fails with positive bias can be reported without narration if the sample concentration is below the reporting limit.
- 10.11 A *Laboratory Control Duplicate Sample* is a second source standard that must be prepared and analyzed with each analytical sequence.
- 10.11.1 Acceptance criteria are the nominal limits listed in the appropriate test code in LIMS. These limits are based on the Audit Accuracy criteria in the reference method. These limits are based on the Replicate Precision criteria in the reference method. If the acceptance criteria are not met, reanalyze. If

reanalysis fails to meet the acceptance criteria, stop analysis and recalibrate or report data with an appropriate qualifier.

- 10.11.2 LCSDs that fail to meet the acceptance criteria are automatically flagged red at Data Entry/Review in LIMS and with an "S" qualifier at reporting. Those that fail to meet the precision criteria are automatically flagged red at Data Entry/Review in LIMS and with an "R" qualifier at reporting.
- 10.11.3 The reporting of data associated with a failed LCSD must be documented with a CAR form. If the failure is considered to have a significant affect on the data, client notification is required using the Case Narrative of the report.
- 10.11.4 Samples associated with a LCSD that fails with positive bias can be reported without narration if the sample concentration is below the reporting limit.
- 10.12 A *Duplicate* analysis must be performed with each analytical sequence.
- 10.12.1 Acceptance criteria are the nominal limits listed in the appropriate test code in LIMS. These limits are based on the Replicate Precision criteria in the reference method.
- 10.12.2 DUPs that fail to meet the precision criteria are automatically flagged red at Data Entry/Review in LIMS and with an "R" qualifier at reporting.
- 10.12.3 The reporting of data associated with a failed DUP must be documented with a CAR form. Client notification is required using the Case Narrative of the report.
- 10.12.4 If the concentration measured in the sample is less than 4 times the reporting limit, the RPD calculation and criteria are not applicable and the precision criteria are results that are ± 1 PQL of each other.

11.0 CALIBRATION AND STANDARDIZATION

- 11.1 Calibration data is documented and retained using the reports from the instrument software. Analytical data must be maintained in accordance with the document control requirements in the Quality Assurance Plan as well as the Document Control SOP.
- 11.2 Perform the required preventative maintenance as necessary. Documentation is retained in the Maintenance Log for the particular instrument used for analysis.
- 11.3 Verify an adequate supply of instrument gases and verify the settings on the pressure regulators. The liquid Nitrogen tank should be fully open, UHP Helium at 60-psi on pressure regulator, and Zero Nitrogen at 60-psi on pressure regulator.
- 11.4 Bake out the system as follows:
 - 11.4.1 On the computer open the Open File Folder and select the FLUSH sequence.
 - 11.4.2 Check that all the inlet positions on the preconcentrator are closed off.

- 11.4.3 Select the "FLUSH" button on the menu bar.
- 11.4.4 After the flush sequence is complete, select the "BAKE" option on the menu bar.
- 11.4.5 Set the temp on the GC oven to 200°C to bake out the column at the same time as the modules.
- 11.4.6 When the bake cycle is complete on the concentrator, return the GC temp to 35°C.
- 11.4.7 The system is ready for use.
- 11.5 A new Initial Calibration (linearity; ICAL) is required as listed below. The concentration of the low standard must be set at or below the PQL for each compound.
- CCV failure that is uncorrectable through appropriate instrument maintenance
 - New column
 - Change electron multiplier
- 11.6 Calibrate the instrument as follows for. The instrument conditions are detailed in section 18.
- 11.6.1 A calibration sequence must start with a BFB tune followed by the calibration standards then an ICV standard.
- 11.6.2 Calibration standards are diluted to desired levels by controlling the flow rate of Working Standard into the cryogenic trap. Internal standards are introduced onto the cryogenic trap prior to introduction of the standard/sample. The various calibration levels are obtained according to the following table.
- 11.6.3 (SCAN Mode)

| Calibration Level | Analyte Concentrations (ppbv) | 20 ppbv Working Standard Volume (cc) | 100 ppbv IS/Surrogate Volume (cc) |
|-------------------|-------------------------------|--------------------------------------|-----------------------------------|
| 1 | 0.5 | 12.5 | 50 |
| 2 | 1 | 25 | 50 |
| 3 | 2 | 50 | 50 |
| 4 | 10 | 250 | 50 |
| 5 | 20 | 500 | 50 |
| 6 | 40 | 1000 | 50 |

NOTES:

- The 100 ppbv Internal Standard Working Standard canister is connected to the Internal Standard port and is not removed until it needs replaced due to expiration or depletion.
- The 20 ppbv Calibration Working Standard is connected at the Calibration Standard port and it is not removed until it needs to be replaced.
- 50 cc of the 100 ppbv Internal Standard is concentrated on the trap for each run prior to the concentration of the samples, standards, or blanks. This does not affect the volume concentrated from the samples, standards, blanks.

11.6.4 (SIM Mode)

| Calibration Level | Analyte Concentrations (ppbv) | Volume (cc) | IS/Surrogate Volume (cc) |
|---|-------------------------------|-------------|--------------------------|
| 1 | .0125 | 25 | 50 |
| 2 | .0250 | 50 | 50 |
| 3 | .050 | 100 | 50 |
| 4 | .0625 | 125 | 50 |
| 5 | .100 | 200 | 50 |
| 6 | .125 | 250 | 50 |
| 7 | std can.250 | 500 | 50 |
| 8 | std can=2.5ppb .500 | 100 | 50 |
| NOTES: <ul style="list-style-type: none"> Instrument quant method is set to report in ppt, not ppb. | | | |

11.7 Generate the corresponding Quantitation Reports, BFB "Tune" Report, and Response Factor Report.

11.7.1 The software calculates RF values for each standard as well as the average RF of all analytes.

11.7.2 On the BFB Report, evaluate the Pass/Fail column. An acceptable tune will pass each of the criteria. Calibration can not continue with a failed tune. Details of Tune evaluation include:

11.7.2.1 All spectra must be background-corrected using a single scan acquired no more than 20 scans prior to the beginning of the BFB peak. Do not subtract part of the BFB peak.

11.7.2.2 Initial evaluation should use the auto-evaluate feature of the software. This feature calculates the average of the apex of the peak as well as the scans immediately on either side of the apex and automatically background corrects using a single scan no more than 20 scans prior to the beginning of the peak.

11.7.2.3 In the event that the auto-evaluation function can not be used as is the case when tuning from a CCV standard, the following procedure should be used:

11.7.2.3.1 Zoom in on the DFTPP peak: click and drag a box around the peak with the left mouse button.

11.7.2.3.2 Enter the average of the spectra across the DFTPP peak: click and drag the right mouse button from 7.54 to 7.62 minutes. (The spectrum should now be displayed in window #1.)

11.7.2.3.3 Position the cursor at the scan at 7.50 minutes and double-click the right mouse button. This selects the background spectrum.

11.7.2.3.4 Select **Tuner/Subtract** to subtract the background spectrum from the average spectrum.

- 11.7.2.3.5 Select **Tuner/Evaluate DFTPP to Screen**. Inspect the tune evaluation report. After inspecting the report close to the MultiVu file.
- 11.7.2.4 Appropriate corrective action (e.g. instrument maintenance, new standard, re-calibration, etc) must be performed if an instrument continues to fail the tune criteria.
- 11.8 The acceptance criteria for an acceptable ICAL are as follows:
- Minimum of 5 levels;
 - %RSD for each analyte must be < 30%; two compounds may have a RSD as high as 40%;
 - The RRT for each compound at each calibration level must be within 0.06 RRT units of the mean RRT for that compound;
 - The area response of each calibration level must be within 40% of the mean area response over the initial calibration range for each internal standard;
 - The retention time shift for each of the internal standards at each calibration level must be within 20 seconds of the mean retention time over the initial calibration range for each internal standard.
- 11.9 If the linearity requirements are not met, take appropriate corrective actions and recalibrate. If the acceptance criteria are met, rename the method "TOMmdd" where mmdd designates the month and date of the new linearity.
- 11.9.1 Dropping levels from the calibration curve is allowable under the following conditions.
- Points may not be dropped solely to meet the acceptance criteria. There must be a justifiable reason for excluding a given standard.
 - Individual analytes may be eliminated from the low or high points
 - Dropping a mid-level standard requires that all analytes be eliminated from that level
 - The required minimum number of calibrated levels remains
 - If the low-level standard is removed from the curve, the PQL must be adjusted accordingly
- 11.10 Analyze an ICV standard. The acceptance criteria must be met before continuing with sample analysis. Analysis of environmental samples cannot proceed without the generation of an acceptable linearity and an acceptable initial verification.
- 11.11 If time remains in the 24 hour time period after meeting the acceptance criteria for the initial calibration, samples may be analyzed. If time does not remain in the 24 hour period after meeting the acceptance criteria for the initial calibration, a new analytical sequence shall commence with the analysis of the instrument performance check standard followed by analysis of a daily calibration standard.

12.0 PROCEDURE

- 12.1 Samples analyzed are documented on the TO-15 Run Log (copy attached). Analytical data is documented and retained using the reports from the instrument software. Analytical data must be maintained in accordance with the document control requirements in the Quality Assurance Plan as well as the Document Control SOP.

12.2 Prep Batch Creation

- 12.2.1 Make a batch in Element.
- 12.2.2 Make a bench sheet in Element. Use the comment field to enter if the dilution was done in the sample container.
- 12.2.3 Make sequence in Element. Add tune, CCV, QC samples, and batch samples. Enter the standard ID and ISTD ID for tune and ccv.
- 12.2.4 Run samples.
- 12.2.5 Look in c:\Smart for daily .REP file for the actual sample amount analyzed by concentrator.
- 12.2.6 Enter the actual sample amount into the bench sheet as the initial and final volume.
- 12.2.7 Using 500cc as the normal sample amount, divide [500/actual sample amt analyzed] to calculate the dilution factor [DF].
- 12.2.8 Multiply the DF by any dilution done in the sample container before analysis.
- 12.2.9 Enter the total dilution amount in the Chemstation multiplier field.
- 12.2.10 Import data into LIMS.
- 12.3 All of the various procedural steps below are performed in the VOA lab.

12.4 Canister Orders

- 12.4.1 When summa canisters are needed for client projects, Project Manager's either enter the details themselves or email project details to analyst who enters this information into the TO-15 Canister Order Log. Information recorded includes:
 - Date of Request
 - Project Manager
 - Client Name and Project
 - Canister Size Needed
 - Flow Rate Needed
 - Grab or Composite Sampling
 - Due Date (date when needed)
 - Date of Order
 - Complete Date

12.5 Canister Cleaning

- 12.5.1 Summary: Canisters are cleaned using the Entech 3100A automated can cleaning system. Cans are evacuated down to 200 mtorr at $85 \pm 5^{\circ}\text{C}$ then pressurized up to 20 psi and re-evacuated. This is done a minimum of six times leaving the canisters at a 50 mtorr final vacuum. A humidification chamber is used to add moisture to the flush gas for assistance in displacing

VOCs off the interior surface of the canisters and manifold tubing. All canisters are pressurized to 20 psi to 30 psi for leak checking and batch analysis to certify that they are clean. If the batch QC canister shows the presence of analytes over detection limits, the entire batch is re-cleaned and re-certified. Alternately each canister can be individually analyzed for the presence of analytes over the detection limit.

- 12.5.2 Check to maintain water level in the humidification chamber at 20-50%.
- 12.5.3 Open doors of Entech 3100A oven and remove the plugs from the cleaning ports to be used. Six to eight canisters can be attached to the manifold depending on their size.
- 12.5.4 Attach each canister inlet to an uncovered port. Do not open canister valves yet.
 - 12.5.4.1 Do not put canisters above 15-psi on the cleaning system. Vent excess pressure in a hood.
 - 12.5.4.2 Plug unused positions on the manifold.
- 12.5.5 Open the can cleaning software on the TO15 system data station and select the "OPEN" folder.
- 12.5.6 Select the appropriate program –
 - 12.5.6.1 "CANCLEAN" for ambient level canisters (ppb – sub ppb range)
 - 12.5.6.2 "CANCLEAN DIRTY" for high level source canisters (>1ppm range)
- 12.5.7 From the "Run" screen, click the rough pump button. Evacuate the cans until the pressure reading is <1PSIA. Click the "All Off" button and observe that the pressure does not rise above 1PSIA after 30 seconds. This step ensures that there are no leaks in the canister connections or thru the canister valves.
- 12.5.8 Open the valves on the canisters.
- 12.5.9 Go back to the Run Control Screen and click on "GO" to start the automated can cleaning cycles.
- 12.5.10 After all cycles are complete the Run Control Screen will display "RUN COMPLETE" in the upper right hand corner. Click on the "STOP" and "ALL OFF" buttons
- 12.5.11 A log of all cleaning and verification is maintained. A copy of the Canister Cleaning Log is provided in section 18.

12.6 Verifying Canister Cleanliness

- 12.6.1 After completing the Canister Cleaning procedure (above), close the valve of one canister. Designate this can with yellow tape. Click the "DILUENT" button on the RUN control screen.

- 12.6.2 Let the pressure in the can go up to about 44-50PSIA on the screen then hit the "STOP" and "ALL OFF" buttons.
- 12.6.3 Open the valve on the closed canister and allow gas from all canisters to fill the canister that will be analyzed for this batch. Close the valve on all the canisters then remove them from the oven.
- 12.6.4 Attach a pressure gauge to each can and record the reading as the initial pressure on a hang tag.
- 12.6.5 Attach the hang tag to each can. Write the composite can number, date of cleaning and oven number on the tag.
- 12.6.6 The cleaned and pressurized canister must "age" a minimum of 12 hours before analysis for verification of cleanliness. Recheck the can pressures and record as the Final pressure on each hang tag.
- 12.6.7 After QC canister is analyzed and show to be <pql for all analytes, write the certified date and instrument number on the hang tag. NOTE: the canisters are stored at pressure for up to 60 days and must be read again before canister is taken for final evacuation.

12.7 Final Evacuation for Sample Collection

- 12.7.1 Canisters require re-evacuation after the canister has been cleaned and verified. Open the can cleaning software and select the "CANCLEAN" option for re-evacuation.
- 12.7.2 Attached the cans in the oven and leave the can valves closed. Hit the rough pump button and allow the pressure to go <1PSIA. Select "All Off" and verify the pressure does not rise above 1PSIA after 30 seconds. If it does, fix the leak in the valve connection and repeat the leak check. Open the can valves and select the rough pump button. When the pressure is < 3.0 PSIA, select the HV pump button and evacuate to < 50-mTorr. Close the canister valves, remove the canisters from the oven and attach a pressure gauge to the canister valve.
- 12.7.3 Open the valve and record the pressure reading from the gauge. The pressure needs to be -26-in Hg (-14-psi) or lower. Place a Microbac sticker tag on the hang tag and record the reading as the starting pressure on the sticker.
- 12.7.4 Close the valve, remove the gauge and place a Swagelok endcap over the valve inlet.
- 12.7.5 Complete the sticker label by filling out the can ID, individually or batch certified, and the prep date (sent to client date).
- 12.7.6 It is recommended that samples be taken within 30 days of evacuation.

12.7.7 Record the cleaning and validation of canisters in the Canister Cleaning and Tracking Log. The following information must be recorded:

- Canister ID
- Gauge
- Canister Size
- Date received
- Status of canister: **O**=Out; **P**=In Process; **V**=Verified
- Date Cleaned
- Oven Number
- QC Instrument Number
- QC Analysis Date
- Analyst's Initials
- Final Pressure
- Date Sent
- Project

12.8 Tedlar Bag Cleaning

- 12.8.1 Connect the bag to inlet of a vacuum pump.
- 12.8.2 Pull a vacuum to evacuate the bag until the bag becomes flattened.
- 12.8.3 Fill the bag with Nitrogen.
- 12.8.4 Repeat the evacuation and fill steps above seven more times for a total of eight cycles leaving the bag full with Nitrogen after the last cycle.
- 12.8.5 Tag the bag as "Clean" and place the bag in the designated area to be verified clean as needed (before shipment to client).

12.9 Setting the Flow of Veriflow Controllers

- 12.9.1 Determine flow rate necessary and orifice size

Flow Rate (mL/min) = Canister Volume (mL) / (Composite hr X 60 min/hr)
(see examples Table I)

| Orifice Size | Flow Rate (mL/min) |
|--------------|--------------------|
| 0.008 | 0.5-2 |
| 0.0012 | 2-4 |
| 0.0016 | 4-8 |
| 0.0020 | 8-20 |
| 0.0030 | 20-40 |
| 0.0060 | 80-350 |

- 12.9.2 Select appropriate flow controller/orifice combination from cabinet.
- 12.9.3 Connect flow controller to single port in the canister cleaning oven, using Swagelok Union. This connection does not have to be tight. (setting of the controller works best at about -20 in Hg).

- 12.9.4 Connect the rotameter to the gooseneck using the union connected to the rotameter tubing, (this connection should be tight).
- 12.9.5 From the NT3100 Cleaner software select "Open" then "CANCLEAN.M30" then "RUN." This will open the "Run Control Screen." Select "Rough Pump." This will pull a vacuum across the flow controller.
- 12.9.6 Remove the cover from the back of the flow controller using 1/8" hex wrench. The adjustment screw under this cap is also 1/8 hex. Turn the set screw until the desired flow is achieved. [NOTE: Rotameter flows are relative to the type of float being used. A float calibration scale is included in section 18.]
- 12.9.7 Reinstall the back cover; turn off the vacuum by selecting "ALL OFF" on the run control screen. Disconnect the rotameter and flow controller from the oven.
- 12.9.8 Place the flow controller in a bubble wrap bag, then into its box and ship on the top of the canister in the canister shipping container.

| Hourly Composite | 6 L Canister (mL/min) | Orifice | 1 L Canister (mL/min) | Orifice |
|------------------|-----------------------|-------------------|-----------------------|---------|
| 48 | 2 | 0.0012 | --- | --- |
| 24 | 4.1 | 0.0012 or 0.0016 | --- | --- |
| 12 | 8.3 | 0.0016 or 0.0020 | 1.4 | 0.0012 |
| 8 | 12.5 | 0.0020 | 2 | 0.0012 |
| 3 | 33.3 | 0.0030 | 5.5 | 0.0016 |
| 1.5 | 66.6 | 0.0060 | 11 | 0.0020 |
| 1 | 100 | Direct connection | 16.6 | 0.0020 |

12.10 Daily Verification

- 12.10.1 The ICAL must be verified each "analytical sequence" prior to sample analysis by analyzing a calibration standard (CCV) at/near the mid-point of the calibration range (typically 10 ppbv). See the Quality Control section for the acceptance criteria. The 24 hour analytical sequence begins with the injection of BFB, continues through the analysis of the CCV, environmental samples and QC samples.
- 12.10.2 Set up a sequence in the Sample Table Log under *Sequence* of the main menu. Enter the sequence as it is to be run, including the applicable analysis method.
- 12.10.3 Click on *Position and Run*.
- 12.10.4 Generate the corresponding BFB "Tune" Report, Evaluate Continuing Calibration Report and Quantitation Reports
 - 12.10.4.1 On the BFB Report, evaluate the Pass/Fail column. An acceptable tune will pass each of the criteria. Analysis can not continue with a failed tune.
 - 12.10.4.2 On the Evaluate Continuing Calibration Report, %Deviation (or %Drift, as appropriate) for each analyte must be < 30%.

- 12.10.5 Analysis of environmental samples cannot proceed without an acceptable continuing verification.
- 12.10.6 After each sample has run, evaluate the chromatogram and quantitate against the current initial linearity (ICAL).

12.11 Sample Analysis

- 12.11.1 Analyze samples using the same instrument conditions and analytical steps used with the calibration standards.
- 12.11.2 Samples yielding an on-column concentration above that of the highest calibration standard must be diluted and reanalyzed as there is less certainty in these data. Smaller volumes of sample may be used for samples requiring a 25x dilution or less. The steps that follow result in a 60-fold dilution. Smaller volumes (e.g. 100 mL for a 300X dilution) can be analyzed if larger dilutions are needed. If necessary, the sample can be serially diluted into a third SUMMA canister.
 - 12.11.2.1 Obtain a cleaned, verified and evacuated canister in addition to the sample canister.
 - 12.11.2.2 Attach the cleaned canister to the outlet of the MFC and attach the sample canister to the inlet of the MFC.
 - 12.11.2.3 Set the MFC to 100 mL/min.
 - 12.11.2.4 Open both canister valves simultaneously and allow the sample to evacuate the original canister for 2.0 minutes. Use a timer. After 2 minutes, shut both valves.
 - 12.11.2.5 Detach the original sample and dilution canister canister from the MFC.
 - 12.11.2.6 Using the 4600a's dynamic static feature. Select the "Flush" button on the software screen and then choose "Dilute to final pressure" enter 2x the current pressure in psi, this will result in a canister pressure of 2 atm. Select "GO" in the software and press "flush" on the diluter.
 - 12.11.2.7 After flushing the fill tubing on the diluter a couple of times, connect the canister and press "pressurize" on the diluter. When pressurization is complete the software will show the actual pressure in the canister. The canister will now be filled with 2 atm of gas or twice the volume of the container, 1200 mL, This will result in a 60x dilution.
- 12.11.3 **Diluting into a Flex Film Bag or equivalent.**
 - 12.11.3.1 Obtain an unused bag and attach to the outlet of the mass flow controller.
 - 12.11.3.2 Attach a zero air source to the inlet of the 100 mL/min MFC.
 - 12.11.3.3 Fill the bag to the desired amount with zero air.
 - 12.11.4 Using a gas tight syringe, draw the desired amount from the sample can.

- 12.11.5 Inject the sample from the syringe into the filled bag via the septum port. The diluted sample is now ready for analysis.

13.0 **CALCULATIONS AND DATA HANDLING**

- 13.1 The HP software will print out all target analytes detected at a concentration at or above the MDL. The analyst must verify every target detected by evaluating the retention time and fit against the applicable CCV standard. These evaluations are accomplished by evaluating the characteristic ions through QEDIT as well as comparing the reference spectra. The Qvalue should be considered, however, the individual spectra must be evaluated in order to confidently identify a given analyte.

- 13.1.1 The data system software evaluates the retention time of each peak as well as a comparison of the characteristic ions to identify the compounds present. Only the primary ion is used for quantitation unless matrix interference is present. A list of the characteristic ions is in section 18. Use of the secondary ion must be documented in the raw data.
- 13.1.2 Minimum levels for each analyte are established in the software at a concentration equivalent to the current on-column MDL value.
- 13.1.3 Manual peak integration's as well as the addition/deletion of analytes must be documented on the Quantitation Report. See the Manual Integration of Chromatographic Peaks SOP for details.

- 13.2 Response factors (RF) are calculated as follows:

$$RF = (A_x)(C_{IS}) / (A_{IS})(C_x)$$

Where: A_x = Area of characteristic ion for compound being measured
 A_{IS} = Area of characteristic ion for compound being measured
 C_{IS} = Concentration of the specific internal standard
 C_x = Concentration of the compound being measured

- 13.3 The software calculates the sample concentration as follows:

INTERNAL STANDARD CALIBRATION

$$\text{Conc.} = \frac{(A_x)(I_s)(V_t)(DF)}{(A_{IS})(RF)(V_o)(V_i)}$$

Where: A_x = Area of characteristic ion for compound being measured
 I_s = Amount of internal standard injected (ng)
 V_t = Volume of total extract, taking into account dilution
 A_{IS} = Area of characteristic ion for the internal standard
RF = Initial average response factor for compound being measured
 V_o = Volume of water extracted (L), or mass of soil extracted (kg)
 V_i = Volume of extract injected (ul)
DF = dilution factor

- 13.4 After review, enter final results into the LIMS system. Results flagged by the LIMS with an "E" qualifier are above the linear range of the instrument. There is less certainty in these data and, if sufficient sample and holding time are available, should be reanalyzed at an appropriate dilution. Details on the procedure for entering analytical data are in the Analytical Data Entry – Organics Section SOP.

14.0 METHOD PERFORMANCE

- 14.1 Initial Demonstration of Capability study data, Method Detection Limit study data and Performance Testing study data are maintained and available from the QA office.

15.0 INSTRUMENT MAINTENANCE

- 15.1 The TO-15 instruments do not require routine maintenance.
- 15.2 Non-routine Maintenance: Any maintenance that is non-routine must be recorded in the non-routine maintenance log book.
- 15.3 For service call Entech at 805-527-5939 if there is a problem with the concentrator, can cleaner, diluter, or autosampler.
- 15.4 For service call Agilent at 1-800-227-9770 if there is a problem with the GC-MS. For service calls, the service technician must provide a breakdown of service. A summary of this breakdown must be documented in the Non-Routine Maintenance log book. A copy of this breakdown must be maintained by the QA Department.

16.0 TROUBLESHOOTING

- 16.1 For troubleshooting techniques consult the Entech Manual or Agilent Manual.
- 16.2 For additional troubleshooting techniques consult the Agilent website or call Entech.

17.0 POLLUTION PREVENTION

- 17.1 The quantity of chemicals purchased should be based on expected usage during their shelf life and the disposal cost of unused material.
- 17.2 Prepare the minimum amount of reagent and standard necessary.

18.0 WASTE MANAGEMENT

- 18.1 Refer to the Sample Disposal SOP for guidance on the disposal of any resulting residue, digestate, distillate, extract or standard.

19.0 REFERENCES

- 19.1 USEPA Compendium Method TO-15, revision January 1999. Compendium of Methods for the Determination of Toxic Organic Compounds in Ambient Air, second edition (EPA/625/R-96/010b).
- 19.2 Entech 3100A Operators Manual, Version 1.0: Smart Lab Based Canister Cleaning System
- 19.3 Entech 7100A Operators Manual, Version 1.0: Entech Instruments 7100A Preconcentrator
- 19.4 HP 5890 GC operators manual
- 19.5 HP 5972 MSD operators manual
- 19.6 Microbac Laboratories Quality Assurance Plan, current revision, all sections
- 19.7 Entech 4600A Dynamic Dilution System Operators Manual Version 1.0
- 19.8 Microbac Laboratories, Inc.-Chicagoland Controlled Document CD 0117- Flowmeter Calibration Data
- 19.9 Microbac Laboratories, Inc.-Chicagoland Controlled Document CD 0118- Setting the Flow of Veriflow Controllers.
- 19.10 Microbac Laboratories, Inc.-Chicagoland Controlled Document CD 0122(0)- TO-15 Marginal Exceedance Calculator.

20.0 TABLES, FORMS, CHECKLISTS, AND OTHER ATTACHMENTS

- 20.1 Analyte Table
- 20.2 Instrument Conditions and Method Settings table
- 20.3 TO-15 Canister Cleaning and Tracking Log
- 20.4 TO-15 Canister Order Log
- 20.5 TO-15 Flow Meter Table
- 20.6 TO-15 Canister Labels
- 20.7 TO-15 Run Log
- 20.8 TO-15 ICAL Review Check list
- 20.9 TO-15 Prep Logsheet
- 20.10 SOP Revision Notification Form listing changes in the revision

SECTION 20.1: ANALYTE TABLE

| AT | &Analyte | CAS# | Alternate ID |
|----|-------------------------------|-----------|-----------------------------|
| A | 1,1,1-Trichloroethane | 71-55-6 | Methyl chloroform |
| A | 1,1,2,2-Tetrachloroethane | 79-34-5 | 1,1,2,2-Tetrachloroethane |
| A | 1,1,2-Trichloro-1,2,2- | 76-13-1 | Freon-113 |
| A | 1,1,2-Trichloroethane | 79-00-5 | 1,1,2-Trichloroethane |
| A | 1,1-Dichloroethane | 75-34-3 | Ethylidene dichloride |
| A | 1,1-Dichloroethene | 75-35-4 | Vinylidene chloride;1,1- |
| A | 1,2,4-Trichlorobenzene | 120-82-1 | 1,2,4-Trichlorobenzene |
| A | 1,2,4-Trimethylbenzene | 95-63-6 | |
| A | 1,2-Dibromoethane | 106-93-4 | Ethylene dibromide |
| A | 1,2-Dichlorobenzene | 95-50-1 | |
| A | 1,2-Dichloroethane | 107-06-2 | |
| A | 1,2-Dichloropropane | 78-87-5 | |
| A | 1,2-Dichlorotetrafluoroethane | 76-14-2 | Freon-114 |
| A | 1,3,5-Trimethylbenzene | 108-67-8 | |
| A | 1,3-Butadiene | 106-99-0 | |
| A | 1,3-Dichlorobenzene | 541-73-1 | |
| A | 1,4-Dichlorobenzene | 106-46-7 | |
| A | 1,4-Dioxane | 123-91-1 | 1,4 Diethylene oxide |
| A | 2-Butanone | 78-93-3 | Methyl ethyl ketone;MEK |
| A | 2-Hexanone | 591-78-6 | |
| A | 2-Propanol | 67-63-0 | |
| A | 4-Ethyltoluene | 622-96-8 | |
| A | 4-Methyl-2-Pentanone | 108-10-1 | Methyl isobutyl ketone;MIBK |
| A | Acetone | 67-64-1 | |
| A | Acrolein | 107-02-8 | |
| A | Benzene | 71-43-2 | |
| A | Benzyl chloride | 100-44-7 | alpha-Chlorotoluene |
| A | Bromodichloromethane | 75-27-4 | |
| A | Bromoform | 75-25-2 | Tribromomethane |
| A | Bromomethane | 74-83-9 | Methyl bromide |
| A | Carbon disulfide | 75-15-0 | |
| A | Carbon tetrachloride | 56-23-5 | |
| A | Chlorobenzene | 108-90-7 | |
| A | Chloroethane | 75-00-3 | Ethyl chloride |
| A | Chloroform | 67-66-3 | |
| A | Chloromethane | 74-87-3 | Methyl chloride |
| A | cis-1,2-Dichloroethene | 156-59-2 | |
| A | cis-1,3-Dichloropropene | 10061-01- | |
| A | Cyclohexane | 110-82-7 | |
| A | Dibromochloromethane | 124-48-1 | Chlorodibromomethane |
| A | Dichlorodifluoromethane | 75-71-8 | Freon-12 |
| A | Ethyl acetate | 141-78-6 | |
| A | Ethyl benzene | 100-41-4 | Ethylbenzene |
| A | Heptane | 142-82-5 | |
| A | Hexachlorobutadiene | 87-68-3 | Hexachloro-1,3-butadiene |
| A | Hexane | 110-54-3 | |
| A | m,p-Xylene | 1330-20-7 | |
| A | Methyl Methacrylate | 80-62-6 | |
| A | Methyl-t-butyl ether | 1634-04-4 | MTBE |
| A | Methylene chloride | 75-09-2 | Dichloromethane |
| A | o-Xylene | 95-47-6 | |
| A | Propylene | 115-07-1 | |
| A | Styrene | 100-42-5 | |
| A | Tetrachloroethene | 127-18-4 | Tetrachloroethylene |

| | | | |
|---|---------------------------|-----------|--------------------|
| A | Tetrahydrofuran | 109-99-9 | |
| A | Toluene | 108-88-3 | |
| A | trans-1,2-Dichloroethene | 156-60-5 | |
| A | trans-1,3-Dichloropropene | 10061-02- | |
| A | Trichloroethene | 79-01-6 | Trichloroethylene |
| A | Trichlorofluoromethane | 75-69-4 | Freon-11 |
| A | Vinyl acetate | 108-05-4 | |
| A | Vinyl chloride | 75-01-4 | Chloroethene |
| B | 1,1,1,2-Tetrachloroethane | 630-20-6 | |
| B | 1,2,3-Trichloropropane | 96-18-4 | |
| B | 3-Chloro-1-propene | 107-05-1 | Allyl Chloride |
| B | Acetonitrile | 75-05-8 | |
| B | Acrylonitrile | 107-13-1 | |
| B | Bromobenzene | 108-86-1 | |
| B | Chlorodifluoromethane | 75-45-6 | Freon22 |
| B | Dibromomethane | 74-95-3 | |
| B | Dichlorofluoromethane | 75-43-4 | |
| B | Ethanol | 64-17-5 | |
| B | Ethyl acrylate | 140-88-5 | |
| B | Ethyl methacrylate | 97-63-2 | |
| B | Hexachloroethane | 67-72-1 | Perchloroethane |
| B | Isooctane | | |
| B | Methyl acrylate | 96-33-3 | |
| B | Methyl iodide | | |
| B | Methyl styrene (alpha) | | Methylstyrene |
| B | n-Octane | | Octane |
| B | Perchloroethane | 67-72-1 | Hexachloroethane |
| B | t-Butanol | | Tertiary Butanol |
| I | 1,4-Difluorobenzene | 540-36-3 | |
| I | Bromochloromethane | 74-97-5 | |
| I | Chlorobenzene-d5 | 3114-55-4 | D5-Chlorobenzene |
| M | Total VOCs | | |
| M | Total Xylenes | 1330-20- | total o,p,m-xylene |
| S | 4-Bromofluorobenzene | 460-00-4 | |
| T | Cumene | 98-82-8 | Isopropylbenzene |
| T | Isopropylbenzene | 98-82-8 | Cumene |
| T | Naphthalene | 91-20-3 | |

SECTION 20.2: INSTRUMENT CONDITIONS AND METHOD SETTINGS (SCAN_MODE)

----- TOPLEVEL PARAMETERS

TO-15 1

Method Information For: C:\HPCHEM\2\METHODS\ACQ.M

Method Sections To Run:

- () Save Copy of Method With Data
- () Pre-Run Cmd/Macro =
- (X) Data Acquisition
- () Data Analysis
- () Post-Run Cmd/Macro =

Method Comments:

8260 AQUISITION METHOD

END OF TOPLEVEL PARAMETERS

----- INSTRUMENT CONTROL PARAMETERS

Sample Inlet: GC
Injection Source: Manual
Injection Location: Rear
Mass Spectrometer: Enabled

HP5890 Temperature Parameters

| Zone Temperatures: | State | Setpoint |
|--------------------|-------|----------|
| Inlet A: | Off | 50 C |
| Inlet B: | On | 240 C |
| Detector A: | Off | 50 C |
| Detector B: | On | 250 C |
| Auxiliary: | Off | 50 C |

Oven Parameters:

| | |
|------------------|--------------|
| Oven Equib Time: | 0.00 minutes |
| Oven Max: | 275 C |
| Oven State: | On |
| Cryo State: | Off |
| Cryo Blast: | Off |
| Ambient: | 25 C |

Oven Program:

| | |
|----------------------|--------------|
| Initial Temperature: | 36 C |
| Initial Time: | 3.00 minutes |

| Level | Rate (C/minute) | Final Temperature (C) | Final Time (minutes) |
|----------------|--------------------|--------------------------|-------------------------|
| 1 | 8.0 | 130 | 0.00 |
| 2 (A) | 15.0 | 180 | 0.00 |
| 3 (B) | 25.0 | 220 | 0.80 |
| Next Run Time: | | 20.48 minutes | |

HP5890 Inlet Pressure Programs

GC Pressure Units: psi

Inlet A:

| | |
|----------------------------|---------|
| Constant Flow: | Off |
| Constant Flow Pressure: | 0.0 psi |
| Constant Flow Temperature: | 50 C |

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Initial Pressure: 0.0 psi
Initial Time: 650.00 minutes TO-15 1

| Level | Rate (psi/minute) | Final Pressure (psi) | Final Time (minutes) |
|-------|-------------------|----------------------|----------------------|
| 1 | 0.00 | 0.0 | 0.00 |
| 2 (A) | 0.00 | 0.0 | 0.00 |
| 3 (B) | 0.00 | 0.0 | 0.00 |

Total Program Time: 650.00 minutes

Column Length: 30.00 m
Column Diameter: 0.530 mm
Gas: He
Vacuum Compensation: Off

Inlet B:

Constant Flow: On
Constant Flow Pressure: 3.5 psi
Constant Flow Temperature: 35 C
Initial Pressure: 3.0 psi
Initial Time: 20.00 minutes

| Level | Rate (psi/minute) | Final Pressure (psi) | Final Time (minutes) |
|-------|-------------------|----------------------|----------------------|
| 1 | 0.00 | 0.0 | 0.00 |
| 2 (A) | 0.00 | 0.0 | 0.00 |
| 3 (B) | 0.00 | 0.0 | 0.00 |

Total Program Time: 20.00 minutes

Column Length: 30.00 m
Column Diameter: 0.250 mm
Gas: He
Vacuum Compensation: On

HP5890 Packed Column Flow Control

Inlet A not used to control packed column flow.

Inlet B not used to control packed column flow.

HP5890 Purge Valve Settings

| Inlet Purge | Init Value | On Time | Off Time | Splitless Injection |
|-------------|------------|---------|----------|---------------------|
| A | On | 0.00 | 0.00 | No |
| B | On | 0.00 | 0.00 | No |

HP5890 Valve and Relay Information

Initial Setpoints:
5890 Valves:
Valve 1: Off Valve 2: Off Valve 3: On Valve 4: On
19405 Valves:
Valve 5: Off Valve 6: Off Valve 7: Off Valve 8: Off
19405 Relays:
Relay 1: Off Relay 2: Off Relay 3: Off Relay 4: Off

HP5890 Detector Information

| Detector | Type | State |
|----------|------|-------|
| A | --- | Off |
| B | --- | Off |

HP5890 Signal Information

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Not saving signal data.

| Signal | Source | Peak Width | Data Rate | Start Data | Stop Data |
|--------|----------|------------|-----------|------------|-----------|
| 1 | Testplot | 0.053 | 5.000 | 0.00 | 1.00 |
| 2 | Testplot | 0.053 | 5.000 | 0.00 | 1.00 |

MS ACQUISITION PARAMETERS

General Information

Tune File : atune.u
Acquisition Mode : Scan

MS Information

Solvent Delay : 1.35 min
EM Absolute : False
EM Offset : -376
Resulting EM Voltage : 1776.5

[Scan Parameters]

Low Mass : 35
High Mass : 300
Threshold : 150
Sample # : 3 A/D Samples 8
Plot 2 low mass : 50
Plot 2 high mass : 550

END OF MS ACQUISITION PARAMETERS

END OF INSTRUMENT CONTROL PARAMETERS

TO-15 1

R_100210.REP

----- Method Parameters

Method Name: C:\Smart\m5.CTD Software ver. 4.17
Report Date/time 2/10/2010 3:00:13 PM

Stream: Sample
Preflush (sec): 10
Trap (cc/min): 60
Volume (cc): 0

Stream: Internal Standard
Preflush (sec): 5
Trap (cc/min): 60
Volume (cc): 50

Stream: Analytical Standard
Preflush (sec): 5
Trap (cc/min): 60
Volume (cc): 250

Stream: Sweep/Purge
Preflush (sec): 5
Trap (cc/min): 60
Volume (cc): 50

Stream: M1 -> M2
Preflush (sec): 5
Trap (cc/min): 5
Volume (cc): 20

Module1:
Trap temp(C): -40 Preheat? No
Preheat temp(C): 10
Desorb temp(C): 10
Bake temp(C): 150
Bake time(Min): 10

Bulk1:
Trap temp(C): 30
Desorb temp(C): 30
Bake temp(C): 150

Module2:
Trap temp(C): -60 Preheat? No
Preheat temp(C): 50
Desorb temp(C): 180
Bake temp(C): 190
Desorb time(C): 3.5

Bulk2:
Trap temp(C): 30
Desorb temp(C): 180
Bake temp(C): 150

Module3:
Trap temp(C): -160 Focus? Yes
Inject temp(C): 150
Inject time(Min): 2.5
Bake temp(C): 100
Bake time(Min): 3

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TO-15 1

R_100210.REP

Bake on EventEx# : 3
Total Time (Min) : 20

Misc:

Sample Xfer temp(C): 80
GC Xfer temp(C): 100
MPOS Valve temp(C): 110
Wait for GC before injecting
Active GC: GC1
Pressure: 110
MPOS Valve temp(C): 110

----- STATUS REPORT -----

METHOD: C:\Smart\m5.CTD DATE: 2/10/2010 Time: 3:00:13 PM
SAMPLE: ical 10 ak13-02 Ver:4.17

| EventEx | 1 | 2 | 3 | 4 | 5 | 6 | 7 | 8 | 9 | 10 | 11 |
|--------------|------|------|------|------|------|------|------|------|------|-----|------|
| duration | 2 | 1 | 62 | 8 | 53 | 9 | 260 | 1 | 1 | 12 | 61 |
| VLV Position | | | | | | | | | | | |
| 2 Pos VLV | 0 | 0 | 0 | 0 | 0 | 0 | 0 | 0 | 0 | 0 | 0 |
| System | 2 | 2 | 2 | 3 | 3 | 4 | 4 | 4 | 4 | 2 | 2 |
| Autosample1 | 1 | 1 | 1 | 1 | 1 | 1 | 1 | 1 | 1 | 1 | 1 |
| Autosample2 | 0 | 0 | 0 | 0 | 0 | 0 | 0 | 0 | 0 | 0 | 0 |
| Temperatures | | | | | | | | | | | |
| Module1 | 85 | 84 | -41 | -62 | -42 | -42 | -41 | -41 | -41 | -38 | -40 |
| Mod1 blkhd | 71 | 71 | 58 | 53 | 49 | 49 | 37 | 37 | 37 | 37 | 35 |
| Module2 | 125 | 125 | -49 | -72 | -66 | -65 | -60 | -60 | -60 | -58 | -53 |
| Mod2 blkhd | 83 | 83 | 65 | 58 | 51 | 50 | 34 | 33 | 33 | 33 | 33 |
| Module3 Max | 97 | 77 | 107 | 106 | 107 | 106 | 107 | 77 | 77 | 78 | 77 |
| Rotary vlvs | 110 | 110 | 110 | 109 | 108 | 109 | 109 | 109 | 109 | 110 | 110 |
| Samp Xfr ln | 79 | 80 | 79 | 80 | 80 | 80 | 80 | 80 | 80 | 80 | 80 |
| GC Xfr ln | 99 | 99 | 99 | 99 | 100 | 100 | 99 | 99 | 99 | 99 | 99 |
| Sample | 0 | 0 | 0 | 0 | 0 | 0 | 0 | 0 | 0 | 0 | 0 |
| Flows | | | | | | | | | | | |
| System | 0 | 0 | 0 | 0 | 60 | 1 | 60 | 60 | 60 | 0 | 60 |
| Split | 0 | 0 | 0 | 0 | 0 | 0 | 0 | 0 | 0 | 0 | 0 |
| Aux/Ext. | 0 | 0 | 0 | 0 | 0 | 0 | 0 | 0 | 0 | 0 | 0 |
| Volume | 0 | 0 | 0 | 0 | 50 | 0 | 250 | 0 | 0 | 0 | 50 |
| Pressures | | | | | | | | | | | |
| System | 22.4 | 22.4 | 21.8 | 30.1 | 28.8 | 16.2 | 12.8 | 12.8 | 12.8 | 22 | 18.7 |
| Aux | 0 | 0 | 0 | 0 | 0 | 0 | 0 | 0 | 0 | 0 | 0 |

----- STATUS REPORT -----

METHOD: C:\Smart\m5.CTD DATE: 2/10/2010 Time: 3:00:13 PM
SAMPLE: ical 10 ak13-02 Software Ver:4.17

| EventEx | 12 | 13 | 14 | 15 | 16 | 17 | 18 | 19 | 20 | 21 |
|--------------|-----|-----|-----|-----|-----|-----|-----|-----|-----|-----|
| duration | 1 | 294 | 1 | 53 | 1 | 210 | 151 | 10 | 606 | 433 |
| VLV Position | | | | | | | | | | |
| 2 Pos VLV | 0 | 0 | 0 | 0 | 0 | 0 | 0 | 0 | 0 | 0 |
| System | 2 | 2 | 2 | 2 | 2 | 2 | 2 | 5 | 2 | 2 |
| Autosample1 | 1 | 1 | 1 | 1 | 1 | 1 | 1 | 1 | 1 | 1 |
| Autosample2 | 0 | 0 | 0 | 0 | 0 | 0 | 0 | 0 | 0 | 0 |
| Temperatures | | | | | | | | | | |
| Module1 | -40 | 14 | 14 | 13 | 13 | 14 | 14 | 16 | 149 | 81 |
| Mod1 blkhd | 35 | 32 | 32 | 32 | 32 | 32 | 32 | 32 | 147 | 64 |
| Module2 | -53 | -49 | -49 | -49 | -49 | 178 | 179 | 180 | 187 | 98 |

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TOPLEVEL PARAMETERS

TO-15 2

Method Information For: C:\HPCHEM\1\METHODS\ACQ.M

Method Sections To Run:

- () Save Copy of Method With Data
- () Pre-Run Cmd/Macro =
- (X) Data Acquisition
- (X) Data Analysis
- () Post-Run Cmd/Macro =

Method Comments:

END OF TOPLEVEL PARAMETERS

INSTRUMENT CONTROL PARAMETERS

Sample Inlet: GC
Injection Source: Manual
Injection Location: Rear
Mass Spectrometer: Enabled

HP5890 Temperature Parameters

| Zone | Temperatures: | State | Setpoint |
|-------------|---------------|-------|----------|
| Inlet A: | Off | 50 C | |
| Inlet B: | On | 200 C | |
| Detector A: | Off | 50 C | |
| Detector B: | On | 250 C | |
| Auxiliary: | Off | 50 C | |

Oven Parameters:

| | |
|------------------|--------------|
| Oven Equib Time: | 0.00 minutes |
| Oven Max: | 300 C |
| Oven State: | On |
| Cryo State: | Off |
| Cryo Blast: | Off |
| Ambient: | 25 C |

Oven Program:

| | |
|----------------------|--------------|
| Initial Temperature: | 36 C |
| Initial Time: | 4.00 minutes |

| Level | Rate (C/minute) | Final Temperature (C) | Final Time (minutes) |
|-------|--------------------|--------------------------|-------------------------|
| 1 | 9.0 | 175 | 0.00 |
| 2 (A) | 40.0 | 220 | 1.60 |
| 3 (B) | 0.0 | 0 | 0.00 |

Next Run Time: 22.17 minutes

HP5890 Inlet Pressure Programs

GC Pressure Units: psi

Inlet A:

| | |
|----------------------------|---------|
| Constant Flow: | Off |
| Constant Flow Pressure: | 0.0 psi |
| Constant Flow Temperature: | 50 C |
| Initial Pressure: | 0.0 psi |

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Initial Time: 650.00 minutes

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| Level | Rate (psi/minute) | Final Pressure (psi) | Final Time (minutes) |
|---------------------|-------------------|----------------------|----------------------|
| 1 | 0.00 | 0.0 | 0.00 |
| 2(A) | 0.00 | 0.0 | 0.00 |
| 3(B) | 0.00 | 0.0 | 0.00 |
| Total Program Time: | | 650.00 minutes | |

Column Length: 30.00 m
Column Diameter: 0.530 mm
Gas: He
Vacuum Compensation: Off

Inlet B:

Constant Flow: On
Constant Flow Pressure: 3.9 psi
Constant Flow Temperature: 35 C
Initial Pressure: 5.3 psi
Initial Time: 0.01 minutes

| Level | Rate (psi/minute) | Final Pressure (psi) | Final Time (minutes) |
|---------------------|-------------------|----------------------|----------------------|
| 1 | 99.00 | 7.0 | 0.20 |
| 2(A) | 99.00 | 5.3 | 0.30 |
| 3(B) | 0.00 | 0.0 | 0.00 |
| Total Program Time: | | 0.54 minutes | |

Column Length: 60.00 m
Column Diameter: 0.320 mm
Gas: He
Vacuum Compensation: On

HP5890 Packed Column Flow Control

Inlet A not used to control packed column flow.

Inlet B not used to control packed column flow.

HP5890 Purge Valve Settings

| Inlet Purge | Init Value | On Time | Off Time | Splitless Injection |
|-------------|------------|---------|----------|---------------------|
| A | On | 0.00 | 0.00 | No |
| B | On | 0.00 | 0.00 | No |

HP5890 Valve and Relay Information

Initial Setpoints:

5890 Valves:
Valve 1: Off Valve 2: Off Valve 3: On Valve 4: On
19405 Valves:
Valve 5: Off Valve 6: Off Valve 7: Off Valve 8: Off
19405 Relays:
Relay 1: Off Relay 2: Off Relay 3: Off Relay 4: Off

HP5890 Detector Information

| Detector | Type | State |
|----------|------|-------|
| A | --- | Off |
| B | --- | Off |

HP5890 Signal Information

Method: ACQ.M

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Not saving signal data.

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| Signal | Source | Peak Width | Data Rate | Start Data | Stop Data |
|--------|----------|------------|-----------|------------|-----------|
| 1 | Testplot | 0.053 | 5.000 | 0.00 | 1.00 |
| 2 | Testplot | 0.053 | 5.000 | 0.00 | 1.00 |

MS ACQUISITION PARAMETERS

General Information

Tune File : bfb.u
Acquisition Mode : Scan

MS Information

Solvent Delay : 4.00 min
EM Absolute : False
EM Offset : -118
Resulting EM Voltage : 1776.5

[Scan Parameters]

Low Mass : 35
High Mass : 300
Threshold : 100
Sample # : 2 A/D Samples 4
Plot 2 low mass : 50
Plot 2 high mass : 550

END OF MS ACQUISITION PARAMETERS

END OF INSTRUMENT CONTROL PARAMETERS

TO-15 2

R_100210.REP

----- Method Parameters

Method Name: C:\Smart\m1.CTD Software ver. 3.19
Report Date/time 2/10/2010 5:29:36 PM

Stream: Sample
Preflush (sec): 10
Trap (cc/min): 60
Volume (cc): 0

Stream: Internal Standard
Preflush (sec): 5
Trap (cc/min): 60
Volume (cc): 50

Stream: Analytical Standard
Preflush (sec): 5
Trap (cc/min): 60
Volume (cc): 250

Stream: Sweep/Purge
Preflush (sec): 5
Trap (cc/min): 60
Volume (cc): 50

Stream: M1 -> M2
Preflush (sec):
Trap (cc/min): 5
Volume (cc): 20

Module1:
Trap temp(C): -40 Preheat? No
Preheat temp(C): 10
Desorb temp(C): 10
Bake temp(C): 150
Bake time(Min): 10

Bulk1:
Trap temp(C): 30
Desorb temp(C): 30
Bake temp(C): 150

Module2:
Trap temp(C): -60 Preheat? No
Preheat temp(C): 50
Desorb temp(C): 180
Bake temp(C): 190
Desorb time(C): 4.5

Bulk2:
Trap temp(C): 30
Desorb temp(C): 100
Bake temp(C): 150

Module3:
Trap temp(C): -160 Focus? Yes
Inject temp(C): 150
Inject time(Min): 3.5
Bake temp(C): 100
Bake time(Min): 3

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TO-15 2

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Bake on EventEx# : 3
Total Time (Min) : 22

Misc:

Sample xfer temp(C): 80
GC xfer temp(C): 110
MPOS Valve temp(C): 110
Wait for GC before injecting
Active GC: GC1
Pressure: 110
MPOS Valve temp(C): 110

----- STATUS REPORT -----

METHOD: C:\Smart\m1.CTD DATE: 2/10/2010 Time: 5:29:36 PM
SAMPLE: ccv/tun 10 ak13-05 Ver.-3.19

| EventEx | 1 | 2 | 3 | 4 | 5 | 6 | 7 | 8 | 9 | 10 | 11 |
|--------------|------|------|------|------|------|------|-----|-----|-----|------|-----|
| duration | 27 | 1 | 79 | 8 | 60 | 9 | 263 | 1 | 1 | 12 | 61 |
| VLV Position | | | | | | | | | | | |
| 2 Pos VLV | 0 | 0 | 0 | 0 | 0 | 0 | 0 | 0 | 0 | 0 | 0 |
| System | 2 | 2 | 2 | 3 | 3 | 4 | 4 | 4 | 4 | 2 | 2 |
| Autosample1 | 5 | 5 | 5 | 5 | 5 | 5 | 5 | 5 | 5 | 5 | 5 |
| Autosample2 | 0 | 0 | 0 | 0 | 0 | 0 | 0 | 0 | 0 | 0 | 0 |
| Temperatures | | | | | | | | | | | |
| Module1 | 37 | 37 | -38 | -51 | -47 | -47 | -40 | -40 | -40 | -38 | -38 |
| Mod1 blkhd | 41 | 41 | 41 | 40 | 39 | 39 | 34 | 34 | 34 | 33 | 33 |
| Module2 | 41 | 41 | -45 | -73 | -68 | -67 | -60 | -60 | -60 | -60 | -55 |
| Mod2 blkhd | 44 | 44 | 44 | 44 | 42 | 42 | 35 | 35 | 35 | 35 | 33 |
| Module3 Max | 44 | 45 | 104 | 105 | 106 | 104 | 106 | 60 | 59 | 59 | 59 |
| Rotary Vlv | 121 | 123 | 127 | 131 | 127 | 127 | 128 | 128 | 128 | 131 | 128 |
| Samp xfr ln | 79 | 78 | 82 | 79 | 82 | 81 | 80 | 80 | 80 | 80 | 80 |
| GC xfr ln | 120 | 122 | 123 | 118 | 115 | 117 | 125 | 125 | 125 | 120 | 115 |
| Sample | 0 | 0 | 0 | 0 | 0 | 0 | 0 | 0 | 0 | 0 | 0 |
| Flows | | | | | | | | | | | |
| System | 0 | 0 | 0 | 0 | 60 | 0 | 60 | 0 | 60 | 0 | 60 |
| Split | 0 | 0 | 0 | 0 | 0 | 0 | 0 | 0 | 0 | 0 | 0 |
| Aux/Ext. | 0 | 0 | 0 | 0 | 0 | 0 | 0 | 0 | 0 | 0 | 0 |
| Volume | 0 | 0 | 0 | 0 | 50 | 0 | 250 | 0 | 0 | 0 | 51 |
| Pressures | | | | | | | | | | | |
| System | 26.4 | 26.4 | 26.2 | 22.2 | 21.3 | 10.6 | 5.7 | 5.7 | 5.7 | 22.3 | 23 |
| Aux | 0 | 0 | 0 | 0 | 0 | 0 | 0 | 0 | 0 | 0 | 0 |

----- STATUS REPORT -----

METHOD: C:\Smart\m1.CTD DATE: 2/10/2010 Time: 5:29:36 PM
SAMPLE: ccv/tun 10 ak13-05 Software Ver. 3.19

| EventEx | 12 | 13 | 14 | 15 | 16 | 17 | 18 | 19 | 20 | 21 |
|--------------|-----|-----|-----|-----|-----|-----|-----|-----|-----|-----|
| duration | 1 | 285 | 1 | 47 | 1 | 270 | 210 | 1 | 600 | 509 |
| VLV Position | | | | | | | | | | |
| 2 Pos VLV | 0 | 0 | 0 | 0 | 0 | 0 | 0 | 0 | 0 | 0 |
| System | 2 | 2 | 2 | 2 | 2 | 2 | 2 | 2 | 2 | 2 |
| Autosample1 | 5 | 5 | 5 | 5 | 5 | 5 | 5 | 5 | 5 | 5 |
| Autosample2 | 0 | 0 | 0 | 0 | 0 | 0 | 0 | 0 | 0 | 0 |
| Temperatures | | | | | | | | | | |
| Module1 | -38 | 16 | 16 | 15 | 15 | 15 | 15 | 15 | 147 | 76 |
| Mod1 blkhd | 33 | 32 | 32 | 32 | 31 | 31 | 31 | 31 | 146 | 61 |
| Module2 | -55 | -49 | -49 | -47 | -47 | 167 | 178 | 175 | 191 | 80 |

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Instrument Conditions and Method Settings (SIM_Mode)

TOPLEVEL PARAMETERS

Method Information For: C:\HPCHEM\2\METHODS\SIM.M

Method Sections To Run:

() Save Copy of Method With Data
() Pre-Run Cmd/Macro =
(X) Data Acquisition
() Data Analysis
() Post-Run Cmd/Macro =

Method Comments:

8260 AQUISITION METHOD

END OF TOPLEVEL PARAMETERS

INSTRUMENT CONTROL PARAMETERS

Sample Inlet: GC
Injection Source: Manual
Injection Location: Rear
Mass Spectrometer: Enabled

HP5890 Temperature Parameters

| Zone Temperatures: | State | Setpoint |
|--------------------|-------|----------|
| Inlet A: | Off | 50 C |
| Inlet B: | On | 200 C |
| Detector A: | Off | 50 C |
| Detector B: | On | 250 C |
| Auxiliary: | Off | 50 C |

Oven Parameters:

| | |
|------------------|--------------|
| Oven Equib Time: | 0.00 minutes |
| Oven Max: | 300 C |
| Oven State: | On |
| Cryo State: | Off |
| Cryo Blast: | Off |
| Ambient: | 25 C |

Oven Program:

| | |
|----------------------|--------------|
| Initial Temperature: | 40 C |
| Initial Time: | 4.00 minutes |

| Level | Rate (C/minute) | Final Temperature (C) | Final Time (minutes) |
|----------------|-----------------|-----------------------|----------------------|
| 1 | 10.0 | 150 | 0.00 |
| 2 (A) | 40.0 | 220 | 6.00 |
| 3 (B) | 0.0 | 0 | 0.00 |
| Next Run Time: | | 22.75 minutes | |

HP5890 Inlet Pressure Programs

GC Pressure Units: psi

Inlet A:

| | |
|----------------------------|---------|
| Constant Flow: | Off |
| Constant Flow Pressure: | 0.0 psi |
| Constant Flow Temperature: | 50 C |

Method: SIM.M

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Initial Pressure: 0.0 psi
Initial Time: 650.00 minutes

| Level | Rate (psi/minute) | Final Pressure (psi) | Final Time (minutes) |
|---------------------|-------------------|----------------------|----------------------|
| 1 | 0.00 | 0.0 | 0.00 |
| 2 (A) | 0.00 | 0.0 | 0.00 |
| 3 (B) | 0.00 | 0.0 | 0.00 |
| Total Program Time: | | 650.00 minutes | |

Column Length: 30.00 m
Column Diameter: 0.530 mm
Gas: He
Vacuum Compensation: Off

Inlet B:
Constant Flow: On
Constant Flow Pressure: 10.0 psi
Constant Flow Temperature: 40 C
Initial Pressure: 3.0 psi
Initial Time: 20.00 minutes

| Level | Rate (psi/minute) | Final Pressure (psi) | Final Time (minutes) |
|---------------------|-------------------|----------------------|----------------------|
| 1 | 0.00 | 0.0 | 0.00 |
| 2 (A) | 0.00 | 0.0 | 0.00 |
| 3 (B) | 0.00 | 0.0 | 0.00 |
| Total Program Time: | | 20.00 minutes | |

Column Length: 60.00 m
Column Diameter: 0.320 mm
Gas: He
Vacuum Compensation: On

HP5890 Packed Column Flow Control

Inlet A not used to control packed column flow.

Inlet B not used to control packed column flow.

HP5890 Purge Valve Settings

| Inlet Purge | Init Value | On Time | Off Time | Splitless Injection |
|-------------|------------|---------|----------|---------------------|
| A | On | 0.00 | 0.00 | No |
| B | On | 0.00 | 0.00 | No |

HP5890 Valve and Relay Information

Initial Setpoints:

| | | | | |
|---------------|--------------|--------------|--------------|--------------|
| 5890 Valves: | Valve 1: Off | Valve 2: Off | Valve 3: On | Valve 4: On |
| 19405 Valves: | Valve 5: Off | Valve 6: Off | Valve 7: Off | Valve 8: Off |
| 19405 Relays: | Relay 1: Off | Relay 2: Off | Relay 3: Off | Relay 4: Off |

HP5890 Detector Information

| Detector | Type | State |
|----------|------|-------|
| A | --- | Off |
| B | --- | Off |

HP5890 Signal Information

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Not saving signal data.

| Signal | Source | Peak Width | Data Rate | Start Data | Stop Data |
|--------|----------|------------|-----------|------------|-----------|
| 1 | Testplot | 0.053 | 5.000 | 0.00 | 1.00 |
| 2 | Testplot | 0.053 | 5.000 | 0.00 | 1.00 |

MS ACQUISITION PARAMETERS

General Information

Tune File : bfb.u
Acquisition Mode : SIM

MS Information

Solvent Delay : 3.65 min
EM Absolute : False
EM Offset : -153
Resulting EM Voltage : 1858.8

[Sim Parameters]

GROUP 1
Group ID : 1
Resolution : Low
Group Start Time : 0.00
Plot 1 Ion : 49.0
Ions/Dwell In Group (Mass, Dwell) (Mass, Dwell) (Mass, Dwell)
(49.0, 100) (83.0, 100) (62.0, 100)
(96.0, 100) (84.0, 100) (63.0, 100)

GROUP 2
Group ID : 2
Resolution : Low
Group Start Time : 7.73
Plot 1 Ion : 83.0
Ions/Dwell In Group (Mass, Dwell) (Mass, Dwell) (Mass, Dwell)
(83.0, 100) (114.0, 100) (117.0, 100)
(62.0, 100) (63.0, 100) (97.0, 100)
(95.0, 100)

GROUP 3
Group ID : 3
Resolution : Low
Group Start Time : 10.60
Plot 1 Ion : 83.0
Ions/Dwell In Group (Mass, Dwell) (Mass, Dwell) (Mass, Dwell)
(83.0, 100) (117.0, 100) (107.0, 100)
(95.0, 100) (166.0, 100)

END OF MS ACQUISITION PARAMETERS

END OF INSTRUMENT CONTROL PARAMETERS

DATA ANALYSIS PARAMETERS

Method: SIM.M

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Method Name: C:\HPCHEM\1\TO-15\T2052410.M

Percent Report Settings

Sort By: Signal

Output Destination

Screen: No
Printer: Yes
File: No

Integration Events: Meth Default

Generate Report During Run Method: No

Signal Correlation Window: 0.020

Qualitative Report Settings

Peak Location of Unknown: Apex

Library to Search Minimum Quality
nist129k.L 0

Integration Events: Meth Default

Report Type: Summary

Output Destination

Screen: No
Printer: Yes
File: No

Generate Report During Run Method: No

Quantitative Report Settings

Report Type: Summary

Output Destination

Screen: Yes
Printer: No
File: No

Generate Report During Run Method: No

TO15-2 ICAL

Calibration Last Updated: Wed Jun 02 16:00:56 2010

Reference Window: 0.50 Minutes
Non-Reference Window: 0.50 Minutes
Correlation Window: 0.05 minutes
Default Multiplier: 1.00
Default Sample Concentration: 0.00

Compound Information

1) Bromochloromethane (ISTD TR)

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Ret. Time 7.25 min., Extract & Integrate from 7.00 to 7.50 min.

| Signal | Rel Resp. | Pct. Unc. (abs) | Integration |
|-----------|-----------|-----------------|----------------------|
| Tgt 49.00 | | | *** METH DEFAULT *** |
| Q1 128.00 | 44.60 | 20.0 | *** METH DEFAULT *** |
| Q2 130.00 | 59.20 | 20.0 | *** METH DEFAULT *** |

| Lvl ID | Conc (ppbv) | Response |
|--------|-------------|----------|
| .5 | 10.000 | 94906 |
| 1 | 10.000 | 95147 |
| 2 | 10.000 | 88645 |
| 10 | 10.000 | 85264 |
| 20 | 10.000 | 76608 |
| 40 | 10.000 | 69500 |

Qualifier Peak Analysis ON ISTD conc: 10.000 ppbv
Curve Fit: Avg. RF

2) Propylene

()

Ret. Time 3.85 min., Extract & Integrate from 3.60 to 4.10 min.

| Signal | Rel Resp. | Pct. Unc. (abs) | Integration |
|-----------|-----------|-----------------|----------------------|
| Tgt 42.00 | | | *** METH DEFAULT *** |
| Q1 39.00 | 132.20 | 20.0 | *** METH DEFAULT *** |
| Q2 41.00 | 152.70 | 20.0 | *** METH DEFAULT *** |

| Lvl ID | Conc (ppbv) | Response |
|--------|-------------|----------|
| .5 | 0.500 | 2248 |
| 1 | 1.000 | 4633 |
| 2 | 2.000 | 12400 |
| 10 | 10.000 | 51481 |
| 20 | 20.000 | 90430 |
| 40 | 40.000 | 147987 |

Qualifier Peak Analysis ON
Curve Fit: Avg. RF

3) Freon-12

()

Ret. Time 3.91 min., Extract & Integrate from 3.66 to 4.16 min.

| Signal | Rel Resp. | Pct. Unc. (abs) | Integration |
|-----------|-----------|-----------------|----------------------|
| Tgt 85.00 | | | *** METH DEFAULT *** |
| Q1 87.00 | 33.50 | 20.0 | *** METH DEFAULT *** |
| Q2 50.00 | 14.30 | 20.0 | *** METH DEFAULT *** |
| Q3 101.00 | 8.60 | 20.0 | *** METH DEFAULT *** |

| Lvl ID | Conc (ppbv) | Response |
|--------|-------------|----------|
| .5 | 0.500 | 23869 |
| 1 | 1.000 | 47299 |
| 2 | 2.000 | 117620 |
| 10 | 10.000 | 434296 |
| 20 | 20.000 | 735609 |
| 40 | 40.000 | 1144457 |

Qualifier Peak Analysis ON
Curve Fit: Avg. RF

4) Freon-114

()

Ret. Time 4.10 min., Extract & Integrate from 3.85 to 4.35 min.

| Signal | Rel Resp. | Pct. Unc. (abs) | Integration |
|-----------|-----------|-----------------|----------------------|
| Tgt 85.00 | | | *** METH DEFAULT *** |
| Q1 135.00 | 45.00 | 20.0 | *** METH DEFAULT *** |

| Lvl ID | Conc (ppbv) | Response |
|--------|-------------|----------|
| .5 | 0.500 | 21242 |

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| | | |
|----|--------|--------|
| 1 | 1.000 | 41568 |
| 2 | 2.000 | 103164 |
| 10 | 10.000 | 353595 |
| 20 | 20.000 | 584090 |
| 40 | 40.000 | 875587 |

Qualifier Peak Analysis ON
Curve Fit: Avg. RF

5) Chloromethane

()

Ret. Time 4.03 min., Extract & Integrate from 3.78 to 4.28 min.

| Signal | Rel Resp. | Pct. Unc. (abs) | Integration |
|-----------|-----------|-----------------|----------------------|
| Tgt 50.00 | | | *** METH DEFAULT *** |
| Q1 52.00 | 32.30 | 20.0 | *** METH DEFAULT *** |
| Q2 49.00 | 11.90 | 20.0 | *** METH DEFAULT *** |

| Lvl ID | Conc (ppbv) | Response |
|--------|-------------|----------|
| .5 | 0.500 | 5401 |
| 1 | 1.000 | 11171 |
| 2 | 2.000 | 28710 |
| 10 | 10.000 | 110961 |
| 20 | 20.000 | 203228 |
| 40 | 40.000 | 339195 |

Qualifier Peak Analysis ON
Curve Fit: Avg. RF

6) Vinyl chloride

()

Ret. Time 4.19 min., Extract & Integrate from 3.94 to 4.44 min.

| Signal | Rel Resp. | Pct. Unc. (abs) | Integration |
|-----------|-----------|-----------------|----------------------|
| Tgt 62.00 | | | *** METH DEFAULT *** |
| Q1 64.00 | 33.90 | 20.0 | *** METH DEFAULT *** |
| Q2 61.00 | 9.50 | 20.0 | *** METH DEFAULT *** |

| Lvl ID | Conc (ppbv) | Response |
|--------|-------------|----------|
| .5 | 0.500 | 5090 |
| 1 | 1.000 | 10485 |
| 2 | 2.000 | 26655 |
| 10 | 10.000 | 104993 |
| 20 | 20.000 | 192042 |
| 40 | 40.000 | 311034 |

Qualifier Peak Analysis ON
Curve Fit: Avg. RF

7) 1,3-Butadiene

()

Ret. Time 4.29 min., Extract & Integrate from 4.04 to 4.54 min.

| Signal | Rel Resp. | Pct. Unc. (abs) | Integration |
|-----------|-----------|-----------------|----------------------|
| Tgt 39.00 | | | *** METH DEFAULT *** |
| Q1 54.00 | 81.40 | 20.0 | *** METH DEFAULT *** |

| Lvl ID | Conc (ppbv) | Response |
|--------|-------------|----------|
| .5 | 0.500 | 4596 |
| 1 | 1.000 | 8995 |
| 2 | 2.000 | 22281 |
| 10 | 10.000 | 85039 |
| 20 | 20.000 | 152141 |
| 40 | 40.000 | 249136 |

Qualifier Peak Analysis ON
Curve Fit: Avg. RF

8) Bromomethane

()

Method: SIM.M

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Ret. Time 4.48 min., Extract & Integrate from 4.23 to 4.73 min.

| Signal | Rel Resp. | Pct. Unc. (abs) | Integration |
|-----------|-----------|-----------------|----------------------|
| Tgt 94.00 | | | *** METH DEFAULT *** |
| Q1 96.00 | 96.30 | 20.0 | *** METH DEFAULT *** |
| Q2 93.00 | 23.10 | 20.0 | *** METH DEFAULT *** |

| Lvl ID | Conc (ppbv) | Response |
|--------|-------------|----------|
| .5 | 0.500 | 5489 |
| 1 | 1.000 | 11615 |
| 2 | 2.000 | 28403 |
| 10 | 10.000 | 115529 |
| 20 | 20.000 | 209376 |
| 40 | 40.000 | 350746 |

Qualifier Peak Analysis ON
Curve Fit: Avg. RF

9) Chloroethane

()

Ret. Time 4.60 min., Extract & Integrate from 4.35 to 4.85 min.

| Signal | Rel Resp. | Pct. Unc. (abs) | Integration |
|-----------|-----------|-----------------|----------------------|
| Tgt 64.00 | | | *** METH DEFAULT *** |
| Q1 66.00 | 33.10 | 20.0 | *** METH DEFAULT *** |
| Q2 49.00 | 32.70 | 20.0 | *** METH DEFAULT *** |

| Lvl ID | Conc (ppbv) | Response |
|--------|-------------|----------|
| .5 | 0.500 | 2344 |
| 1 | 1.000 | 5334 |
| 2 | 2.000 | 13461 |
| 10 | 10.000 | 53214 |
| 20 | 20.000 | 100551 |
| 40 | 40.000 | 163077 |

Qualifier Peak Analysis ON
Curve Fit: Avg. RF

10) Ethanol

()

Ret. Time 4.65 min., Extract & Integrate from 4.40 to 4.90 min.

| Signal | Rel Resp. | Pct. Unc. (abs) | Integration |
|-----------|-----------|-----------------|----------------------|
| Tgt 45.00 | | | *** METH DEFAULT *** |
| Q1 46.00 | 37.70 | 20.0 | *** METH DEFAULT *** |

| Lvl ID | Conc (ppbv) | Response |
|--------|-------------|----------|
| .5 | 0.500 | 1418 |
| 1 | 1.000 | 2828 |
| 2 | 2.000 | 6583 |
| 10 | 10.000 | 28337 |
| 20 | 20.000 | 52486 |
| 40 | 40.000 | 90514 |

Qualifier Peak Analysis ON
Curve Fit: Avg. RF

11) Acrolein

()

Ret. Time 4.91 min., Extract & Integrate from 4.66 to 5.16 min.

| Signal | Rel Resp. | Pct. Unc. (rel) | Integration |
|-----------|-----------|-----------------|-------------|
| Tgt 56.00 | | | RTEINT.P |
| Q1 55.00 | 66.10 | 20.0 | RTEINT.P |

| Lvl ID | Conc (ppbv) | Response |
|--------|-------------|----------|
| .5 | 0.500 | 376 |
| 1 | 1.000 | 867 |
| 2 | 2.000 | 2187 |

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| | | |
|----|--------|-------|
| 10 | 10.000 | 9514 |
| 20 | 20.000 | 18689 |
| 40 | 40.000 | 34135 |

Qualifier Peak Analysis ON
Curve Fit: Avg. RF

12) Freon-11

()

Ret. Time 5.14 min., Extract & Integrate from 4.89 to 5.39 min.

| Signal | Rel Resp. | Pct. Unc. (abs) | Integration |
|------------|-----------|-----------------|----------------------|
| Tgt 101.00 | | | *** METH DEFAULT *** |
| Q1 103.00 | 65.90 | 20.0 | *** METH DEFAULT *** |
| Q2 105.00 | 10.70 | 20.0 | *** METH DEFAULT *** |

| Lvl ID | Conc (ppbv) | Response |
|--------|-------------|----------|
| .5 | 0.500 | 19308 |
| 1 | 1.000 | 38895 |
| 2 | 2.000 | 94010 |
| 10 | 10.000 | 347401 |
| 20 | 20.000 | 606049 |
| 40 | 40.000 | 989674 |

Qualifier Peak Analysis ON
Curve Fit: Avg. RF

13) 2-Propanol

()

Ret. Time 5.14 min., Extract & Integrate from 4.89 to 5.39 min.

| Signal | Rel Resp. | Pct. Unc. (rel) | Integration |
|-----------|-----------|-----------------|----------------------|
| Tgt 45.00 | | | *** METH DEFAULT *** |
| Q1 43.00 | 21.60 | 20.0 | *** METH DEFAULT *** |
| Q2 41.00 | 8.20 | 20.0 | *** METH DEFAULT *** |
| Q3 59.00 | 3.40 | 20.0 | *** METH DEFAULT *** |

| Lvl ID | Conc (ppbv) | Response |
|--------|-------------|----------|
| .5 | 0.500 | 9427 |
| 1 | 1.000 | 19241 |
| 2 | 2.000 | 45992 |
| 10 | 10.000 | 166116 |
| 20 | 20.000 | 295226 |
| 40 | 40.000 | 527642 |

Qualifier Peak Analysis ON
Curve Fit: Avg. RF

14) 1,1-Dichloroethene

()

Ret. Time 5.58 min., Extract & Integrate from 5.33 to 5.83 min.

| Signal | Rel Resp. | Pct. Unc. (abs) | Integration |
|-----------|-----------|-----------------|----------------------|
| Tgt 96.00 | | | *** METH DEFAULT *** |
| Q1 61.00 | 201.60 | 20.0 | *** METH DEFAULT *** |
| Q2 63.00 | 67.70 | 20.0 | *** METH DEFAULT *** |

| Lvl ID | Conc (ppbv) | Response |
|--------|-------------|----------|
| .5 | 0.500 | 3789 |
| 1 | 1.000 | 8366 |
| 2 | 2.000 | 21000 |
| 10 | 10.000 | 84471 |
| 20 | 20.000 | 156095 |
| 40 | 40.000 | 270266 |

Qualifier Peak Analysis ON
Curve Fit: Avg. RF

15) Methylene chloride

()

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Ret. Time 5.65 min., Extract & Integrate from 5.40 to 5.90 min.

| Signal | Rel Resp. | Pct. Unc. (abs) | Integration |
|-----------|-----------|-----------------|----------------------|
| Tgt 84.00 | | | *** METH DEFAULT *** |
| Q1 86.00 | 63.00 | 20.0 | *** METH DEFAULT *** |
| Q2 49.00 | 141.40 | 20.0 | *** METH DEFAULT *** |

| Lvl ID | Conc (ppbv) | Response |
|--------|-------------|----------|
| .5 | 0.500 | 4532 |
| 1 | 1.000 | 8868 |
| 2 | 2.000 | 20575 |
| 10 | 10.000 | 77435 |
| 20 | 20.000 | 139702 |
| 40 | 40.000 | 242201 |

Qualifier Peak Analysis ON
Curve Fit: Avg. RF

16) Freon-113

()

Ret. Time 5.84 min., Extract & Integrate from 5.59 to 6.09 min.

| Signal | Rel Resp. | Pct. Unc. (abs) | Integration |
|------------|-----------|-----------------|----------------------|
| Tgt 101.00 | | | *** METH DEFAULT *** |
| Q1 151.00 | 85.90 | 20.0 | *** METH DEFAULT *** |
| Q2 153.00 | 58.60 | 20.0 | *** METH DEFAULT *** |
| Q3 103.00 | 62.80 | 20.0 | *** METH DEFAULT *** |

| Lvl ID | Conc (ppbv) | Response |
|--------|-------------|----------|
| .5 | 0.500 | 9492 |
| 1 | 1.000 | 19546 |
| 2 | 2.000 | 47748 |
| 10 | 10.000 | 178924 |
| 20 | 20.000 | 314831 |
| 40 | 40.000 | 523302 |

Qualifier Peak Analysis ON
Curve Fit: Avg. RF

17) Carbon disulfide

()

Ret. Time 5.91 min., Extract & Integrate from 5.66 to 6.16 min.

| Signal | Rel Resp. | Pct. Unc. (abs) | Integration |
|-----------|-----------|-----------------|----------------------|
| Tgt 76.00 | | | *** METH DEFAULT *** |
| Q1 78.00 | 9.50 | 20.0 | *** METH DEFAULT *** |
| Q2 44.00 | 19.20 | 20.0 | *** METH DEFAULT *** |

| Lvl ID | Conc (ppbv) | Response |
|--------|-------------|----------|
| .5 | 0.500 | 36777 |
| 1 | 1.000 | 74278 |
| 2 | 2.000 | 59068 |
| 10 | 10.000 | 225175 |
| 20 | 20.000 | 407735 |
| 40 | 40.000 | 697108 |

Qualifier Peak Analysis ON
Curve Fit: Linear, forced through origin

18) Methyl-t-butyl ether

()

Ret. Time 6.51 min., Extract & Integrate from 6.26 to 6.76 min.

| Signal | Rel Resp. | Pct. Unc. (rel) | Integration |
|-----------|-----------|-----------------|----------------------|
| Tgt 73.00 | | | *** METH DEFAULT *** |
| Q1 41.00 | 19.80 | 20.0 | *** METH DEFAULT *** |
| Q2 43.00 | 24.70 | 20.0 | *** METH DEFAULT *** |
| Q3 57.00 | 18.70 | 20.0 | *** METH DEFAULT *** |

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| Lvl ID | Conc (ppbv) | Response |
|--------|-------------|----------|
| .5 | 0.500 | 11304 |
| 1 | 1.000 | 23938 |
| 2 | 2.000 | 58592 |
| 10 | 10.000 | 223071 |
| 20 | 20.000 | 395861 |
| 40 | 40.000 | 673621 |

Qualifier Peak Analysis ON
Curve Fit: Avg. RF

19) Acetone

()

Ret. Time 4.99 min., Extract & Integrate from 4.74 to 5.24 min.

| Signal | Rel Resp. | Pct. Unc. (rel) | Integration |
|-----------|-----------|-----------------|----------------------|
| Tgt 43.00 | | | *** METH DEFAULT *** |
| Q1 58.00 | 20.90 | 20.0 | *** METH DEFAULT *** |
| Q2 42.00 | 7.40 | 20.0 | *** METH DEFAULT *** |

| Lvl ID | Conc (ppbv) | Response |
|--------|-------------|----------|
| .5 | 0.500 | 14946 |
| 1 | 1.000 | 28339 |
| 2 | 2.000 | 54615 |
| 10 | 10.000 | 216319 |
| 20 | 20.000 | 388599 |
| 40 | 40.000 | 665327 |

Qualifier Peak Analysis ON
Curve Fit: Avg. RF

20) trans-1,2-Dichloroethene

()

Ret. Time 6.34 min., Extract & Integrate from 6.09 to 6.59 min.

| Signal | Rel Resp. | Pct. Unc. (abs) | Integration |
|-----------|-----------|-----------------|----------------------|
| Tgt 96.00 | | | *** METH DEFAULT *** |
| Q1 61.00 | 156.80 | 20.0 | *** METH DEFAULT *** |
| Q2 98.00 | 61.40 | 20.0 | *** METH DEFAULT *** |

| Lvl ID | Conc (ppbv) | Response |
|--------|-------------|----------|
| .5 | 0.500 | 4052 |
| 1 | 1.000 | 8528 |
| 2 | 2.000 | 21131 |
| 10 | 10.000 | 84658 |
| 20 | 20.000 | 154686 |
| 40 | 40.000 | 273598 |

Qualifier Peak Analysis ON
Curve Fit: Avg. RF

21) 1,1-Dichloroethane

()

Ret. Time 6.49 min., Extract & Integrate from 6.24 to 6.74 min.

| Signal | Rel Resp. | Pct. Unc. (abs) | Integration |
|-----------|-----------|-----------------|----------------------|
| Tgt 63.00 | | | *** METH DEFAULT *** |
| Q1 65.00 | 33.10 | 20.0 | *** METH DEFAULT *** |
| Q2 83.00 | 15.80 | 20.0 | *** METH DEFAULT *** |

| Lvl ID | Conc (ppbv) | Response |
|--------|-------------|----------|
| .5 | 0.500 | 7725 |
| 1 | 1.000 | 16252 |
| 2 | 2.000 | 38981 |
| 10 | 10.000 | 147775 |
| 20 | 20.000 | 264281 |
| 40 | 40.000 | 444009 |

Qualifier Peak Analysis ON
Curve Fit: Avg. RF

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22) Vinyl acetate ()
Ret. Time 6.56 min., Extract & Integrate from 6.31 to 6.81 min.

| Signal | Rel Resp. | Pct. Unc. (abs) | Integration |
|-----------|-----------|-----------------|----------------------|
| Tgt 43.00 | | | *** METH DEFAULT *** |
| Q1 86.00 | 7.00 | 20.0 | *** METH DEFAULT *** |

| Lvl ID | Conc (ppbv) | Response |
|--------|-------------|----------|
| .5 | 0.500 | 3620 |
| 1 | 1.000 | 7560 |
| 2 | 2.000 | 9093 |
| 10 | 10.000 | 38143 |
| 20 | 20.000 | 66366 |
| 40 | 40.000 | 134856 |

Qualifier Peak Analysis ON
Curve Fit: Avg. RF

23) cis-1,2-Dichloroethene ()
Ret. Time 7.12 min., Extract & Integrate from 6.87 to 7.37 min.

| Signal | Rel Resp. | Pct. Unc. (abs) | Integration |
|-----------|-----------|-----------------|----------------------|
| Tgt 96.00 | | | *** METH DEFAULT *** |
| Q1 61.00 | 142.10 | 20.0 | *** METH DEFAULT *** |
| Q2 98.00 | 64.40 | 20.0 | *** METH DEFAULT *** |

| Lvl ID | Conc (ppbv) | Response |
|--------|-------------|----------|
| .5 | 0.500 | 3977 |
| 1 | 1.000 | 8412 |
| 2 | 2.000 | 20165 |
| 10 | 10.000 | 83121 |
| 20 | 20.000 | 150631 |
| 40 | 40.000 | 271048 |

Qualifier Peak Analysis ON
Curve Fit: Avg. RF

24) 1,4-Difluorobenzene (ISTD TR)
Ret. Time 8.81 min., Extract & Integrate from 8.56 to 9.06 min.

| Signal | Rel Resp. | Pct. Unc. (abs) | Integration |
|------------|-----------|-----------------|----------------------|
| Tgt 114.00 | | | *** METH DEFAULT *** |

| Lvl ID | Conc (ppbv) | Response |
|--------|-------------|----------|
| .5 | 10.000 | 203074 |
| 1 | 10.000 | 208539 |
| 2 | 10.000 | 211638 |
| 10 | 10.000 | 199080 |
| 20 | 10.000 | 198767 |
| 40 | 10.000 | 183656 |

Qualifier Peak Analysis ON ISTD conc: 10.000 ppbv
Curve Fit: Avg. RF

25) Ethyl acetate ()
Ret. Time 7.26 min., Extract & Integrate from 7.01 to 7.51 min.

| Signal | Rel Resp. | Pct. Unc. (abs) | Integration |
|-----------|-----------|-----------------|----------------------|
| Tgt 70.00 | | | *** METH DEFAULT *** |
| Q1 43.00 | 1146.60 | 20.0 | *** METH DEFAULT *** |
| Q2 61.00 | 141.10 | 20.0 | *** METH DEFAULT *** |

| Lvl ID | Conc (ppbv) | Response |
|--------|-------------|----------|
| .5 | 0.500 | 987 |

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| | | |
|----|--------|-------|
| 1 | 1.000 | 2231 |
| 2 | 2.000 | 5281 |
| 10 | 10.000 | 22739 |
| 20 | 20.000 | 40291 |
| 40 | 40.000 | 73382 |

Qualifier Peak Analysis ON
Curve Fit: Avg. RF

26) Hexane

()

Ret. Time 7.27 min., Extract & Integrate from 7.02 to 7.52 min.

| Signal | Rel Resp. | Pct. Unc. (abs) | Integration |
|-----------|-----------|-----------------|----------------------|
| Tgt 57.00 | | | *** METH DEFAULT *** |
| Q1 41.00 | 88.30 | 20.0 | *** METH DEFAULT *** |

| Lvl ID | Conc (ppbv) | Response |
|--------|-------------|----------|
| .5 | 0.500 | 4157 |
| 1 | 1.000 | 8581 |
| 2 | 2.000 | 21272 |
| 10 | 10.000 | 84296 |
| 20 | 20.000 | 144376 |
| 40 | 40.000 | 258888 |

Qualifier Peak Analysis ON
Curve Fit: Avg. RF

27) Chloroform

()

Ret. Time 7.34 min., Extract & Integrate from 7.09 to 7.59 min.

| Signal | Rel Resp. | Pct. Unc. (abs) | Integration |
|-----------|-----------|-----------------|----------------------|
| Tgt 83.00 | | | *** METH DEFAULT *** |
| Q1 85.00 | 66.60 | 20.0 | *** METH DEFAULT *** |

| Lvl ID | Conc (ppbv) | Response |
|--------|-------------|----------|
| .5 | 0.500 | 10086 |
| 1 | 1.000 | 20702 |
| 2 | 2.000 | 50131 |
| 10 | 10.000 | 188210 |
| 20 | 20.000 | 331022 |
| 40 | 40.000 | 590252 |

Qualifier Peak Analysis ON
Curve Fit: Avg. RF

28) Tetrahydrofuran

()

Ret. Time 7.66 min., Extract & Integrate from 7.41 to 7.91 min.

| Signal | Rel Resp. | Pct. Unc. (abs) | Integration |
|-----------|-----------|-----------------|----------------------|
| Tgt 42.00 | | | *** METH DEFAULT *** |
| Q1 41.00 | 63.60 | 20.0 | *** METH DEFAULT *** |
| Q2 72.00 | 44.00 | 20.0 | *** METH DEFAULT *** |
| Q3 71.00 | 43.30 | 20.0 | *** METH DEFAULT *** |

| Lvl ID | Conc (ppbv) | Response |
|--------|-------------|----------|
| .5 | 0.500 | 4904 |
| 1 | 1.000 | 11049 |
| 2 | 2.000 | 28495 |
| 10 | 10.000 | 115803 |
| 20 | 20.000 | 214057 |
| 40 | 40.000 | 346149 |

Qualifier Peak Analysis ON
Curve Fit: Avg. RF

29) 1,1,1-Trichloroethane

()

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Ret. Time 8.15 min., Extract & Integrate from 7.90 to 8.40 min.

| Signal | Rel Resp. | Pct. Unc. (abs) | Integration |
|-----------|-----------|-----------------|----------------------|
| Tgt 97.00 | | | *** METH DEFAULT *** |
| Q1 99.00 | 64.10 | 20.0 | *** METH DEFAULT *** |
| Q2 61.00 | 44.50 | 20.0 | *** METH DEFAULT *** |

| Lvl ID | Conc (ppbv) | Response |
|--------|-------------|----------|
| .5 | 0.500 | 14319 |
| 1 | 1.000 | 29104 |
| 2 | 2.000 | 73120 |
| 10 | 10.000 | 280278 |
| 20 | 20.000 | 489627 |
| 40 | 40.000 | 794608 |

Qualifier Peak Analysis ON
Curve Fit: Avg. RF

30) Carbon tetrachloride ()

Ret. Time 8.67 min., Extract & Integrate from 8.42 to 8.92 min.

| Signal | Rel Resp. | Pct. Unc. (abs) | Integration |
|------------|-----------|-----------------|----------------------|
| Tgt 117.00 | | | *** METH DEFAULT *** |
| Q1 119.00 | 99.80 | 20.0 | *** METH DEFAULT *** |

| Lvl ID | Conc (ppbv) | Response |
|--------|-------------|----------|
| .5 | 0.500 | 13587 |
| 1 | 1.000 | 27522 |
| 2 | 2.000 | 67065 |
| 10 | 10.000 | 247401 |
| 20 | 20.000 | 429152 |
| 40 | 40.000 | 687842 |

Qualifier Peak Analysis ON
Curve Fit: Avg. RF

31) Cyclohexane ()

Ret. Time 8.77 min., Extract & Integrate from 8.52 to 9.02 min.

| Signal | Rel Resp. | Pct. Unc. (abs) | Integration |
|-----------|-----------|-----------------|----------------------|
| Tgt 84.00 | | | *** METH DEFAULT *** |
| Q1 56.00 | 113.40 | 20.0 | *** METH DEFAULT *** |
| Q2 41.00 | 77.80 | 20.0 | *** METH DEFAULT *** |

| Lvl ID | Conc (ppbv) | Response |
|--------|-------------|----------|
| .5 | 0.500 | 5508 |
| 1 | 1.000 | 11872 |
| 2 | 2.000 | 30368 |
| 10 | 10.000 | 111517 |
| 20 | 20.000 | 192100 |
| 40 | 40.000 | 311539 |

Qualifier Peak Analysis ON
Curve Fit: Avg. RF

32) Benzene ()

Ret. Time 8.54 min., Extract & Integrate from 8.29 to 8.79 min.

| Signal | Rel Resp. | Pct. Unc. (abs) | Integration |
|-----------|-----------|-----------------|----------------------|
| Tgt 78.00 | | | *** METH DEFAULT *** |
| Q1 77.00 | 23.80 | 20.0 | *** METH DEFAULT *** |

| Lvl ID | Conc (ppbv) | Response |
|--------|-------------|----------|
| .5 | 0.500 | 14361 |
| 1 | 1.000 | 30034 |
| 2 | 2.000 | 72197 |

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| | | |
|----|--------|--------|
| 10 | 10.000 | 268811 |
| 20 | 20.000 | 472579 |
| 40 | 40.000 | 762489 |

Qualifier Peak Analysis ON
Curve Fit: Avg. RF

33) 1,2-Dichloroethane ()

Ret. Time 7.95 min., Extract & Integrate from 7.70 to 8.20 min.

| Signal | Rel Resp. | Pct. Unc. (abs) | Integration |
|-----------|-----------|-----------------|----------------------|
| Tgt 62.00 | | | *** METH DEFAULT *** |
| Q1 64.00 | 33.80 | 20.0 | *** METH DEFAULT *** |

| Lvl ID | Conc (ppbv) | Response |
|--------|-------------|----------|
| .5 | 0.500 | 10748 |
| 1 | 1.000 | 22592 |
| 2 | 2.000 | 53004 |
| 10 | 10.000 | 210139 |
| 20 | 20.000 | 379413 |
| 40 | 40.000 | 666067 |

Qualifier Peak Analysis ON
Curve Fit: Avg. RF

34) 2-Butanone ()

Ret. Time 6.76 min., Extract & Integrate from 6.51 to 7.01 min.

| Signal | Rel Resp. | Pct. Unc. (rel) | Integration |
|-----------|-----------|-----------------|----------------------|
| Tgt 72.00 | | | *** METH DEFAULT *** |
| Q1 43.00 | 517.60 | 20.0 | *** METH DEFAULT *** |
| Q2 57.00 | 37.90 | 20.0 | *** METH DEFAULT *** |

| Lvl ID | Conc (ppbv) | Response |
|--------|-------------|----------|
| .5 | 0.500 | 1774 |
| 1 | 1.000 | 3755 |
| 2 | 2.000 | 7590 |
| 10 | 10.000 | 31386 |
| 20 | 20.000 | 59356 |
| 40 | 40.000 | 107870 |

Qualifier Peak Analysis ON
Curve Fit: Avg. RF

35) Trichloroethene ()

Ret. Time 9.43 min., Extract & Integrate from 9.18 to 9.68 min.

| Signal | Rel Resp. | Pct. Unc. (abs) | Integration |
|-----------|-----------|-----------------|----------------------|
| Tgt 95.00 | | | *** METH DEFAULT *** |
| Q1 130.00 | 66.80 | 20.0 | *** METH DEFAULT *** |
| Q2 132.00 | 65.10 | 20.0 | *** METH DEFAULT *** |

| Lvl ID | Conc (ppbv) | Response |
|--------|-------------|----------|
| .5 | 0.500 | 6924 |
| 1 | 1.000 | 15123 |
| 2 | 2.000 | 34830 |
| 10 | 10.000 | 123592 |
| 20 | 20.000 | 205003 |
| 40 | 40.000 | 335323 |

Qualifier Peak Analysis ON
Curve Fit: Avg. RF

36) 1,4-Dioxane ()

Ret. Time 9.41 min., Extract & Integrate from 9.16 to 9.66 min.

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| Signal | Rel Resp. | Pct. Unc. (rel) | Integration |
|-----------|-----------|-----------------|----------------------|
| Tgt 88.00 | | | *** METH DEFAULT *** |
| Q1 58.00 | 64.00 | 20.0 | *** METH DEFAULT *** |
| Q2 43.00 | 38.40 | 20.0 | *** METH DEFAULT *** |

| Lvl ID | Conc (ppbv) | Response |
|--------|-------------|----------|
| .5 | 0.500 | 3127 |
| 1 | 1.000 | 6831 |
| 2 | 2.000 | 16494 |
| 10 | 10.000 | 57918 |
| 20 | 20.000 | 92063 |
| 40 | 40.000 | 154969 |

Qualifier Peak Analysis ON
Curve Fit: Avg. RF

37) Methyl methacrylate ()

Ret. Time 9.57 min., Extract & Integrate from 9.32 to 9.82 min.

| Signal | Rel Resp. | Pct. Unc. (rel) | Integration |
|-----------|-----------|-----------------|----------------------|
| Tgt 41.00 | | | RTEINT.P |
| Q1 69.00 | 69.20 | 20.0 | RTEINT.P |
| Q2 39.00 | 64.60 | 20.0 | *** METH DEFAULT *** |
| Q3 100.00 | 23.70 | 20.0 | *** METH DEFAULT *** |

| Lvl ID | Conc (ppbv) | Response |
|--------|-------------|----------|
| .5 | 0.500 | 6213 |
| 1 | 1.000 | 13692 |
| 2 | 2.000 | 37019 |
| 10 | 10.000 | 146414 |
| 20 | 20.000 | 261230 |
| 40 | 40.000 | 442172 |

Qualifier Peak Analysis ON
Curve Fit: Avg. RF

38) Heptane ()

Ret. Time 9.67 min., Extract & Integrate from 9.42 to 9.92 min.

| Signal | Rel Resp. | Pct. Unc. (abs) | Integration |
|-----------|-----------|-----------------|----------------------|
| Tgt 43.00 | | | *** METH DEFAULT *** |
| Q1 57.00 | 49.70 | 20.0 | *** METH DEFAULT *** |
| Q2 71.00 | 52.40 | 20.0 | *** METH DEFAULT *** |

| Lvl ID | Conc (ppbv) | Response |
|--------|-------------|----------|
| .5 | 0.500 | 7487 |
| 1 | 1.000 | 16387 |
| 2 | 2.000 | 41944 |
| 10 | 10.000 | 150824 |
| 20 | 20.000 | 254656 |
| 40 | 40.000 | 445730 |

Qualifier Peak Analysis ON
Curve Fit: Avg. RF

39) 1,2-Dichloropropane ()

Ret. Time 9.23 min., Extract & Integrate from 8.98 to 9.48 min.

| Signal | Rel Resp. | Pct. Unc. (abs) | Integration |
|-----------|-----------|-----------------|----------------------|
| Tgt 63.00 | | | *** METH DEFAULT *** |
| Q1 62.00 | 76.00 | 20.0 | *** METH DEFAULT *** |
| Q2 41.00 | 73.50 | 20.0 | *** METH DEFAULT *** |

| Lvl ID | Conc (ppbv) | Response |
|--------|-------------|----------|
| .5 | 0.500 | 5151 |
| 1 | 1.000 | 10614 |

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| | | |
|----|--------|--------|
| 2 | 2.000 | 28155 |
| 10 | 10.000 | 109205 |
| 20 | 20.000 | 192927 |
| 40 | 40.000 | 303197 |

Qualifier Peak Analysis ON
Curve Fit: Avg. RF

40) Bromodichloromethane

()

Ret. Time 9.40 min., Extract & Integrate from 9.15 to 9.65 min.

| Signal | Rel Resp. | Pct. Unc. (abs) | Integration |
|-----------|-----------|-----------------|----------------------|
| Tgt 83.00 | | | *** METH DEFAULT *** |
| Q1 85.00 | 67.60 | 20.0 | *** METH DEFAULT *** |
| Q2 127.00 | 7.10 | 20.0 | *** METH DEFAULT *** |

| Lvl ID | Conc (ppbv) | Response |
|--------|-------------|----------|
| .5 | 0.500 | 16571 |
| 1 | 1.000 | 33825 |
| 2 | 2.000 | 81359 |
| 10 | 10.000 | 295900 |
| 20 | 20.000 | 491184 |
| 40 | 40.000 | 754782 |

Qualifier Peak Analysis ON
Curve Fit: Avg. RF

41) cis-1,3-Dichloropropene

()

Ret. Time 10.18 min., Extract & Integrate from 9.93 to 10.43 min.

| Signal | Rel Resp. | Pct. Unc. (abs) | Integration |
|-----------|-----------|-----------------|----------------------|
| Tgt 75.00 | | | *** METH DEFAULT *** |
| Q1 110.00 | 16.30 | 20.0 | *** METH DEFAULT *** |

| Lvl ID | Conc (ppbv) | Response |
|--------|-------------|----------|
| .5 | 0.500 | 9380 |
| 1 | 1.000 | 20517 |
| 2 | 2.000 | 46172 |
| 10 | 10.000 | 174722 |
| 20 | 20.000 | 303625 |
| 40 | 40.000 | 510258 |

Qualifier Peak Analysis ON
Curve Fit: Avg. RF

42) D5-Chlorobenzene

(ISTD TR)

Ret. Time 12.82 min., Extract & Integrate from 12.57 to 13.07 min.

| Signal | Rel Resp. | Pct. Unc. (abs) | Integration |
|------------|-----------|-----------------|----------------------|
| Tgt 117.00 | | | *** METH DEFAULT *** |
| Q1 82.00 | 83.90 | 20.0 | *** METH DEFAULT *** |
| Q2 119.00 | 33.80 | 20.0 | *** METH DEFAULT *** |
| Q3 54.00 | 39.80 | 20.0 | *** METH DEFAULT *** |

| Lvl ID | Conc (ppbv) | Response |
|--------|-------------|----------|
| .5 | 10.000 | 161735 |
| 1 | 10.000 | 173839 |
| 2 | 10.000 | 177317 |
| 10 | 10.000 | 187597 |
| 20 | 10.000 | 194775 |
| 40 | 10.000 | 189115 |

Qualifier Peak Analysis ON ISTD conc: 10.000 ppbv
Curve Fit: Avg. RF

43) Toluene

()

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Ret. Time 11.09 min., Extract & Integrate from 10.84 to 11.34 min.

| Signal | Rel Resp. | Pct. Unc. (abs) | Integration |
|-----------|-----------|-----------------|----------------------|
| Tgt 92.00 | | | *** METH DEFAULT *** |
| Q1 91.00 | 170.50 | 20.0 | *** METH DEFAULT *** |

| Lvl ID | Conc (ppbv) | Response |
|--------|-------------|----------|
| .5 | 0.500 | 9330 |
| 1 | 1.000 | 20086 |
| 2 | 2.000 | 45881 |
| 10 | 10.000 | 174371 |
| 20 | 20.000 | 304268 |
| 40 | 40.000 | 543102 |

Qualifier Peak Analysis ON
Curve Fit: Avg. RF

44) trans-1,3-Dichloropropene ()

Ret. Time 10.66 min., Extract & Integrate from 10.41 to 10.91 min.

| Signal | Rel Resp. | Pct. Unc. (abs) | Integration |
|-----------|-----------|-----------------|----------------------|
| Tgt 75.00 | | | *** METH DEFAULT *** |
| Q1 110.00 | 15.40 | 20.0 | *** METH DEFAULT *** |

| Lvl ID | Conc (ppbv) | Response |
|--------|-------------|----------|
| .5 | 0.500 | 11842 |
| 1 | 1.000 | 26420 |
| 2 | 2.000 | 50011 |
| 10 | 10.000 | 203450 |
| 20 | 20.000 | 358057 |
| 40 | 40.000 | 630244 |

Qualifier Peak Analysis ON
Curve Fit: Avg. RF

45) 1,1,2-Trichloroethane ()

Ret. Time 10.83 min., Extract & Integrate from 10.58 to 11.08 min.

| Signal | Rel Resp. | Pct. Unc. (abs) | Integration |
|-----------|-----------|-----------------|----------------------|
| Tgt 83.00 | | | *** METH DEFAULT *** |
| Q1 97.00 | 106.90 | 20.0 | *** METH DEFAULT *** |
| Q2 85.00 | 67.00 | 20.0 | *** METH DEFAULT *** |

| Lvl ID | Conc (ppbv) | Response |
|--------|-------------|----------|
| .5 | 0.500 | 6011 |
| 1 | 1.000 | 12438 |
| 2 | 2.000 | 30683 |
| 10 | 10.000 | 112716 |
| 20 | 20.000 | 198098 |
| 40 | 40.000 | 329492 |

Qualifier Peak Analysis ON
Curve Fit: Avg. RF

46) 4-Methyl-2-pentanone ()

Ret. Time 10.20 min., Extract & Integrate from 9.95 to 10.45 min.

| Signal | Rel Resp. | Pct. Unc. (rel) | Integration |
|-----------|-----------|-----------------|----------------------|
| Tgt 43.00 | | | *** METH DEFAULT *** |
| Q1 58.00 | 34.40 | 20.0 | *** METH DEFAULT *** |
| Q2 85.00 | 15.30 | 20.0 | *** METH DEFAULT *** |
| Q3 100.00 | 8.70 | 20.0 | *** METH DEFAULT *** |

| Lvl ID | Conc (ppbv) | Response |
|--------|-------------|----------|
| .5 | 0.500 | 13806 |
| 1 | 1.000 | 30049 |

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| | | |
|----|--------|--------|
| 2 | 2.000 | 68725 |
| 10 | 10.000 | 242418 |
| 20 | 20.000 | 414052 |
| 40 | 40.000 | 712215 |

Qualifier Peak Analysis ON
Curve Fit: Avg. RF

47) Tetrachloroethene

()

Ret. Time 12.19 min., Extract & Integrate from 11.94 to 12.44 min.

| Signal | Rel Resp. | Pct. Unc. (abs) | Integration |
|------------|-----------|-----------------|----------------------|
| Tgt 166.00 | | | *** METH DEFAULT *** |
| Q1 168.00 | 52.10 | 20.0 | *** METH DEFAULT *** |
| Q2 129.00 | 38.00 | 20.0 | *** METH DEFAULT *** |

| Lvl ID | Conc (ppbv) | Response |
|--------|-------------|----------|
| .5 | 0.500 | 16053 |
| 1 | 1.000 | 29796 |
| 2 | 2.000 | 65466 |
| 10 | 10.000 | 239064 |
| 20 | 20.000 | 407089 |
| 40 | 40.000 | 634928 |

Qualifier Peak Analysis ON
Curve Fit: Quadratic, forced through origin

48) Dibromochloromethane

()

Ret. Time 11.51 min., Extract & Integrate from 11.26 to 11.76 min.

| Signal | Rel Resp. | Pct. Unc. (abs) | Integration |
|------------|-----------|-----------------|----------------------|
| Tgt 129.00 | | | *** METH DEFAULT *** |
| Q1 127.00 | 77.10 | 20.0 | *** METH DEFAULT *** |

| Lvl ID | Conc (ppbv) | Response |
|--------|-------------|----------|
| .5 | 0.500 | 12100 |
| 1 | 1.000 | 25741 |
| 2 | 2.000 | 62615 |
| 10 | 10.000 | 234971 |
| 20 | 20.000 | 413741 |
| 40 | 40.000 | 714182 |

Qualifier Peak Analysis ON
Curve Fit: Avg. RF

49) 1,2-Dibromoethane

()

Ret. Time 11.74 min., Extract & Integrate from 11.49 to 11.99 min.

| Signal | Rel Resp. | Pct. Unc. (rel) | Integration |
|------------|-----------|-----------------|----------------------|
| Tgt 107.00 | | | *** METH DEFAULT *** |
| Q1 109.00 | 91.80 | 20.0 | *** METH DEFAULT *** |
| Q2 79.00 | 15.50 | 20.0 | *** METH DEFAULT *** |

| Lvl ID | Conc (ppbv) | Response |
|--------|-------------|----------|
| .5 | 0.500 | 10960 |
| 1 | 1.000 | 22941 |
| 2 | 2.000 | 50344 |
| 10 | 10.000 | 191447 |
| 20 | 20.000 | 346059 |
| 40 | 40.000 | 597086 |

Qualifier Peak Analysis ON
Curve Fit: Avg. RF

50) 2-Hexanone

()

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Ret. Time 11.32 min., Extract & Integrate from 11.07 to 11.57 min.

| Signal | Rel Resp. | Pct. Unc. (rel) | Integration |
|-----------|-----------|-----------------|----------------------|
| Tgt 43.00 | | | *** METH DEFAULT *** |
| Q1 58.00 | 46.20 | 20.0 | *** METH DEFAULT *** |
| Q2 57.00 | 16.10 | 20.0 | *** METH DEFAULT *** |
| Q3 100.00 | 6.90 | 20.0 | *** METH DEFAULT *** |

| Lvl ID | Conc (ppbv) | Response |
|--------|-------------|----------|
| .5 | 0.500 | 13450 |
| 1 | 1.000 | 28623 |
| 2 | 2.000 | 62833 |
| 10 | 10.000 | 247525 |
| 20 | 20.000 | 432981 |
| 40 | 40.000 | 749129 |

Qualifier Peak Analysis ON
Curve Fit: Avg. RF

51) Chlorobenzene

()

Ret. Time 12.87 min., Extract & Integrate from 12.62 to 13.12 min.

| Signal | Rel Resp. | Pct. Unc. (abs) | Integration |
|------------|-----------|-----------------|----------------------|
| Tgt 112.00 | | | *** METH DEFAULT *** |
| Q1 77.00 | 82.50 | 20.0 | *** METH DEFAULT *** |
| Q2 114.00 | 32.20 | 20.0 | *** METH DEFAULT *** |

| Lvl ID | Conc (ppbv) | Response |
|--------|-------------|----------|
| .5 | 0.500 | 9663 |
| 1 | 1.000 | 21112 |
| 2 | 2.000 | 47959 |
| 10 | 10.000 | 179593 |
| 20 | 20.000 | 325304 |
| 40 | 40.000 | 577513 |

Qualifier Peak Analysis ON
Curve Fit: Avg. RF

52) Ethylbenzene

()

Ret. Time 13.24 min., Extract & Integrate from 12.99 to 13.49 min.

| Signal | Rel Resp. | Pct. Unc. (abs) | Integration |
|-----------|-----------|-----------------|----------------------|
| Tgt 91.00 | | | *** METH DEFAULT *** |
| Q1 106.00 | 22.00 | 20.0 | *** METH DEFAULT *** |

| Lvl ID | Conc (ppbv) | Response |
|--------|-------------|----------|
| .5 | 0.500 | 19299 |
| 1 | 1.000 | 44720 |
| 2 | 2.000 | 112393 |
| 10 | 10.000 | 415572 |
| 20 | 20.000 | 745820 |
| 40 | 40.000 | 1261926 |

Qualifier Peak Analysis ON
Curve Fit: Avg. RF

53) m,p-Xylene

()

Ret. Time 13.43 min., Extract & Integrate from 13.18 to 13.68 min.

| Signal | Rel Resp. | Pct. Unc. (abs) | Integration |
|------------|-----------|-----------------|----------------------|
| Tgt 106.00 | | | *** METH DEFAULT *** |
| Q1 91.00 | 286.10 | 20.0 | *** METH DEFAULT *** |

| Lvl ID | Conc (ppbv) | Response |
|--------|-------------|----------|
| .5 | 1.000 | 11153 |
| 1 | 2.000 | 25025 |
| 2 | 4.000 | 59383 |

Method: SIM.M

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| | | |
|----|--------|--------|
| 10 | 20.000 | 209772 |
| 20 | 40.000 | 389286 |
| 40 | 80.000 | 734582 |

Qualifier Peak Analysis ON
Curve Fit: Avg. RF

54) o-Xylene

()

Ret. Time 13.92 min., Extract & Integrate from 13.67 to 14.17 min.

| Signal | Rel Resp. | Pct. Unc. (abs) | Integration |
|------------|-----------|-----------------|----------------------|
| Tgt 106.00 | | | *** METH DEFAULT *** |
| Q1 91.00 | 296.60 | 20.0 | *** METH DEFAULT *** |

| Lvl ID | Conc (ppbv) | Response |
|--------|-------------|----------|
| .5 | 0.500 | 5197 |
| 1 | 1.000 | 11463 |
| 2 | 2.000 | 28195 |
| 10 | 10.000 | 98514 |
| 20 | 20.000 | 183206 |
| 40 | 40.000 | 344249 |

Qualifier Peak Analysis ON
Curve Fit: Avg. RF

55) Styrene

()

Ret. Time 13.80 min., Extract & Integrate from 13.55 to 14.05 min.

| Signal | Rel Resp. | Pct. Unc. (abs) | Integration |
|------------|-----------|-----------------|----------------------|
| Tgt 104.00 | | | *** METH DEFAULT *** |
| Q1 78.00 | 72.30 | 20.0 | *** METH DEFAULT *** |

| Lvl ID | Conc (ppbv) | Response |
|--------|-------------|----------|
| .5 | 0.500 | 9082 |
| 1 | 1.000 | 21488 |
| 2 | 2.000 | 47287 |
| 10 | 10.000 | 177129 |
| 20 | 20.000 | 321611 |
| 40 | 40.000 | 581031 |

Qualifier Peak Analysis ON
Curve Fit: Avg. RF

56) Bromoform

()

Ret. Time 13.52 min., Extract & Integrate from 13.27 to 13.77 min.

| Signal | Rel Resp. | Pct. Unc. (abs) | Integration |
|------------|-----------|-----------------|----------------------|
| Tgt 173.00 | | | *** METH DEFAULT *** |
| Q1 175.00 | 52.60 | 20.0 | *** METH DEFAULT *** |

| Lvl ID | Conc (ppbv) | Response |
|--------|-------------|----------|
| .5 | 0.500 | 22609 |
| 1 | 1.000 | 46758 |
| 2 | 2.000 | 109135 |
| 10 | 10.000 | 403765 |
| 20 | 20.000 | 887272 |
| 40 | 40.000 | 1075001 |

Qualifier Peak Analysis ON
Curve Fit: Avg. RF

57) 4-Bromofluorobenzene

()

Ret. Time 14.41 min., Extract & Integrate from 14.16 to 14.66 min.

| Signal | Rel Resp. | Pct. Unc. (abs) | Integration |
|--------|-----------|-----------------|-------------|
|--------|-----------|-----------------|-------------|

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| | | | | |
|-----|--------|--------|------|----------------------|
| Tgt | 95.00 | | | *** METH DEFAULT *** |
| Q1 | 174.00 | 122.50 | 20.0 | *** METH DEFAULT *** |
| Q2 | 176.00 | 130.60 | 20.0 | *** METH DEFAULT *** |
| Q3 | 75.00 | 52.50 | 20.0 | *** METH DEFAULT *** |

| Lvl ID | Conc (ppbv) | Response |
|--------|-------------|----------|
| .5 | 10.000 | 167711 |
| 1 | 10.000 | 177104 |
| 2 | 10.000 | 177751 |
| 10 | 10.000 | 186695 |
| 20 | 10.000 | 195944 |
| 40 | 10.000 | 192077 |

Qualifier Peak Analysis ON
Curve Fit: Avg. RF

58) 1,1,2,2-Tetrachloroethane ()

Ret. Time 13.91 min., Extract & Integrate from 13.66 to 14.16 min.

| Signal | Rel Resp. | Pct. Unc. (abs) | Integration |
|--------|-----------|-----------------|----------------------|
| Tgt | 83.00 | | *** METH DEFAULT *** |
| Q1 | 131.00 | 7.50 | *** METH DEFAULT *** |
| Q2 | 85.00 | 66.00 | *** METH DEFAULT *** |

| Lvl ID | Conc (ppbv) | Response |
|--------|-------------|----------|
| .5 | 0.500 | 15159 |
| 1 | 1.000 | 30787 |
| 2 | 2.000 | 73117 |
| 10 | 10.000 | 233076 |
| 20 | 20.000 | 374190 |
| 40 | 40.000 | 585894 |

Qualifier Peak Analysis ON
Curve Fit: Quadratic, forced through origin

59) 4-Ethyltoluene ()

Ret. Time 15.31 min., Extract & Integrate from 15.06 to 15.56 min.

| Signal | Rel Resp. | Pct. Unc. (abs) | Integration |
|--------|-----------|-----------------|----------------------|
| Tgt | 105.00 | | *** METH DEFAULT *** |
| Q1 | 120.00 | 23.80 | *** METH DEFAULT *** |

| Lvl ID | Conc (ppbv) | Response |
|--------|-------------|----------|
| .5 | 0.500 | 17538 |
| 1 | 1.000 | 40888 |
| 2 | 2.000 | 101688 |
| 10 | 10.000 | 369724 |
| 20 | 20.000 | 678404 |
| 40 | 40.000 | 1203019 |

Qualifier Peak Analysis ON
Curve Fit: Avg. RF

60) 1,3,5-Trimethylbenzene ()

Ret. Time 15.39 min., Extract & Integrate from 15.14 to 15.64 min.

| Signal | Rel Resp. | Pct. Unc. (abs) | Integration |
|--------|-----------|-----------------|----------------------|
| Tgt | 105.00 | | *** METH DEFAULT *** |
| Q1 | 120.00 | 38.90 | *** METH DEFAULT *** |

| Lvl ID | Conc (ppbv) | Response |
|--------|-------------|----------|
| .5 | 0.500 | 14625 |
| 1 | 1.000 | 32989 |
| 2 | 2.000 | 80301 |
| 10 | 10.000 | 290059 |
| 20 | 20.000 | 527153 |
| 40 | 40.000 | 928852 |

Method: SIM.M

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Qualifier Peak Analysis ON
Curve Fit: Avg. RF

61) 1,2,4-Trimethylbenzene ()

Ret. Time 15.82 min., Extract & Integrate from 15.57 to 16.07 min.

| Signal | Rel Resp. | Pct. Unc. (abs) | Integration |
|------------|-----------|-----------------|----------------------|
| Tgt 105.00 | | | *** METH DEFAULT *** |
| Q1 120.00 | 36.80 | 20.0 | *** METH DEFAULT *** |
| Q2 119.00 | 9.80 | 20.0 | *** METH DEFAULT *** |
| Q3 77.00 | 17.30 | 20.0 | *** METH DEFAULT *** |

| Lvl ID | Conc (ppbv) | Response |
|--------|-------------|----------|
| .5 | 0.500 | 15395 |
| 1 | 1.000 | 34225 |
| 2 | 2.000 | 84068 |
| 10 | 10.000 | 308093 |
| 20 | 20.000 | 559426 |
| 40 | 40.000 | 982588 |

Qualifier Peak Analysis ON
Curve Fit: Avg. RF

62) alpha-Chlorotoluene ()

Ret. Time 15.96 min., Extract & Integrate from 15.71 to 16.21 min.

| Signal | Rel Resp. | Pct. Unc. (abs) | Integration |
|-----------|-----------|-----------------|----------------------|
| Tgt 91.00 | | | *** METH DEFAULT *** |
| Q1 126.00 | 12.60 | 20.0 | *** METH DEFAULT *** |
| Q2 65.00 | 12.60 | 20.0 | *** METH DEFAULT *** |
| Q3 39.00 | 9.40 | 20.0 | *** METH DEFAULT *** |

| Lvl ID | Conc (ppbv) | Response |
|--------|-------------|----------|
| .5 | 0.500 | 16730 |
| 1 | 1.000 | 35912 |
| 2 | 2.000 | 60179 |
| 10 | 10.000 | 221833 |
| 20 | 20.000 | 381095 |
| 40 | 40.000 | 594043 |

Qualifier Peak Analysis ON
Curve Fit: Quadratic, forced through origin

63) 1,3-Dichlorobenzene ()

Ret. Time 15.98 min., Extract & Integrate from 15.73 to 16.23 min.

| Signal | Rel Resp. | Pct. Unc. (abs) | Integration |
|------------|-----------|-----------------|----------------------|
| Tgt 146.00 | | | *** METH DEFAULT *** |
| Q1 111.00 | 34.10 | 20.0 | *** METH DEFAULT *** |
| Q2 148.00 | 69.00 | 20.0 | *** METH DEFAULT *** |

| Lvl ID | Conc (ppbv) | Response |
|--------|-------------|----------|
| .5 | 0.500 | 16064 |
| 1 | 1.000 | 33769 |
| 2 | 2.000 | 63471 |
| 10 | 10.000 | 218504 |
| 20 | 20.000 | 379913 |
| 40 | 40.000 | 643886 |

Qualifier Peak Analysis ON
Curve Fit: Linear, forced through origin

64) 1,4-Dichlorobenzene ()

Ret. Time 16.05 min., Extract & Integrate from 15.80 to 16.30 min.

Method: SIM.M Wed Jul 07 15:18:27 2010 Page: 22

| Signal | Rel Resp. | Pct. Unc. (abs) | Integration |
|------------|-----------|-----------------|----------------------|
| Tgt 146.00 | | | *** METH DEFAULT *** |
| Q1 111.00 | 33.30 | 20.0 | *** METH DEFAULT *** |
| Q2 148.00 | 69.80 | 20.0 | *** METH DEFAULT *** |

| Lvl ID | Conc (ppbv) | Response |
|--------|-------------|----------|
| .5 | 0.500 | 17305 |
| 1 | 1.000 | 35897 |
| 2 | 2.000 | 61753 |
| 10 | 10.000 | 215213 |
| 20 | 20.000 | 394100 |
| 40 | 40.000 | 663005 |

Qualifier Peak Analysis ON
Curve Fit: Linear, forced through origin

65) 1,2-Dichlorobenzene ()

Ret. Time 16.39 min., Extract & Integrate from 16.14 to 16.64 min.

| Signal | Rel Resp. | Pct. Unc. (abs) | Integration |
|------------|-----------|-----------------|----------------------|
| Tgt 146.00 | | | *** METH DEFAULT *** |
| Q1 111.00 | 36.30 | 20.0 | *** METH DEFAULT *** |
| Q2 148.00 | 69.40 | 20.0 | *** METH DEFAULT *** |

| Lvl ID | Conc (ppbv) | Response |
|--------|-------------|----------|
| .5 | 0.500 | 13839 |
| 1 | 1.000 | 29613 |
| 2 | 2.000 | 62541 |
| 10 | 10.000 | 223321 |
| 20 | 20.000 | 401902 |
| 40 | 40.000 | 655136 |

Qualifier Peak Analysis ON
Curve Fit: Avg. RF

66) 1,2,4-Trichlorobenzene ()

Ret. Time 18.06 min., Extract & Integrate from 17.81 to 18.31 min.

| Signal | Rel Resp. | Pct. Unc. (abs) | Integration |
|------------|-----------|-----------------|----------------------|
| Tgt 180.00 | | | *** METH DEFAULT *** |
| Q1 182.00 | 101.70 | 20.0 | *** METH DEFAULT *** |
| Q2 145.00 | 19.20 | 20.0 | *** METH DEFAULT *** |
| Q3 109.00 | 16.10 | 20.0 | *** METH DEFAULT *** |

| Lvl ID | Conc (ppbv) | Response |
|--------|-------------|----------|
| .5 | 0.500 | 30222 |
| 1 | 1.000 | 64103 |
| 2 | 2.000 | 86238 |
| 10 | 10.000 | 309040 |
| 20 | 20.000 | 514429 |
| 40 | 40.000 | 720641 |

Qualifier Peak Analysis ON
Curve Fit: Quadratic, forced through origin

67) Naphthalene ()

Ret. Time 18.17 min., Extract & Integrate from 17.92 to 18.42 min.

| Signal | Rel Resp. | Pct. Unc. (rel) | Integration |
|------------|-----------|-----------------|-------------|
| Tgt 128.00 | | | RTEINT.P |
| Q1 127.00 | 13.30 | 20.0 | RTEINT.P |
| Q2 129.00 | 11.20 | 20.0 | RTEINT.P |

| Lvl ID | Conc (ppbv) | Response |
|--------|-------------|----------|
| .5 | 0.500 | 14691 |
| 1 | 1.000 | 32151 |

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| | | |
|----|--------|--------|
| 2 | 2.000 | 75832 |
| 10 | 10.000 | 264815 |
| 20 | 20.000 | 484547 |
| 40 | 40.000 | 867153 |

Qualifier Peak Analysis ON
Curve Fit: Avg. RF

68) Hexachloro-1,3-butadiene

()

Ret. Time 18.55 min., Extract & Integrate from 18.30 to 18.80 min.

| Signal | Rel Resp. | Pct. Unc. (abs) | Integration |
|------------|-----------|-----------------|----------------------|
| Tgt 225.00 | | | *** METH DEFAULT *** |
| Q1 259.60 | 39.70 | 20.0 | *** METH DEFAULT *** |
| Q2 190.00 | 67.20 | 20.0 | *** METH DEFAULT *** |
| Q3 118.00 | 40.90 | 20.0 | *** METH DEFAULT *** |

| Lvl ID | Conc (ppbv) | Response |
|--------|----------------------------|----------|
| .5 | 0.500 | 14665 |
| 1 | 1.000 | 30735 |
| 2 | 2.000 | 76832 |
| 10 | 10.000 | 207077 |
| 20 | 20.000 | 296743 |
| 40 | not used for this compound | |

Qualifier Peak Analysis ON
Curve Fit: Avg. RF

END OF DATA ANALYSIS PARAMETERS

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SECTION 20.3: CANISTER CLEANING AND TRACKING LOG

E: TO-15 \ TO-15 Canister Cleaning and Tracking Log.xls.

TO-15 Canister Tracking

| Status (O=Out, P=In Process, V=Verified) | | | | | | | | | | | | | |
|--|-------|------|---------------|--------|--------------|--------|------------|---------|-----------|---------|-----------|-----------|---|
| Canister ID | GAUGE | Size | Date Received | Status | Date Cleaned | Oven # | QC Instr.# | QC Date | Analy. | Initial | Final Pre | Date Sent | Project |
| 1 | | 6 | | V | 1/22/2010 | 2 | | 1 | 1/26/2010 | 30 | 30 | | |
| 100 | | 6L | | O | | | | | | | | | |
| 2 | | 6 | | P | 122109 | | | | | 30 | 30 | | |
| 2 | 207 | 6 | | O | 122109 | 2 | | | | 30 | 30 | 123109 | indy pickup |
| 2 | | 6 | | V | 1/22/2010 | 2 | | 1 | 1/26/2010 | 30 | 29 | | |
| 3 | | 6 | | P | 122109 | | | | | 30 | 30 | | |
| 3 | 139 | 6 | | O | 122109 | 2 | | | | 30 | 30 | 123109 | indy pickup |
| 3 | | 6 | | V | 1/22/2010 | 2 | | 1 | 1/26/2010 | 30 | 30 | | |
| 4 | | 6 | | V | 10910 | 2 | | 2 | 11210 | 30 | 29 | | |
| 5 | | 6 | 122109 | P | | | | | | | | | MICROBAC-CENTRUM |
| 7 | | 6 | | V | 10910 | 2 | | 2 | 11210 | 28 | 27 | | |
| 8 | 205 | 6 | | O | | | | | | | | 1/8/2010 | hydrotech |
| 8 | | 6 | | V | 121509 | | | | | 30 | 30 | | |
| 9 | | 6 | | V | 1/22/2010 | 2 | | 1 | 1/26/2010 | 30 | 30 | | |
| 11 | | 6 | | V | 121009 | 2 | | | | 28 | 28 | | |
| 11 | | 6 | | O | | | | | | | | 11210 | mwh |
| 12 | 224 | 6 | | O | 110409 | 2 | | | | 26 | 26 | 123109 | indy pickup |
| 12 | | 6 | | V | 1/22/2010 | 2 | | 1 | 1/26/2010 | 30 | 30 | | |
| 13 | | 6 | | V | 121709 | 2 | | | | 28 | 28 | | |
| 14 | | 6 | | V | 10910 | 2 | | 2 | 11210 | 30 | 29 | | |
| 17 | | 6 | | V | 11410 | 2 | | 2 | 11810 | 28 | 28 | | |
| 18 | | 6 | | P | 122209 | 1 | | | | 23.5 | | | |
| 18 | 65 | 6 | | V | | | | | | | | 1/8/2010 | SW client? KCI RUSH |
| 19 | | 6 | | V | 11310 | 2 | | 2 | 11510 | 30 | 30 | | |
| 24 | | 6 | | V | 10510 | 2 | | 2 | 10710 | 25 | 25 | | |
| 24 | 209 | 6 | | O | | | | | | | | 11210 | mwh |
| 25 | | 6 | | V | 11310 | 2 | | 2 | 11510 | 30 | 30 | | |
| 26 | | 6 | | V | 10910 | 2 | | 2 | 11210 | 28 | 28 | | |
| 26 | | 6 | | V | 10910 | 2 | | 2 | 11210 | 30 | 29 | | |
| 26 | | 6 | | V | | | | | | | | 11310 | microbac ballimore returned unused 1/27 |
| 32 | | 6 | | V | 120209 | 2 | | 1 | 120709 | 28 | 29 | | |
| 33 | | 6 | | V | 11310 | 2 | | 2 | 11510 | 30 | 30 | | |
| 34 | | 6 | | V | 112809 | 2 | | | | 27 | 27 | | |
| 34 | | 6 | | O | | | | | | | | 11210 | mwh |
| 36 | 62 | 6 | | O | | | | | | | | 1/8/2010 | hydrotech |
| 38 | 208 | 6 | | O | | | | | | | | 1/8/2010 | hydrotech |
| 95 | | 6 | 122109 | P | | | | | | | | | MICROBAC-CENTRUM |
| 96 | | 6 | | P | 122209 | 1 | | | | 27 | | | |
| 96 | | 6 | | V | 123009 | 2 | | | | 27 | 27 | | |
| 96 | | 6 | | O | | | | | | | | 11210 | mwh |
| 97 | | 6 | 122109 | P | | | | | | | | | MICROBAC-CENTRUM |
| 97 | | 6 | | V | 10810 | 2 | | 2 | 11210 | 26.5 | 26 | | |
| 97 | | 6 | | V | | | | | | | | 11310 | microbac ballimore returned unused 1/27 |
| 99 | | 6 | | V | 10510 | 2 | | 2 | 10710 | 25 | 24.5 | | |
| 100 | | 6 | | O | 121509 | | | | | | | 122109 | MWH |
| 100 | | 6 | | V | 11410 | 2 | | 2 | 11810 | 28 | 28 | | |
| 101 | | 6 | | P | 122209 | 1 | | | | 27 | | | |

SECTION 20.4: TO-15 CANISTER ORDER LOG

E: TO-15 \ TO-15 Canister Cleaning and Tracking Log.xls.

TO-15 Canister Orders

[illegible]

SECTION 20.5: FLOW METER TABLE

032-41

FLOWMETER CALIBRATION DATA

DOCUMENT No. 590

Tube Number: 032-41(GL,SA,ST,CA,TA)
Units : std ml/min

Metering Temperature: 70 deg.F
Metering Pressure : 14.70 psia

STD. Conditions: 1 atmos @ 70 deg.F
Accuracy : Standard

SCALE READINGS AT CENTER OF FLOAT

| Glass (GL) | | Sapphire (SA) | | Stainless Steel (ST) | | Carboloy (CA) | | Tantalum (TA) | |
|---------------------------|---------------|----------------|---------------|----------------------|---------------|----------------|---------------|----------------|---------------|
| Float Density : 2.55 g/ml | | 3.98 g/ml | | 8.04 g/ml | | 14.98 g/ml | | 16.58 g/ml | |
| Scale Readings | Flow [ml/min] | Scale Readings | Flow [ml/min] | Scale Readings | Flow [ml/min] | Scale Readings | Flow [ml/min] | Scale Readings | Flow [ml/min] |
| A I R | | | | | | | | | |
| 150 --- | 49.1 | 150 --- | 73.0 | 150 --- | 143.1 | 150 --- | 266.7 | 150 --- | 266.1 |
| 140 --- | 42.8 | 140 --- | 63.7 | 140 --- | 128.5 | 140 --- | 223.2 | 140 --- | 240.4 |
| 130 --- | 37.7 | 130 --- | 56.8 | 130 --- | 113.5 | 130 --- | 198.9 | 130 --- | 216.4 |
| 120 --- | 32.7 | 120 --- | 49.2 | 120 --- | 103.3 | 120 --- | 182.3 | 120 --- | 191.0 |
| 110 --- | 27.9 | 110 --- | 42.5 | 110 --- | 92.5 | 110 --- | 164.3 | 110 --- | 167.1 |
| 100 --- | 23.7 | 100 --- | 36.4 | 100 --- | 80.3 | 100 --- | 143.9 | 100 --- | 142.3 |
| 90 --- | 20.5 | 90 --- | 31.3 | 90 --- | 69.2 | 90 --- | 125.1 | 90 --- | 123.1 |
| 80 --- | 17.3 | 80 --- | 26.3 | 80 --- | 60.1 | 80 --- | 109.6 | 80 --- | 103.6 |
| 70 --- | 13.9 | 70 --- | 21.1 | 70 --- | 48.9 | 70 --- | 89.3 | 70 --- | 84.5 |
| 60 --- | 11.3 | 60 --- | 16.8 | 60 --- | 38.1 | 60 --- | 73.1 | 60 --- | 67.0 |
| 50 --- | 8.3 | 50 --- | 12.7 | 50 --- | 33.0 | 50 --- | 61.8 | 50 --- | 49.6 |
| 40 --- | 6.7 | 40 --- | 10.0 | 40 --- | 26.7 | 40 --- | 50.3 | 40 --- | 40.8 |
| 30 --- | 4.7 | 30 --- | 6.9 | 30 --- | 19.5 | 30 --- | 36.9 | 30 --- | 28.1 |
| 20 --- | 3.3 | 20 --- | 4.6 | 20 --- | 12.8 | 20 --- | 24.4 | 20 --- | 18.9 |
| 10 --- | 2.0 | 10 --- | 3.0 | 10 --- | 8.4 | 10 --- | 16.0 | 10 --- | 11.5 |

| Scale Readings | Flow [ml/min] | Scale Readings | Flow [ml/min] | Scale Readings | Flow [ml/min] | Scale Readings | Flow [ml/min] | Scale Readings | Flow [ml/min] |
|------------------|---------------|----------------|---------------|----------------|---------------|----------------|---------------|----------------|---------------|
| W A T E R | | | | | | | | | |
| 150 --- | 0.49 | 150 --- | 0.98 | 150 --- | 2.34 | 150 --- | 4.7 | 150 --- | 5.1 |
| 140 --- | 0.43 | 140 --- | 0.86 | 140 --- | 2.09 | 140 --- | 4.3 | 140 --- | 4.5 |
| 130 --- | 0.38 | 130 --- | 0.76 | 130 --- | 1.85 | 130 --- | 3.8 | 130 --- | 4.0 |
| 120 --- | 0.33 | 120 --- | 0.66 | 120 --- | 1.63 | 120 --- | 3.2 | 120 --- | 3.5 |
| 110 --- | 0.28 | 110 --- | 0.56 | 110 --- | 1.45 | 110 --- | 2.8 | 110 --- | 3.0 |
| 100 --- | 0.24 | 100 --- | 0.47 | 100 --- | 1.25 | 100 --- | 2.3 | 100 --- | 2.6 |
| 90 --- | 0.20 | 90 --- | 0.40 | 90 --- | 1.05 | 90 --- | 1.9 | 90 --- | 2.2 |
| 80 --- | 0.16 | 80 --- | 0.33 | 80 --- | 0.90 | 80 --- | 1.6 | 80 --- | 1.8 |
| 70 --- | 0.13 | 70 --- | 0.26 | 70 --- | 0.72 | 70 --- | 1.3 | 70 --- | 1.4 |
| 60 --- | 0.10 | 60 --- | 0.20 | 60 --- | 0.58 | 60 --- | 1.1 | 60 --- | 1.1 |
| 50 --- | 0.07 | 50 --- | 0.15 | 50 --- | 0.48 | 50 --- | 0.8 | 50 --- | 0.7 |
| 40 --- | 0.06 | 40 --- | 0.12 | 40 --- | 0.37 | 40 --- | 0.6 | 40 --- | 0.6 |
| 30 --- | 0.04 | 30 --- | 0.08 | 30 --- | 0.26 | 30 --- | 0.4 | 30 --- | 0.5 |
| 20 --- | 0.03 | 20 --- | 0.06 | 20 --- | 0.19 | 20 --- | 0.3 | 20 --- | 0.3 |
| 10 --- | 0.02 | 10 --- | 0.04 | 10 --- | 0.12 | 10 --- | 0.2 | 10 --- | 0.2 |

Microbac Laboratories, Inc. - Controlled Document #117
E:\Policies and Procedures (SOPs)\Technical Operations\Organics\Controlled Documents

SECTION 20.6: TO-15 CANISTER LABELS



Microbac Laboratories, Inc.
250 West 84th Drive
Merrillville, IN 46410
219-769-8378

Sample ID: _____ Sample Date: _____

Time started: _____ @ _____ in Hg

Time ended: _____ @ _____ in Hg

Canister Certification

Canister ID: _____

Date Cleaned: _____ ☐ individually certified

Date Verified: _____ ☐ batch certified

Initial Pressure: _____ Final Pressure: _____

SECTION 20.7: RUN LOG

TO-15 RUN LOG

INST #

DATE _____

IS/SURR/TUNE# _____

[illegible]

SECTION 20.8: ICAL REVIEW CHECKLIST

Microbac Laboratories
ICAL Review Checklist
Method TO-15

Instrument ID: _____

Analyst: _____

Calibration
Date: _____

Quant. Method Name: _____

1st Level Technical Review

| Review Element | Evaluation | Comments (use this space as needed) |
|---|--|-------------------------------------|
| %RSD of all* individual target analytes ≤ 30 ? (* 2 compounds may have RSD up to 40) | <input type="checkbox"/> Yes <input type="checkbox"/> No <input type="checkbox"/> NA | |
| Minimum of 5 calibration levels for all components? | <input type="checkbox"/> Yes <input type="checkbox"/> No <input type="checkbox"/> NA | |
| Method properly calibrated and saved? (including) <ul style="list-style-type: none"> • RTs correct? • Concentrations correct? • Reference spectra updated? • ICV acceptance criteria met? | <input type="checkbox"/> Yes <input type="checkbox"/> No <input type="checkbox"/> NA <input type="checkbox"/> Yes <input type="checkbox"/> No <input type="checkbox"/> NA <input type="checkbox"/> Yes <input type="checkbox"/> No <input type="checkbox"/> NA <input type="checkbox"/> Yes <input type="checkbox"/> No <input type="checkbox"/> NA | |

Initials: _____

Date: _____

2nd Level Technical Review

Above assessment accurate

☐ Yes ☐ No

Data accurate in LIMS

☐ Yes ☐ No

If "No", list unacceptable evaluation(s): _____

LIMS QA Validation performed

☐ Yes ☐ No

Initials: _____

Date: _____

Revision 1, February 2010

SECTION 20.9: PREP LOGSHEET

Microbac Laboratories- Chicago Division
TO-15 AIR Standard Preparation Logbook

Page: 1

[illegible]

Revised July 10, 2009

SECTION 20.10: SOP REVISION FORM



SOP Revision Notification / Annual Review Form

SOP Name **DETERMINATION OF VOLATILE ORGANIC COMPOUNDS (VOCs) IN AIR COLLECTED IN SPECIALLY-PREPARED CANISTERS AND ANALYZED BY GC/MS**

| | | | |
|---|--------------|-------------------------|-------------------------|
| X | New Revision | Old Revision # <u>3</u> | New Revision # <u>4</u> |
|---|--------------|-------------------------|-------------------------|

SUMMARY OF CHANGES:

1. Edited section 10.7.1, which refers to ICVs: ~~"Acceptance criteria are the nominal limits listed in the appropriate test code in LIMS. These limits are based on the NELAC certification. The Marginal Exceedance criteria in section 10.12 may be used to evaluate ICVs. The number of allowable Marginal Exceedances is based on the total method analyte list. Acceptance criteria are a response <30% deviation from the initial calibration standard. NOTE: if linear regression was used to assess the linearity of the initial calibration, percent drift is used in place of percent deviation. Two compounds are allowed above the 30% deviation but must be less than 40% . If the acceptance criteria are not met, reanalyze."~~
2. Edited section 10.10.1, which refers to LCSs: ~~"Acceptance criteria are the nominal limits listed in the appropriate test code in LIMS. These limits are based on the NELAC certification. The Marginal Exceedance criteria may be used to evaluate LCSs. The number of allowable Marginal Exceedances is based on the total method analyte list. If the acceptance criteria are not met, reanalyze. Acceptance criteria are a response <30% deviation from the initial calibration standard. NOTE: if linear regression was used to assess the linearity of the initial calibration, percent drift is used in place of percent deviation. Two compounds are allowed above the 30% deviation but must be less than 40% . If the acceptance criteria are not met, reanalyze."~~
3. Deleted section 10.13: "[10.13] Marginal Exceedances [10.13.1] The laboratory may use marginal exceedances for the evaluation of second source standards when results fall outside of Control Limits. The Marginal Exceedance Limits provide an addition 10% range from the Control Limits for each analyte. [10.13.2] The number of allowable marginal exceedances is based on the number of analytes in the LCS or ICV. If more analytes exceed the LCS and ICV control limits than is allowed, or if any on analyte exceeds the ME limits; then the LCS or ICV fails and corrective action is necessary. [10.13.3] The number of allowable Marginal Exceedances can be found using the TO-15 Marginal Exceedance Calculator for on the server at E:Forms\QC Forms\TO-15 Marginal Exceedance Calculator.exl. Add Percent Recoveries by copying from LIMs and pasting them into Column E. Count the number of False results in column J. [10.13.4] The number of allowable Marginal Exceedances is as follows: [•] >90 analytes in LCS, 5 analytes allowed in ME of the LCS control limit. [•] 71-90 analytes in LCS, 4 analytes allowed in ME of the LCS control limit. [•] 51-70 analytes in LCS, 3 analytes allowed in ME of the LCS control limit. [•] 31-50 analytes in LCS, 2 analytes allowed in ME of

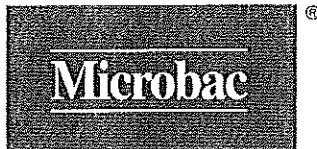


the LCS control limit. [•] 11-30 analytes in LCS, 1 analytes allowed in ME of the LCS control limit. [•] <11 analytes in LCS, 0 analytes allowed in ME of the LCS control limit. [10.12.5] **NOTE: THIS SECTION WAS MISNUMBERED. IT SHOULD HAVE BEEN 10.13.5** Exceedances must be random. If they are not, corrective action is required. Non-random exceedances are defined as three consecutive occurrences of the bias for any given analyte. Track exceedances by creating a new worksheet in the Marginal Exceedance Calculator and copying and pasting the calculated ME results onto the new worksheet. Label the worksheet with the run date. Keep worksheets in order of run date. Look back over the most recent three or four runs to ensure that the same MEs are not being repeated."

4. Added section 12.2: "[12.2] Prep Batch Creation [12.2.1] Make a batch in Element. [12.2.2] Make a bench sheet in Element. Use the comment field to enter if the dilution was done in the sample container. [12.2.3] Make sequence in Element. Add tune, CCV, QC samples, and batch samples. Enter the stanard ID and ISTD ID for tune and ccv. [12.2.4] Run samples. [12.2.5] Look in c:\Smart for daily .REP file for the actual sample amount analyzed by concentrator. [12.2.6] Enter the actual sample amount into the bench sheet as the initial and final volume. [12.2.7] Using 500cc as the normal sample amount, divide [500/actual sample amt analyzed] to calculate the dilution factor [DF]. [12.2.8] Multiply the DF by any dilution done in the sample container before analysis. [12.2.9] Enter the total dilution amount in the Chemstation multiplier field. [12.2.10] Import data into LIMS."

By signing below, I certify that I have been *notified* about the approval of a *new revision* to the above mentioned SOP. I realize it is *my responsibility* to read, understand and perform the procedure as set forth in this new revision.

| Initials & Date | Initials & Date | Initials & Date |
|-----------------|-----------------|-----------------|
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Microbac Laboratories, Inc.
SOP Revision Request Form

Request

Date: 10/10/2011

Requested by: Donna Ruokonen

SOP ID: 608-8081(6) SOP, 608-8082(6) SOP, 624-8260(6), 625-8270(11), 8151A(1), EXP8330(0), GRO-DRO-TPH(0), TO-11(0), TO15(5), and TPH-ERO(1),

Requested Change(s):
Section 10 for surrogates add the following:

-In the instances where samples are reported from multiple runs, the surrogate(s) must be reported from the first reported run or the least diluted if multiple dilutions exist.

Approval

Unit Supervisor: [Signature]

Date: 10-10-2011

QA Manager: [Signature]

Date: 10-10-2011

Notification

I am aware of these change(s) and I will implement them the next time I perform this procedure.

Initials & Date

Initials & Date

Initials & Date

Distribution

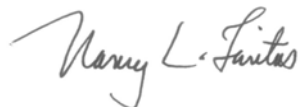
Upon approval and notification, make sufficient copies to be attached to the controlled copies of the SOP, which have been distributed in the lab. Attach this original form to the original SOP maintained in the QA office. Attach the copies of this form to the copies available in the lab. Notify all appropriate personnel of the change approved by this form. The related SOP should be revised ASAP but in no longer than 3 months from the date of this approval.

Upon signed approval and attachment to the above listed SOP, the changes listed are considered required elements of the SOP.

**STANDARD OPERATING PROCEDURE FOR
GC/MS DETERMINATION OF VOLATILE ORGANICS COMPOUNDS
AND VOLATILE PETROLEUM HYDROCARBONS**

Revision Author: Nancy Tavitas

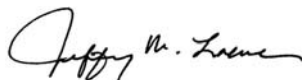
This SOP is effective upon signed approval by the following:



5/15/09

Organics Manager

Date



5/16/2009

QA/QC Manager

Date

DISCLAIMER: This SOP has been developed for use at the Microbac Laboratories, Merrillville, Indiana facility. It is intended for use by trained analysts. As written, this SOP may not be specifically applicable to the activities of other organizations.

1.0 TABLE OF CONTENTS

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2.0 SCOPE AND APPLICATION

- 2.1 This is a GC/MS procedure for the determination of volatile organic compounds. This procedure is applicable to the analysis of aqueous, non-aqueous liquid and solid matrix samples. The applicable compounds and their routine reporting limits are listed in the applicable test codes in LIMS. This procedure can also be used to determine the concentration of Volatile Petroleum Hydrocarbons, specifically gasoline or its individual components, which can be reported as Gasoline Range Organics.
- 2.2 The different and distinct autosampler units are employed as part of this procedure.
- The Tekmar 2016 autosampler is used to analyze samples according to SW-846 Method 5030B, which is a purge-and-trap procedure for aqueous and water miscible liquid samples. In certain applications, the 2016ALS and Method 5030B may be used for the analysis of soil samples as well.
 - The Solatek 72 autosampler is used to analyze samples according to SW-846 Method 5035, which utilizes 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). The Solateks can also be used for the analysis of water samples.
- 2.3 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.

3.0 SUMMARY

- 3.1 Samples are loaded onto an autosampler and the volatile compounds are introduced into the gas chromatograph using the purge-and-trap technique. The analytes are directly injected onto the capillary column of the GC. The column is temperature programmed to separate the analytes that are detected, using ion counts, with a mass spectrometer interfaced to a gas chromatograph. Target ions are extracted from the total ion count to identify target compounds.
- 3.2 Water samples are collected in glass vials with zero headspace. When using the Tekmar 2016, an aliquot of sample is transferred into a test tube using a gas-tight syringe. When using the Solatek, an aliquot of sample (typically 5-ml or 25-ml) is withdrawn directly from the sample vial.
- 3.3 Soil samples are collected in a variety of sample containers dependent upon the application. When using the Tekmar 2016 for low level solid samples, 5g of sample is weighed into a test tube and 5-ml of lab pure water added prior to loading the tube onto the autosampler. When using the Solatek for low level solid samples, 5-g of sample is added to a vial (either during collection or at the lab), which is loaded onto the autosampler for analysis. For medium and high level solid samples, 5g of

sample is extracted with 5-ml methanol and a portion of this extract is diluted with lab pure water, purged and analyzed.

- 3.4 The calibration range for most target analytes using 5ml or 5g of sample is 5-200 ppb.
- 3.5 This procedure is based on the reference methods listed in section 17 of this document and follows the applicable criteria of SW-846 Methods 8000C/8260B or EPA Method 624.. This procedure contains no significant deviations from the reference methods.

4.0 DEFINITIONS

- 4.1 A list of definitions is in the Quality Assurance Plan. In addition to the terms defined in the QAP.

5.0 INTERFERENCES

- 5.1 Chemical interference is minimized through the use of ion counts and internal standards.
- 5.2 Occasionally, samples may foam while being purged. This problem can be corrected through dilution or the use of a silicone-based anti-foaming agent.
- 5.3 Contamination may occur from impurities in the purge gas, carryover from high-level samples or environmental contamination resulting from the introduction of target analytes into the lab. Method blanks are used to verify that the system is clean and suitable for analysis. Analyzing additional blanks and/or baking out the column between analyses can eliminate carryover contamination.

6.0 SAFETY

- 6.1 Consult the current revision of the Chemical Hygiene Plan. Requirements for the use of personal protective equipment (e.g. safety glasses, lab coats, gloves) as well as other area-specific safety requirements (e.g. gas cylinders) and MSDS sheets are addressed in the CHP.

7.0 EQUIPMENT AND SUPPLIES

The following is a list of materials needed to perform the steps of this procedure as written. See the reference method(s) for equipment and supply specifications.

- 7.1 All volumetric glassware used shall be ASTM Class A. Glass microliter syringes are considered Class A provided they come with a certificate attesting to the accuracy of the syringe. Syringes without vendor certification are considered Class B glassware. Class B glassware must be verified for accuracy on an annual basis and labeled with an appropriate correction factor.

7.2 GC/MS System consisting of the following:

- 7.2.1 HP 5890 Gas Chromatograph or HP 6890 Gas Chromatograph
- 7.2.2 Tekmar SOLAtek® 72 autosampler
- 7.2.3 Tekmar 2016 ALS with disposable 25-ml autosampler tubes
- 7.2.4 Tekmar LSC 2000, LSC3000 or LSC3100 purge and trap concentrator
- 7.2.5 Vocab 3000 Trap: Supelco Purge Trap K catalog # 24920-U. Equivalent products from other vendors may be used.
- 7.2.6 HP 5971, 5972, or 5973 Mass Selective Detector
- 7.2.7 Chromatographic column: J&W Scientific DB-624 catalog # 128-1324: dimensions: length 25m, ID 0.2mm, film thickness 1.12um; or Phenomenex Zebron ZB-624 catalog # 7HG-G005-27: dimensions: length 30m, ID 0.25mm, film thickness 1.4um. Equivalent products from other vendors may be used.
- 7.3 Computer with MS Chemstation software, monitor, and printer
- 7.4 Various gas-tight syringes including 10-ul, 25-ul, 50-ul, 100-ul, 500-ul and 1000-ul.
- 7.5 5-ml gas-tight syringes with Luer-lock tip
- 7.6 2-ml autosampler vials with crimp tops or screw caps
- 7.7 1-ml micro reaction vessels with mini-inert valves
- 7.8 40-ml glass VOA vials with Teflon lined screw cap septa
- 7.9 Top loading balance capable of ± 0.01 g sensitivity
- 7.10 Disposable glass pipettes

8.0 REAGENTS AND STANDARDS

- 8.1 All reagents used must be analytical reagent (AR) grade or higher. All standards must be traceable to NIST, when available. Certificates of traceability must be obtained from the manufacturer. All reagents and standards must be documented in the appropriate preparation logbook. Refer to the requirements in the Labeling of Standards, Reagents, Digestates and Extracts SOP.

8.2 Reagents

All reagents are stored in the VOA lab unless otherwise noted.

8.2.1 Lab pure water (DI water): Analyte free water is prepared as described in the Quality Assurance Plan supplemented with additional carbon filtration after the general treatment. DI water may be obtained from the designated tap in the VOA lab.

8.2.2 Methanol, purge and trap grade

8.3 Standards

All standards are stored in the standards freezer in the VOA lab unless otherwise instructed by the manufacturer's recommendation. Expiration of prepared standards must be performed in accordance with the Labeling of Standards, Reagents, Digestates and Extracts SOP.

NOTE: The following solutions are used to prepare standards for the analysis of individual compounds. Refer to the last step in this standards section for the preparation of standards for "gasoline". The expiration date for stock standards is one year from the date the stock standard is opened, or sooner, if the vendor expiration date is earlier.

8.3.1 Stock Internal Standard (I.S.), 2000 ug/ml each in methanol. Equivalent products from other vendors may be used.

| VENDOR | CAT # | COMPOUNDS |
|--------------|------------------|---|
| Accustandard | M-8260A/B-IS-10X | Chlorobenzene-d5; 1,4-Dichlorobenzene-d5; Fluorobenzene |

8.3.2 Stock Surrogate (SURR) Standard, 2000 ug/ml each in methanol. Equivalent products from other vendors may be used.

| VENDOR | CAT # | COMPOUNDS |
|--------------|------------------|---|
| Accustandard | M-8260A/B-SS-10X | 4-Bromofluorobenzene; Dibromofluoromethane; 1,2-Dichloroethane-d5; Toluene-d8 |

8.3.3 Working I.S./SURR Solution, 50 ug/ml each: In a 10-ml volumetric flask, dilute 250-ul of Stock I.S. and Stock SURR standards to the mark with methanol. Prepare new solution at a minimum of every 6-months. The addition of 5-ul of this solution to 5-ml/5-g of sample yields a concentration of 50 ug/L (50 ug/kg). Addition of 5-ul of this solution to 25-ml of sample yields a concentration of 10-ug/L.

8.3.4 Stock Calibration Standards, 2000 ug/ml each in methanol (unless otherwise noted). Equivalent products from other vendors may be used.

| Stock Standard | VENDOR | CAT # | COMPOUNDS |
|-------------------------|--------------|----------------|---|
| Non-gases | Accustandard | M-502A-R2-10X | See Table in Section 18.0 |
| 8260 Mix 2 | Supelco | 46831-U | Acetone; 2-Butanone; Carbon disulfide; 2- Chloroethylvinyl ether; 2- Hexanone; Iodomethane; 4- Methyl-2-pentanone; Vinyl acetate |
| Gases | Accustandard | M-502B-10X | Bromomethane; Chloroethane; Chloromethane; Dichlorodifluoromethane; Trichlorofluoromethane; Vinyl chloride |
| Acrolein/ Acrylonitrile | Accustandard | M-603-10X | Acrolein; Acrylonitrile @ 10,000 ug/ml each (in water) |
| Acetonitrile | Accustandard | APP-9-005-10X | Acetonitrile @ 1000 ug/ml |
| MTBE | Accustandard | S-078-10X | Methyl-t-butyl ether |
| Misc. | Accustandard | Custom-S-15177 | 1,3-Butadiene; Hexane; Methyl methacrylate; Methyl acrylate; Ethyl acrylate; Butyl acrylate; Cyclohexane; 2-Methylnaphthalene; Di-cyclopentadiene; @2000 ug/ml each |

8.3.5 Intermediate Calibration Standards: In separate 1-ml volumetric flasks, prepare the following dilutions of the stock calibration standards with methanol. Prepare new solutions at a minimum of every 6-months with the exception of the Gases, which must be prepared on a weekly basis. Transfer each standard to a 1.0-ml micro-reaction vessel with mini-inert valve.

| Standard Name | Stock Standard | Vol. Stock Cal. Std., ul | Final Conc., ug/ml |
|-------------------------------------|---|--------------------------------|--------------------------|
| VOA Calibration Std./MTBE | (1) Non-gases; (2) 8260 Mix 2; (3) MTBE | 25 each | 50 |
| Acrolein/Acrylonitrile/Acetonitrile | (1) Acrolein/Acrylonitrile; (2) Acetonitrile | (1) 50 (2) 500 | 500 |
| VOA Misc. Std. | Miscellaneous | 25 | 50 |
| VOA Gas Std. | Gases | 25 | 50 |

8.3.6 Working Calibration Standards (Linearity, ICAL):

Water Linearity (5-ml) SOLATEK - In separate 25-ml volumetric flasks; prepare the following dilutions using each of the 4 intermediate calibration standards into lab pure water. For the 5 ppb standard, use a 50-ml volumetric flask. Transfer the prepared standards to 40-ml VOA vials.

| Linearity Standard # | Vol. Inter. Cal. Std, ul | Final Conc. ug/L |
|----------------------|--------------------------|------------------|
| 1 | 5 | 5 |
| 2 | 5 | 10 |
| 3 | 10 | 20 |
| 4 | 25 | 50 |
| 5 | 35 | 70 |
| 6 | 50 | 100 |
| 7 | 100 | 200 |

Water Linearity (25-ml) - In separate 50-ml volumetric flasks, prepare the following dilutions using each of the 4 intermediate calibration standards into lab pure water. Transfer the prepared standards to 40-ml VOA vials leaving zero headspace.

| Linearity Standard # | Vol. Inter. Cal. Std, ul | Final Conc. ug/L |
|----------------------|--------------------------|------------------|
| 1 | 1 | 1 |
| 2 | 2 | 2 |
| 3 | 4 | 4 |
| 4 | 8 | 8 |
| 5 | 10 | 10 |
| 6 | 20 | 20 |
| 7 | 40 | 40 |

Soil Linearity SOLATEK – Using a 5.0-ml syringe; individually prepare the following dilutions using each of the 4 intermediate calibration standards into 5.0-ml lab pure water. Transfer the prepared standards to 40-ml VOA vials for analysis.

| Linearity Standard # | Vol. Inter. Cal. Std, ul | Final Conc. ug/L |
|----------------------|---------------------------|------------------|
| 1 | 1 ($V_f = 10\text{ml}$) | 5 |
| 2 | 1 | 10 |
| 3 | 2 | 20 |
| 4 | 5 | 50 |
| 5 | 7 | 70 |
| 6 | 10 | 100 |
| 7 | 20 | 200 |

Water/Soil Linearity TEKMAR 2016 - In a 5-ml syringe, individually prepare the following dilutions using each of the 4 intermediate calibration standards into 5.0-ml lab pure water and 5-uL of the working ISTD/SURR solution. Transfer the prepared standards to separate ALS tubes for analysis.

| Linearity Standard # | Vol. Inter. Cal. Std, ul | Final Conc. ug/L |
|----------------------|---------------------------|------------------|
| 1 | 1 ($V_f = 10\text{ml}$) | 5 |
| 2 | 1 | 10 |
| 3 | 2 | 20 |
| 4 | 5 | 50 |
| 5 | 7 | 70 |
| 6 | 10 | 100 |
| 7 | 20 | 200 |

8.3.7 Working Calibration Verification (CCV) Standard (5-ml waters/soils), 50 ug/L each: Use the Linearity Standard #4. For 25-ml waters use Linearity Standard #5: 10-ug/L.

8.3.8 Stock Verification/Spike Standards, 2000 ug/ml each (unless otherwise noted) in methanol. Equivalent products from other vendors may be used.

| GROUP | VENDOR | CAT # | COMPOUNDS |
|------------------|---------|---------|---|
| Non-gases | Supelco | 502111 | See Table in Section 18.0 |
| Gases | Supelco | 48799-U | Bromomethane; Chloroethane; Chloromethane; Dichlorodifluoromethane; Trichlorofluoromethane; Vinyl chloride |
| 8240B Cal Mix 2 | Supelco | 47364 | Acetone, Acetonitrile, Acrylonitrile, 2-Butanone, 2-Hexanone, 4-methyl-2-pentanone |
| MTBE | Restek | 30402 | Methyl-t-butyl ether |
| Carbon Disulfide | Restek | 30258 | Carbon Disulfide |
| 2-CEVE | Restek | 30265 | 2-Chloroethylvinyl ether |
| Vinyl Acetate | Restek | 30216 | Vinyl Acetate |
| Acrolein | Restek | 30499 | Acrolein (10,000-ug/ml) |

8.3.9 Intermediate Verification/Spike Standards: In separate volumetric flasks, prepare the following four standard by diluting the stock calibration verification standards with methanol. Prepare new solutions at a minimum of every 6-months with the exception of the Gases, which must be prepared on a weekly basis.

| STD | VENDOR & CATALOG # | Vol. Stock Verif. Std., ul | Final Vol., ml | Final Conc., ug/ml |
|--------------|-----------------------------------|----------------------------|----------------|--------------------|
| Check Std #1 | Supelco 502111 | 25 | 1.0 | 50 |
| Check Gases | Supelco 48799-U | 25 | 1.0 | 50 |
| Check Std #2 | Supelco 47364 | 25 | 1.0 | 50 |
| Check Std #3 | Restek 30265, 30216, 30402, 30258 | 25 each | 1.0 | 50 |
| | Restek 30499 | 5 | | 50 |

8.3.10 Initial Calibration Verification (ICV) Standard:

For SOLATEK Water Calibrations – In a 25-ml volumetric flask, dilute 25-ul of each intermediate verification/spike standard to the mark with lab pure water. Transfer to a 40-ml VOA vial. (50-ug/L)

For SOLATEK 25-ml Water Calibrations – In a 50-ml volumetric flask dilute 10-ul of each intermediate verification/spike standard to the mark with lab pure water. Transfer to a 40-ml VOA vial. (10-ug/L)

For SOLATEK Soil Calibrations – In a 5.0-ml syringe, add 5.0-ul of each intermediate verification/spike standard to 5.0-ml lab pure water. Transfer to a 40-ml VOA vial for analysis. (50-ug/kg)

For TEKMAR 2016 Calibrations – In a 5.0-ml syringe, add 5.0-ul of each intermediate verification/spike standard as well as 5-ul of the Working I.S./SURR solution to 5.0-ml lab pure water. Transfer to a 25-ml autosampler tube for analysis. (50-ug/L)

8.3.11 Laboratory Control Sample (LCS):

For SOLATEK 5-ml Water Calibrations – In a 25-ml volumetric flask, add 10-ul of each intermediate verification/spike standard and dilute to the mark with lab pure water. Transfer to a 40-ml VOA vial. This prepares a LCS of 20 ug/L.

For SOLATEK 25-ml Water Calibrations – In a 50-ml volumetric flask, add 10-ul of each of the intermediate verification/spike standards and dilute to the mark with lab pure water. This prepares an LCS of 10-ug/L

For SOLATEK Soil Calibrations – In a 5.0-ml syringe, add 5.0-ul of each intermediate verification/spike standard to 5.0-ml of lab pure water. Transfer to a 40-ml VOA vial. This prepares a LCS of 50 ug/kg.

For TEKMAR 2016 Water Analyses – In a 5.0-ml syringe, add 2.0- μ l of each intermediate verification/spike standard as well as 5- μ l of the Working I.S./SURR solution to 5.0-ml lab pure water. Transfer to a 25-ml autosampler tube. This prepares a LCS of 20 μ g/L.

For TEKMAR 2016 Soil Analyses – In a 5.0-ml syringe, add 5.0- μ l of each intermediate verification/spike standard as well as 5- μ l of the Working I.S./SURR solution to 5.0-ml lab pure water. Transfer to a 25-ml autosampler tube. This prepares a LCS of 50 μ g/kg.

8.3.12 Matrix Spike / Matrix Spike Duplicate (MS/MSD):

For SOLATEK 5-ml Water Analyses – In a 25-ml volumetric flask, measure 25-ml of sample and add 25- μ l of each intermediate verification/spike standard to prepare a MS/MSD at 50 μ g/L (Method 8260B); use 10- μ l of each spike standard to prepare a MS/MSD at 20 μ g/L (Method 624).

For SOLATEK 25-ml Water Analysis – In a 50-ml volumetric flask, measure 50-ml of sample, add 10- μ l of each intermediate verification/spike standard to prepare a MS/MSD at 10- μ g/L.

For SOLATEK Soil analyses – To 5g of sample in a VOA vial, add 5.0- μ l each intermediate verification/spike standard to prepare a MS/MSD at 50 μ g/Kg.

For TEKMAR 2016 Water Analyses – To 5.0-ml sample in 5-ml syringe, add 5.0- μ l of each intermediate verification/spike standard as well as 5- μ l of the Working I.S./SURR solution to prepare a MS/MSD at 50 μ g/L (Method 8260B); use 2.0- μ l of each spike standard to prepare a MS/MSD at 20 μ g/L (Method 624). Transfer the spiked sample to a 25-ml autosampler tube for analysis.

For TEKMAR 2016 Soil analyses – In a 5.0-ml syringe, add 5.0- μ l of each intermediate verification/spike standard as well as 5- μ l of the Working I.S./SURR solution to 5.0-ml lab pure water. Transfer the spike to a 25-ml autosampler tube containing 5g sample. This prepares a MS/MSD at 50 μ g/Kg.

8.3.13 Method Blank (MBLK): Lab pure water is used for the MBLK associated with the analysis of water and methanol extracted samples. The MBLK must be spiked with 5- μ l of the Working I.S./SURR solution.

GASOLINE STANDARDS

8.3.14 Stock Gasoline Calibration Standard: Restek catalog #030206 contains unleaded gasoline at 50,000 μ g/ml in methanol. Equivalent products from other vendors may be used.

8.3.15 Working Gasoline Calibration Standard, 2500 μ g/ml: In a 1-ml volumetric flask, dilute 50- μ l of the stock gasoline calibration standard to the mark with methanol.

- 8.3.16 Working Calibration Standards (Linearity, ICAL): A minimum of 5 standards need to be prepared and analyzed.

Water 5-ml Linearity SOLATEK - In separate 25-ml volumetric flasks, prepare the following dilutions of the working Gasoline Calibration Standard with lab pure water. Transfer the prepared standards to 40-ml VOA vials.

| Linearity Standard # | Vol. Working Gas Cal. Std, ul | Final Conc. ug/L |
|----------------------|-------------------------------|------------------|
| 1 | 2 | 200 |
| 2 | 5 | 500 |
| 3 | 10 | 1000 |
| 4 | 25 | 2500 |
| 5 | 50 | 5000 |
| 6 | 60 | 6000 |

Soil Linearity SOLATEK – Using a 5.0-ml syringe, individually prepare the following dilutions of the working Gasoline Calibration Standard with 5.0-ml lab pure water. Transfer the prepared standards to 40-ml VOA vials for analysis.

| Linearity Standard # | Vol. Working Gas Cal. Std, ul | Final Conc. ug/Kg |
|----------------------|-------------------------------|-------------------|
| 1 | 1 | 500 |
| 2 | 2 | 1000 |
| 3 | 4 | 2000 |
| 4 | 5 | 2500 |
| 5 | 10 | 5000 |
| 6 | 12 | 6000 |

Water/Soil Linearity TEKMAR 2016 – Using a 5.0-ml syringe, individually prepare the following dilutions of the working Gasoline Calibration Standard with 5.0-ml lab pure water and 5- μ L of the working ISTD/SURR solution. Transfer the prepared standards to separate ALS tubes for analysis.

| Linearity Standard # | Vol. Working Gas Cal. Std, ul | Final Conc. ug/L |
|----------------------|--------------------------------|------------------|
| 1 | 2 (to a final volume of 25-ml) | 200 |
| 2 | 1 | 500 |
| 3 | 2 | 1000 |
| 4 | 4 | 2000 |
| 5 | 5 | 2500 |
| 6 | 10 | 5000 |

- 8.3.17 Stock Gasoline Verification Standard: Accustandard catalog #GA-001-40X contains unleaded gasoline at 20,000 ug/ml in methanol. Equivalent products from other vendors may be used.
- 8.3.18 Working Gasoline Verification Standard, 5000 ug/ml: In a 1-ml volumetric flask, dilute 250- μ L of the stock gasoline verification standard to the mark with methanol.

8.3.19 Initial Calibration Verification (ICV) Standard, 1000 ug/L:

For SOLATEK 5-ml Water Calibrations – In a 25-ml volumetric flask, dilute 5- μ l of the working gasoline verification standard to the mark with lab pure water. Transfer to a 40-ml VOA vial.

For SOLATEK Soil Calibrations – In a 5.0-ml syringe, add 1.0- μ l of the working gasoline verification standard to 5.0-ml lab pure water. Transfer to a 40-ml VOA vial for analysis.

For TEKMAR 2016 Calibrations – In a 5.0-ml syringe, add 1.0- μ l of the working gasoline verification standard as well as 5- μ l of the Working I.S./SURR solution to 5.0-ml lab pure water. Transfer to a 25-ml autosampler tube for analysis.

9.0 SAMPLE COLLECTION, PRESERVATION, AND HOLDING TIMES

- 9.1 The client or other trained personnel collect samples. Samples received at the laboratory are considered representative unless otherwise noted. Water samples should be dechlorinated prior to acidification. This is a sample collection activity that must be performed in the field.
- 9.2 Water samples should be collected in a 40-ml glass vial with a Teflon lined lid (VOA vial) leaving zero headspace. Preservation consists of HCl to pH < 2 and storage in the range of 0.1-6°C. Samples are stored in the dedicated VOA coolers and analysis must be performed within the maximum allowable hold time of 14-days from collection for acid preserved water samples.
- 9.2.1 The analyst must measure the pH after sample analysis has been performed. The result of these checks is noted on the injection log. Any sample not meeting the requirement of pH < 2 must be qualified in a Case Narrative to the client.
- 9.2.2 NOTE: 2-Chloroethyl vinyl ether and Acrolein will degrade with acidification.
- 9.2.3 Based on the data reported in section 9.3 of Method 624, unpreserved samples must be analyzed within 7-days from collection.
- 9.3 Soil samples should be collected according to the specifications described below. The collection steps include the preservation technique.
- 9.3.1 Method 5035: All samples received for analysis in strict accordance with Method 5035 must be chemically preserved with sodium bisulfate (NaHSO_4) or methanol and retained at 4°C until analysis. The maximum allowable hold time for bisulfate preserved samples is 14-days from collection.

- 9.3.1.1 Method 5035-IN: Samples received for programs that allow freezing as an alternative to the chemical preservation must be frozen at $-12 \pm 2^{\circ}\text{C}$ within 24-hours of receipt at the lab to inhibit biodegradation. This thermal preservation technique provides for a maximum hold time of 7-days from collection. Without the thermal preservation (i.e. freezing), samples must be analyzed within 48-hours of collection. Samples stored at 4°C are retained in the dedicated VOA cooler and samples stored at -12°C are retained in the organics freezer.
- 9.3.1.2 Low level samples. At the time of sample collection, 5g of sample are placed into a pre-weighed VOA vial. The vials are sealed, chilled to 4°C , and must be shipped by the client for receipt within 48-hours of collection. Upon receipt, the vials are re-weighed to obtain the weight of sample, which is recorded in the VOA Soil Preparation Logbook (copy attached).
- 9.3.1.3 Medium/High level samples. At the time of sample collection, 5g of sample are placed into a pre-weighed VOA vial containing 5-ml of purge-and-trap grade methanol. The vials are sealed, chilled to 4°C , and shipped to the laboratory. Upon receipt, the vials are re-weighed to obtain the weight of sample, which is recorded in the VOA Soil Preparation Logbook.
- 9.3.1.4 Field-unpreserved samples. The sample is collected in an air-tight storage container. These devices collect the sample in a storage chamber that may be sealed leaving zero headspace. Acceptable examples include Encore samplers or other coring devices. The samples are then chilled to 4°C and shipped to the laboratory for receipt within 48-hours of collection.
- 9.3.2 Method 5030: All soil samples received for analysis in according to Method 5030 are unpreserved. The sample is collected in container with minimal headspace and unopened until analysis. Acceptable examples include glass jars with Teflon lined lids. The samples are then chilled to 4°C and shipped to the laboratory. These samples have a maximum hold time of 14-days from collection.

10.0 QUALITY CONTROL

- 10.1 An *Initial Demonstration of Capability* study must be performed prior to the initial analysis for each analyst and whenever substantial change has occurred in the procedure or instrument. Refer to the Capability and Detection Limit Studies SOP for details.
- 10.2 A *Method Detection Limit* study must be performed for each new procedure, annually thereafter, and whenever a change in instrument occurs. Refer to the Capability and Detection Limit Studies SOP for details.

- 10.3 A *BFB Tune Check* must be performed at the beginning of each 12-hour sequence. A 1- μ l (50-ng) injection of the working tune solution (which contains 4-Bromofluorobenzene) must meet the following ion abundance criteria. Alternately, BFB may be evaluated from the purged analysis of the Internal Standard/Surrogate standard. If the criteria are not met, reanalyze. An acceptable tune must be obtained before samples can be analyzed.

BFB (4-BROMOFLUOROBENZENE)
KEY IONS and ION ABUNDANCE CRITERIA
METHODS 624 / 8260B

| Mass | Ion Abundance Criteria |
|------|------------------------------------|
| 50 | 15 – 40% of mass 95 |
| 75 | 30 – 60 % of mass 95 |
| 95 | Base peak, 100% relative abundance |
| 96 | 5 – 9 % of mass 95 |
| 173 | < 2 % of mass 174 |
| 174 | > 50 % of mass 95 |
| 175 | 5 – 9 % of mass 174 |
| 176 | > 95 but < 101 % of mass 174 |
| 177 | 5 – 9 % of mass 176 |

- 10.4 *Internal Standards* (I.S.) must be added to all standards, QC samples and environmental samples.
- 10.4.1 Acceptance criteria for CCV standards are RT \pm 30 seconds from that in the midpoint level standard of the most recent initial calibration (ICAL) and area counts within the range of -50 to +100% of those in the same (concentration level) linearity standard. These criteria are evaluated by the data system and printed on the Continuing Calibration Report. Failures in the CCV are automatically flagged on this report.
- 10.4.1.1 If the acceptance criteria are not met for the CCV, perform instrument maintenance if warranted, and then reanalyze the CCV or perform a new initial calibration.
- 10.4.2 There are no absolute acceptance criteria requirements for samples. The RT and Area for samples should be compared to that of the CCV for that day and assessed against the criteria for the CCV (i.e. RT \pm 30 seconds and area counts within the range of -50 to +100%). These data are used as guidance in evaluating the response for samples.

- 10.4.2.1 If the acceptance criteria are not met for a sample, evaluate the chromatogram for obvious matrix effects/interferences. If interferences are evident, the sample should be diluted and reanalyzed. If the internal standard response for the sample is below 25% the analyst should consult with the Unit Supervisor and use discretion on whether to report, dilute and reanalyze, or re-extract and analyze the sample. This discretion is dependent on the experience of the analyst and their Supervisor and factors such as a consistent response for the I.S. should be considered. If reanalysis yields poor internal standard response, or if reanalysis is performed beyond the hold time, both sets of data may be reported to the client and a Case Narrative written.
- 10.4.2.2 For samples having a known matrix effect it is allowable to forego reanalysis due to "failing" I.S. response. These data, however, must be reported to the client with a Case Narrative informing them of the suspect data.
- 10.5 *Surrogate* (SURR) compounds must be added to all quality control samples, blanks, and samples.
- 10.5.1 Acceptance criteria are listed in the appropriate test code in LIMS.
- 10.5.2 Surrogate standards that fail to meet the acceptance criteria are automatically flagged in LIMS with an "S" qualifier.
- 10.5.3 The reporting of data associated with failing surrogate standards must be documented with a CAR form.
- 10.5.4 If the acceptance criteria are not met, reanalyze the sample. If insufficient sample is available for reanalysis, report the original result with a Case Narrative to the client. If reanalysis fails to meet the acceptance criteria the original results should be reported with a Case Narrative to the client. If reanalysis does meet the acceptance criteria but was performed beyond the maximum hold time both sets of results should be reported to the client with an appropriate Case Narrative.
- 10.6 An *Initial Calibration Verification* (ICV) standard must be analyzed after the initial linearity (ICAL) and before the analysis of samples can begin. This is the analysis of a second source standard.
- 10.6.1 Acceptance criteria are listed in the appropriate test code in LIMS. If the acceptance criteria are not met, reanalyze. If reanalysis fails to meet the acceptance criteria, stop analysis and recalibrate or report data with an appropriate qualifier.
- 10.6.2 ICV standards that fail to meet the acceptance criteria are automatically flagged in LIMS with an "S" qualifier.
- 10.6.3 The reporting of data associated with a failed ICV must be documented with a CAR form. If the failure is considered to have a significant affect on the data, client notification is required using the Case Narrative of the report.

- 10.6.4 Samples associated with an ICV that fails with a positive bias can be reported without narration if the sample concentration is below the reporting limit.
- 10.7 A *Continuing Calibration Verification* (CCV) standard is a calibration source standard and must be analyzed at the beginning of each analytical sequence following an acceptable instrument tune. The 12-hour analytical sequence begins with the injection of BFB, continues through the analysis of the CCV, samples and QC samples.
- 10.7.1 Acceptance criteria for Method 8260B are the RF criteria (see below) for SPCCs and a RF for CCCs $\leq 20\%$ difference or 20% drift from the initial calibration. If the CCCs are not included in the list of analytes for a specific project, then all analytes must meet the 20% difference or drift criteria. An "Evaluate Continuing Calibration Report" is generated to evaluate this criteria. If any targets are quantitated using a least squares regression equation instead of the average response factor, this report must be generated using both relative response factor and calculated concentrations.

SPCC Criteria

≥ 0.300 for Chlorobenzene and 1,1,2,2-Tetrachloroethane
 ≥ 0.100 for Chloromethane, and 1,1-Dichloroethane, and Bromoform
For 25-ml water analyses, the SPCC for Bromoform must be ≥ 0.073 .

CCC Compounds

1,1-Dichloroethene, 1,2-Dichloropropane, Chloroform, Ethylbenzene, Toluene and Vinyl chloride

- 10.7.2 Method 624 does not require the analysis of a CCV standard but, rather, evaluates the continuing integrity of the calibration using the laboratory control sample. If acceptable criteria are not met, reanalyze. If reanalysis fails to meet the acceptance criteria, recalibrate.
- 10.7.3 The reporting of data associated with a failed CCV must be documented with a CAR form. If the failure is considered to have a significant effect on the data, client notification is required using the Case Narrative of the report.
- 10.7.4 Samples associated with a CCV that fails with a positive bias can be reported without narration if the sample concentration is below the reporting limit.

- 10.8 A *Method Blank* (MBLK) must be prepared and analyzed at the beginning of each analytical sequence, batch of maximum 20 samples, or at a minimum of one per day, whichever is more frequent.
- 10.8.1 The acceptance criteria are $< \text{PQL}$. If the acceptance criteria are not met, the MBLK is reanalyzed. If samples are analyzed against a MBLK with targets $> \text{PQL}$, they may be reported as long as there are no targets detected in the sample $> \text{PQL}$ for the outliers in the MBLK. Otherwise, they are reanalyzed. Samples for compliance with our Wisconsin DNR certification must be evaluated down to the current MDL and corrective action taken if the blank exceeds the routine PQL.
- 10.8.2 MBLKs that fail to meet the acceptance criteria cause the sample results to be automatically flagged in LIMS with a "B" qualifier. MBLKs that are below the reporting limit but above the MDL are flagged in LIMS with a "b" qualifier. "b" flagged data is considered as meeting the acceptance criteria.
- 10.8.3 The reporting of data associated with a failed control sample must be documented with a CAR form. If the failure is considered to have a significant effect on the data, client notification is required using the Case Narrative of the report.
- 10.8.4 Samples associated with a MBLK that fails with positive bias can be reported without narration if the sample concentration is $< \text{PQL}$ or greater than 10 times the blank contamination.
- 10.9 A *Laboratory Control Sample* is a second source standard that must be prepared and analyzed at the beginning of each analytical sequence, batch of maximum 20 samples, or at a minimum of one per day, whichever is more frequent. This standard serves as the daily calibration check for EPA Method 624.
- 10.9.1 Acceptance criteria are listed in the appropriate test code in LIMS. If the acceptance criteria are not met, reanalyze. If reanalysis fails to meet the acceptance criteria, re-extract and analyze the associated samples (alternatively, report data with an appropriate qualifier).
- 10.9.2 If insufficient sample is available for reanalysis the original result should be reported with a Case Narrative notifying the client of the quality control failure. If the hold time has expired and an acceptable re-analysis performed, both sets of data should be reported and the appropriate result flagged with an "H" qualifier as defined in the LIMS.
- 10.9.3 LCSs that fail to meet the acceptance criteria are automatically flagged in LIMS with an "S" qualifier.
- 10.9.4 The reporting of data associated with a failed LCS must be documented with a CAR form. If the failure is considered to have a significant effect on the data, client notification is required using the Case Narrative of the report.

- 10.9.5 Samples associated with a LCS that fails with positive bias can be reported without narration if the sample concentration is below the reporting limit.
- 10.10A *Matrix Spike and Matrix Spike Duplicate* sample must be prepared and analyzed with each batch of maximum 20 samples per matrix and at a minimum of one per day.
- 10.10.1 Acceptance criteria are listed in the appropriate test code in LIMS. (Note: the accuracy criteria have been met provided at least either the MS or MSD meet the %R criteria.) If the accuracy criteria are not met in the MS or MSD, and the LCS is in control, assume matrix interference and report the results with an appropriate Case Narrative. If the precision criteria are not met, report the results with an appropriate Case Narrative.
- 10.10.2 MS/MSD's that fail to meet the accuracy criteria are automatically flagged in LIMS with an "S" qualifier. MSD's that fail to meet the precision criteria are automatically flagged in LIMS with an "R" qualifier.
- 10.10.3 The reporting of data associated with a failed MS/MSD must be documented with a CAR form. If the failure is considered to have a significant effect on the data, client notification is required using the Case Narrative of the report.
- 10.10.4 Samples associated with a MS/MSD that fails the accuracy criteria with positive bias can be reported without narration if the sample concentration is below the reporting limit.
- 10.10.5 If the concentration measured in the sample is greater than 4-times the concentration of the spike, the spike amount used is insufficient and the MS/MSD not applicable.
- 10.10.6 An LCS and and LCSD are run if there is insufficient sample available for an MS/MSD to be run, or if the samples analyzed in the sequence have severe matrix issues that in turn make an MS/MSD analysis inadvisable.
- 10.10.7 The list of spiked compounds is the same as that for the LCS.

11.0 CALIBRATION AND STANDARDIZATION

Calibration data is documented and retained using the printouts from the instrument software and the ICAL Review Checklist (copy attached). Analytical data must be maintained in accordance with the document control requirements in the Quality Assurance Plan as well as the Document Control SOP.

- 11.1 Perform the required preventive maintenance. Documentation is maintained in the PM Logbook for each instrument.

- 11.2 A new Initial Calibration (linearity; ICAL) is required as listed below.
- CCV failure that is uncorrectable through appropriate instrument maintenance
 - Source is cleaned
 - New column is installed
 - Replacement of electron multiplier, entrance lens, draw-out lens, repeller, ion source chamber or injection port.
- 11.3 Instrument conditions and method settings are listed in section 18.0.
- 11.4 Purging and transfer to the Concentrator is initiated through the Solatek autosampler or the Tekmar 2016 ALS. The GC/MS is enabled by using the Method – Run pull down menus on the instrument top window on the computer. When using the Solatek, the internal standards and surrogates are automatically added to each standard/sample. When using the Tekmar 2016, these are manually added to each analysis by adding 5.0- μ l of the Working I.S./SURR Solution to the 5.0-ml of sample in the syringe.
- 11.5 Set up a sequence in the Sample Table Log in *Sequence* of the main menu. Enter the sequence as it is to be run, including the applicable analysis method. A calibration sequence must start with a BFB tune followed by the calibration standards then an ICV standard.
- 11.6 Click on *Position and Run*.
- 11.7 Generate the corresponding Quantitation Reports, BFB “Tune” Report and Response Factor Report.
- 11.7.1 On the BFB Report, evaluate the Pass/Fail column. An acceptable tune will Pass each of the criteria. Calibration can not continue with a failed tune. Details of Tune evaluation include:
- Initial evaluation should use the auto-evaluate feature of the software. This feature evaluates the apex of the peak as well as the scans immediately on either side of the apex and automatically background corrects using a single scan no more than 20 scans prior to the beginning of the peak.
 - If the auto-evaluation fails, select a different single spectrum or the average of several spectra. If these approaches fail, add additional spectra and re-evaluate. Guidance for these options is in the Environmental Data Analysis User’s Guide for the Enviroquant software.
 - Background correction must use a single scan acquired no more than 20 scans prior to the beginning of the BFB peak. No part of the BFB peak may be subtracted.
 - Appropriate corrective action (e.g. instrument maintenance, new standard, re-calibration, etc) must be performed if an instrument continues to fail the tune criteria.
- 11.7.2 The software calculates RF values for each standard as well as the average RF of all analytes.
- 11.7.3 On the Response Factor Report, circle the %RSD values of each CCC and place a check mark next to the average RF of each SPCC.

11.8 The acceptance criteria for an acceptable ICAL are as follows:

11.8.1 For Method 8260B

- Minimum of 5 levels
- Average RF for the SPCCs must meet the following:
 - ≥ 0.300 for Chlorobenzene and 1,1,2,2-Tetrachloroethane
 - ≥ 0.100 for Chloromethane, and 1,1-Dichloroethane, and Bromoform (for 25-ml waters, Bromoform ≥ 0.073).
- %RSD for the CCCs must be $< 30\%$
- Where these criteria are met and the %RSD of all individual compounds is ≤ 15 , the calibration is considered linear and the average RF is used for concentration calculations.

11.8.1.1 If the %RSD $> 15\%$ for any analyte, the following may be used

- Narrow the calibration range by replacing one or more of the calibration standards with standards that cover a narrower range.
- The concentration of the low standard must be at or below the PQL for every target analyte.
- Employ the least squares regression equation. The origin (0.0) may not be included as an extra calibration point, but Method 8000C allows the forcing of the regression curve through zero.
- Linear (first order)-requires a minimum of 5 calibration standard levels.
- Non-Linear (second order) quadratic- requires a minimum of 6 calibration standard levels.
- Non-Linear (third order) polynomial-requires a minimum of 7 calibration standard levels.

Note: Method 8000C allows for the evaluation of linearity using either the correlation coefficient (r) or the coefficient of determination (r^2). Regardless of the statistical model used by the software (i.e. r or r^2), the acceptance criteria are greater than or equal to 0.99.

11.8.1.2 All ICAL standards need to be analyzed within the same tune.

11.8.2 For Method 624

- Minimum of 3 levels
- average RF for each compound must have %RSD < 35

Where these criteria are met, the calibration is considered linear and the average RF is used for concentration calculations.

11.8.2.1 If the %RSD is $> 35\%$ for any analyte, the same criteria as for Method 8260B (Section 11.8.1.1) above applies.

11.8.2.2 All ICAL standards need to be analyzed within the same tune.

11.9 If the linearity requirements are not met, take appropriate corrective actions and recalibrate. If the acceptance criteria are met, rename the method "WTRmmdd" or "SOILmmdd", (or similar) where mmdd designates the month and date of the new linearity.

11.9.1 Dropping levels from the calibration curve is allowable under the following conditions.

- Points may not be dropped solely to meet the acceptance criteria. There must be a justifiable reason for excluding a given standard.
- Individual analytes may be eliminated from the low or high points.
- Dropping a mid-level standard requires that all analytes be eliminated from that level.
- The required minimum number of calibrated levels remains (5 levels for 8260B, 3 levels for 624).
- If the low-level standard is removed from the curve, the PQL must be adjusted accordingly.

11.10 Analyze an ICV standard. The acceptance criteria must be met before continuing with sample analysis. Analysis of environmental samples cannot proceed without the generation of an acceptable linearity and an acceptable initial verification.

12.0 PROCEDURE

Preparation data is documented and retained using the printouts from the Injection Log and the VOA Soil Preparation Log (copy attached). This data must be maintained in accordance with the document control requirements in the Quality Assurance Plan as well as the Document Control SOP.

12.1 SAMPLE PREPARATION – Water Samples Using the Solatek Autosampler

12.1.1 Load the VOA vials onto the autosampler. The purge and trap unit removes either a 5-ml or 25-ml aliquot of sample and adds 5ul of the internal standard/surrogate solution for analysis. Dilutions up to 1:10 can be programmed and prepared by the autosampler, but are typically prepared in volumetric flasks and transferred to VOA vials for analysis. Higher dilutions, if necessary, must be prepared in volumetric flasks and transferred to VOA vials for analysis.

12.2 SAMPLE PREPARATION – Soil Samples Using the Solatek Autosampler

12.2.1 For **low-level samples collected directly into VOA vials** - Load the VOA vials onto the autosampler. The vials contain a stir bar and 5g of sample and the NaHSO₄ preservative (if used). The purge and trap unit adds 5ul of the internal standard/surrogate solution and 15-ml lab pure water for analysis.

12.2.2 For **low-level samples collected with coring devices** – Upon receipt in the lab, allow the unopened storage device to reach ambient temperature, extrude the sample into a tared VOA vial containing a stir bar and record the sample weight. Record the weight in the logbook and place the vial on the autosampler. The

purge and trap unit adds 5ul of the internal standard/surrogate solution and 15-ml lab pure water for analysis.

- 12.2.3 For **medium/high level samples collected with methanol preservation** – Transfer 1-ml of the extract to a 50-ml volumetric flask partially filled with lab pure water. (If the sample is expected to be very high in concentration use a lesser sample volume than 1-ml as the maximum sample:water ratio is 100-ul sample per 5.0-ml water.) Dilute the sample to the mark with lab pure water, transfer the solution to a VOA vial and load the vial onto the autosampler. Analyze the sample as a water sample (i.e. non-heated purge). The purge and trap unit adds 5ul of the internal standard/surrogate solution for analysis.

12.3 SAMPLE PREPARATION – Water Samples Using the Tekmar 2016 ALS

- 12.3.1 Remove 5.0-ml of sample from the VOA vial using a gas-tight syringe. If dilutions are necessary, use lesser sample diluted to a final volume of 5.0-ml in the syringe.
- 12.3.2 Add 5-ul of the Working I.S./SURR Solution to the syringe.
- 12.3.3 Using the sample valve on the ALS, transfer the sample into a 25-ml autosampler tube.

12.4 SAMPLE PREPARATION – Soil Samples Using the Tekmar 2016 ALS

- 12.4.1 For **low-level samples** – Using the top loading balance, transfer 5g of sample into a tared autosampler tube and connect the tube onto the ALS. If the concentrations are expected to be high, use 1g or 0.5g of sample.
- 12.4.2 Add 5.0-ml of lab pure water in a gas-tight syringe then add 5.0-ul of the Working I.S./SURR Solution to the syringe.
- 12.4.3 Using the sample valve on the ALS, transfer the water/I.S./SURR into the autosampler tube.
- 12.4.4 For **medium/high level samples** – Using a top loading balance, transfer 5g of sample into a tared VOA vial.
- 12.4.5 Using a disposable glass pipette, add 5-ml methanol then cap and shake the vial for 2-minutes.
- 12.4.6 Transfer approximately 2-ml of the extract into a vial for storage/retain. This extract may be stored in the dark at 4°C until analysis.
- 12.4.7 Add 5.0-ml of lab pure water in a gas-tight syringe then add 5.0-ul of the Working I.S./SURR Solution and 100-ul of the extract to the syringe. (Note: 200-ul of methanol extract may be used when absolutely necessary, but 100-ul of extract is the normal maximum amount.)

12.4.8 Using the sample valve on the ALS, transfer the sample into the autosampler tube.

12.5 ANALYSIS

Analytical data is documented and retained using the printouts from the instrument software. Analytical data must be maintained in accordance with the document control requirements in the Quality Assurance Plan as well as the Document Control SOP.

12.5.1 Perform the required preventive maintenance. Documentation is maintained in the PM Logbook for each instrument.

12.5.2 When using the Solatek, the internal standards and surrogates are automatically added to each standard/sample. When using the Tekmar 2016, these are manually added to each analysis by adding 5.0-ul of the Working I.S./SURR Solution to the 5.0-ml of sample in the syringe.

12.5.3 The ICAL must be verified each "analytical sequence" prior to sample analysis by analyzing a calibration standard (CCV) at/near the mid-point of the curve (typically 50 ug/L). See the Quality Control section for the acceptance criteria. The 12-hour analytical sequence begins with the injection of BFB, continues through the analysis of the CCV, environmental samples and QC samples.

12.5.4 Set up a sequence in the Sample Table Log under *Sequence* of the main menu. Enter the sequence as it is to be run, including the applicable analysis method.

12.5.5 Click on *Position and Run*.

12.5.6 After each sample has run, evaluate the chromatogram and quantitate against the current initial linearity (ICAL).

12.5.7 Generate the corresponding Quantitation Reports, BFB "Tune" Report and Evaluate Continuing Calibration Report.

12.5.7.1 On the BFB Report, evaluate the Pass/Fail column. An acceptable tune will Pass each of the criteria. Analysis can not continue with a failed tune.

12.5.7.2 On the Evaluate Continuing Calibration Report, circle the %Dev values of each CCC as well as the Area of each internal standard, and place a check mark next to the CCRF of each SPCC.

12.5.8 Analysis of environmental samples cannot proceed without an acceptable continuing verification.

12.5.9 If the concentration of any target compound in a sample exceeds the initial calibration range, a new aliquot must be diluted and analyzed.

13.0 **CALCULATIONS AND DATA HANDLING**

Analytical data must be maintained in accordance with the document control requirements in the Quality Assurance Plan as well as the Document Control SOP.

13.1 The HP software will print out all target analytes detected at a concentration at or above the MDL. The analyst must verify every target detected by evaluating the retention time and fit against the applicable CCV standard. These evaluations are accomplished by evaluating the characteristic ions through QEDIT as well as comparing the reference spectra. The Qvalue should be considered, however, the individual spectra must be evaluated in order to confidently identify a given analyte.

13.1.1 The data system software evaluates the retention time of each peak as well as a comparison of the characteristic ions to identify the compounds present. The characteristic ions of the reference spectrum are the three ions of greatest intensity (or any ions having a relative intensity greater than 30% if less than three ions are present). The following criteria are used for qualitative identification. An analyte may be confirmed as a proper identification only if these criteria are met.

- The characteristic ions of a compound must have a relative retention time of ± 0.06 minutes of the standard ($RT \pm 30$ sec for Method 624).
- The relative intensities of the characteristic ions are within 20% of those ions in the reference spectrum (30% for Method 8260B). Example: for an ion having an abundance of 50% in the reference spectrum, the corresponding abundance in a sample can range from 30% to 70% (Method 624) or 20% to 80% (Method 8260B) as appropriate.
- Structural isomers that produce very similar mass spectra should be identified as individual isomers if they have sufficiently different retention times, otherwise, the isomers having a resolution of $< 25\%$ are identified as isomeric pairs.

13.1.2 Minimum levels for each analyte are established in the software at a concentration equivalent to the current on-column MDL value.

13.1.3 Manual peak integrations as well as the addition/deletion of analytes must be documented on the Quantitation Report. See the Manual Integration of Chromatographic Peaks SOP for details. When manual integrations are required, a printout of the original integration and the manual integration is obtained and filed with the sample quantitation report.

13.2 Response factors (RF) are calculated as follows:

$$RF = (A_x)(C_{IS}) / (A_{IS})(C_x)$$

Where: A_x = Area of characteristic ion for compound being measured
 A_{IS} = Area of characteristic ion for compound being measured
 C_{IS} = Concentration of the specific internal standard
 C_x = Concentration of the compound being measured

- 13.3 The software calculates the sample concentration as follows:

INTERNAL STANDARD CALIBRATION

$$\text{Conc.} = \frac{(A_x)(I_s)(V_t)(DF)}{(A_{IS})(RF)(V_o)(V_i)}$$

Where: A_x = Area of characteristic ion for compound being measured

I_s = Amount of internal standard injected (ng)

V_t = Volume of total extract, taking into account dilution

A_{IS} = Area of characteristic ion for the internal standard

RF = Initial average response factor for compound being measured

V_o = Volume of water extracted (L), or mass of soil extracted (kg)

V_i = Volume of extract injected (ul)

DF = dilution factor

TOTAL ION CHROMATOGRAPHY

Integration for "gasoline" is performed using an HP chemstation macro. Integration includes the sum of all ions eluting between 2-methyl pentane and 1,2,4-Trichlorobenzene that are observed in the CCV or calibration standard, as appropriate, less the ions attributable to all internal standards and surrogates. The sum of these peaks is reported as "Gasoline Range Organics".

$$\text{Conc., ppb} = \frac{(A_x)(C_{std})(DF)}{(A_{std})(V_o)}$$

Where: A_x = Total ion area of sample

C_{std} = Concentration of standard injected (ng)

DF = dilution factor

A_{std} = Total ion area of standard

V_o = Volume of water purged (ml) or mass of soil purged (g)

NOTE: $(A_x)(C_{std}) / (A_{std})(RF)$ is calculated by the computer.

- 13.4 When the analyte curve fit is quadratic or quad forced and the analyte response is beyond the linear range for the ICAL. What would be considered an "E" value result may quantitate as a negative result. The report will print out as "Below Cal". Care must be taken to ensure that this analyte is not missed and reported as a non-detect. Consultation with supervisor or QA director is advised.
- 13.5 After review, enter the final results into the LIMS system. Results flagged by the LIMS with an "E" qualifier are above the linear range of the instrument. There is less certainty in this data and, if sufficient extract and holding time are available, should be reanalyzed at an appropriate dilution. Details on the procedure for entering analytical data are in the Data Entry SOP.

- 13.6 The LIMS calculates the dry-weight concentration for solid samples as follows:

$$\text{Conc. Dry} = \frac{(\text{wet weight conc.})}{(100 - \% \text{ Moisture})}$$

14.0 METHOD PERFORMANCE

- 14.1 Initial Demonstration of Capability study data, Method Detection Limit study data and Performance Testing study data are maintained and available from the QA office.

15.0 POLLUTION PREVENTION

- 15.1 The quantity of chemicals purchased should be based on expected usage during their shelf life and the disposal cost of unused material.
- 15.2 Prepare the minimum amount of reagent and standard necessary.

16.0 WASTE MANAGEMENT

- 16.1 Refer to the Sample Disposal SOP for guidance on the disposal of any resulting residue, digestate, distillate, extract or standard.

17.0 REFERENCES

- 17.1 USEPA Method 624
- 17.2 SW-846 Method 5030B
- 17.3 SW-846 Method 5035
- 17.4 SW-846 Method 8260B
- 17.5 SW-846 Method 8000C
- 17.6 Indiana Dept of Environmental Management Non-rule Policy Document for Indiana Modified Method 5035, adopted April 20, 2000
- 17.7 Environmental Data Analysis User's Guide, HP G1032C Enviroquant Target Compound Software, 1992
- 17.8 Microbac Laboratories Quality Assurance Plan, current revision, all sections

18.0 TABLES, FORMS, CHECKLISTS, AND OTHER ATTACHMENTS

Non-gases Calibration Standard analyte list (1 page)
VOA Soil Preparation Log (1 page)
ICAL Review Checklist (1 page)
Instrument Conditions and Method Settings (3 pages)
SOP Revision Form (1 page)

Uncontrolled Document

| Calibration Standard Compounds | |
|--|--|
| Non-gases Accustandard M-502A-R2- 10X | Benzene; Bromobenzene; cis-1,3-Dichloropropene; Bromodichloromethane; Bromoform; n-Butylbenzene; sec-Butylbenzene; tert-Butylbenzene; Carbon tetrachloride; Chlorobenzene; Chloroform; 2-Chlorotoluene; 4-Chlorotoluene; Dibromochloromethane; 1,2-Dibromoethane; Dibromomethane; 1,2-Dibromo-3-chloropropane; 1,2-Dichlorobenzene; 1,3-Dichlorobenzene; 1,4-Dichlorobenzene; 1,1-Dichloroethane; 1,2-Dichloroethane; 1,1-Dichloroethene; cis-1,2-Dichloroethene; ; trans-1,2-Dichloroethene; 1,2-Dichloropropane; 1,3-Dichloropropane; 2,2-Dichloropropane; 1,1-Dichloropropene; trans-1,3-Dichloropropene; Ethylbenzene; Hexachlorobutadiene; Isopropylbenzene; p-Isopropyltoluene; Methylene chloride; Naphthalene; n-Propylbenzene; Styrene; 1,1,1,2-Tetrachloroethane; 1,1,2,2-Tetrachloroethane; Tetrachloroethene; Toluene; 1,1,1-Trichloroethane; 1,1,2-Trichloroethane; Trichloroethene; 1,2,3-Trichlorobenzene; 1,2,4-Trichlorobenzene; 1,2,3-Trichloropropane; 1,2,4-Trimethylbenzene; 1,3,5-Trimethylbenzene; o-Xylene; m-Xylene; p-Xylene |

| Miscellaneous Standard Compounds | |
|--|--|
| Non-gases Accustandard S-15177 | 1,3-Butadiene; Hexane; Methyl methacrylate; Methyl acrylate; Ethyl acrylate; Butyl acrylate; Cyclohexane; 2-Methylnaphthalene; Dicyclopentadiene |

| ICV/LCS/MS/MSD (2 nd source verification) Standard Compounds | |
|---|---|
| Non-gases Supelco 502111 | Benzene; Bromobenzene; cis-1,3-Dichloropropene; Bromodichloromethane; Bromoform; n-Butylbenzene; sec-Butylbenzene; tert-Butylbenzene; Carbon tetrachloride; Chlorobenzene; Chloroform; 2-Chlorotoluene; 4-Chlorotoluene; Dibromochloromethane; 1,2-Dibromoethane; Dibromomethane; 1,2-Dibromo-3-chloropropane; 1,2-Dichlorobenzene; 1,3-Dichlorobenzene; 1,4-Dichlorobenzene; 1,1-Dichloroethane; 1,2-Dichloroethane; 1,1-Dichloroethene; cis-1,2-Dichloroethene; ; trans-1,2-Dichloroethene; 1,2-Dichloropropane; 1,3-Dichloropropane; 2,2-Dichloropropane; 1,1-Dichloropropene; trans-1,3-Dichloropropene; Ethylbenzene; Isopropylbenzene; p-Isopropyltoluene; Methylene chloride; Naphthalene; n-Propylbenzene; Styrene; 1,1,1,2-Tetrachloroethane; 1,1,2,2-Tetrachloroethane; Tetrachloroethene; Toluene; 1,1,1-Trichloroethane; 1,1,2-Trichloroethane; Trichloroethene; 1,2,3-Trichlorobenzene; 1,2,4-Trichlorobenzene; 1,2,3-Trichloropropane; 1,2,4-Trimethylbenzene; 1,3,5-Trimethylbenzene; o-Xylene; m-Xylene; p-Xylene |

Microbac Laboratories - Chicagoland Division

VOA Soil Preparation

[illegible]

* U (unpreserved), SB (sodium bisulfate)
M (methanol), E (Encore or similar)

*** PFac = 5/SAMP WT

revision: a 4-04

Page 1 of 1

Microbac Laboratories
ICAL Review Checklist – VOA

Instrument ID: _____

Analyst: _____

Linearity Date: _____

Quant. Method Name: _____

1st Level Technical Review

| Review Element | Evaluation | Comments (use this space as needed) |
|---|--|-------------------------------------|
| SW-846 Method 8260B/8000C | | |
| %RSD of CCCs <30 and circled? | <input type="checkbox"/> Yes <input type="checkbox"/> No <input type="checkbox"/> NA | |
| Avg. RF of SPCCs meet method criteria and check-marked? | <input type="checkbox"/> Yes <input type="checkbox"/> No <input type="checkbox"/> NA | |
| %RSD of all individual target components ≤15? | <input type="checkbox"/> Yes <input type="checkbox"/> No <input type="checkbox"/> NA | |
| Minimum of 5 calibration levels for all components? | <input type="checkbox"/> Yes <input type="checkbox"/> No <input type="checkbox"/> NA | |
| EPA Method 624 | | |
| %RSD of each compound <35 or when using Quadratic Regression or Linear Regression, $r^2 \geq 0.99$ for each compound? | <input type="checkbox"/> Yes <input type="checkbox"/> No <input type="checkbox"/> NA | |
| Minimum of 3 calibration levels for all components? | <input type="checkbox"/> Yes <input type="checkbox"/> No <input type="checkbox"/> NA | |
| Miscellaneous | | |
| Method properly calibrated and saved? (including) | | |
| • RTs correct? | <input type="checkbox"/> Yes <input type="checkbox"/> No <input type="checkbox"/> NA | |
| • Concentrations correct? | <input type="checkbox"/> Yes <input type="checkbox"/> No <input type="checkbox"/> NA | |
| • Reference spectra updated? | <input type="checkbox"/> Yes <input type="checkbox"/> No <input type="checkbox"/> NA | |
| • ICV acceptance criteria met? | <input type="checkbox"/> Yes <input type="checkbox"/> No <input type="checkbox"/> NA | |

Initials: _____

Date: _____

2nd Level Technical Review

Above assessment accurate ☐ Yes ☐ No

Data accurate in LIMS ☐ Yes ☐ No

If "No", list unacceptable evaluation(s): _____

LIMS QA Validation performed ☐ Yes ☐ No

Initials: _____

Date: _____

| VOA CCCs | VOA SPCCs |
|--|--|
| 1,1-Dichloroethene, 1,2-Dichloropropane, Chloroform, Ethylbenzene, Toluene, Vinyl chloride | Criteria ≥ 0.300 Chlorobenzene, 1,1,2,2-Tetrachloroethane |
| | Criteria ≥ 0.100 Chloromethane, 1,1-Dichloroethane, Bromoform |

Revision 3, 5/14/09

Instrument Conditions and Method Settings
(May vary by instrument)

5890 and 6890 Gas Chromatograph

Inlet: 250°C
Detector: 250°C

5973 MS

Interface: 180°C
MS Source: 230°C
MS Quad: 150°C

BFB-aq.m

Initial temperature: 35°C
Initial time: 2.0-minutes
Rate: 20°C / minute
Final temperature: 200°C
Final time: 0.0-minutes
Run time: 10.25-minutes

VOA-aq.m

Initial temperature: 35°C
Initial time: 2.0-minutes
Rate A: 8°C / minute
Final temperature: 130°C
Final time: 0.0-minutes
Rate B: 15°C / minute
Final temperature: 180°C
Final time: 0.0-minutes
Rate C: 25°C / minute
Final temperature: 230°C
Final time: 3.0-minutes
Run time: 22.21-minutes

| Standard Solatek Water Method (may vary by ALS) | | | |
|---|----------|---------------------|-----------------|
| Variable | Value | Variable | Value |
| Rinse Water Temp | 90°C | Turbo Cool Temp | -20°C |
| Sample Cup Temp | 30°C | GC Start | Start of Desorb |
| Sample Needle Temp | 38°C | GC Cycle Time | 0.00 min |
| Transfer Line Temp | 150°C | Sample Heater | Off |
| Soil Valve Temp | 100°C | Sample Temp | 40°C |
| Sample Sweep Time | 0.50 min | Sample Preheat Time | 0.00 min |
| Needle Rinse Volume | 7 mL | Purge Time | 11.00 min |
| Needle Sweep Time | 0.50 min | Dry Purge Time | 4.00 min |
| Bake Rinse Volume | 7 mL | Desorb Preheat Temp | 245°C |
| Bake Sweep Time | 0.50 min | Desorb Time | 1.00 min |
| Bake Drain Time | 0.50 min | Desorb Temp | 250°C |
| Number of Bake Rinses | 2 | Bake Time | 10.00 min |
| Valve Oven Temp | 150°C | Bake Temp | 260°C |
| Transfer Line Temp | 150°C | Cryofocuser | Off |
| Sample Mount Temp | 40°C | Standby Temp | 100°C |
| MCS Temp | 40°C | Focus Temp | -150°C |
| MCS Bake Time | 310°C | Inject Time | 1.00 min |
| Purge Ready Temp | 35°C | Inject Temp | 180°C |
| Purge Temp | 0°C | Purge Temp | 0°C |

| Standard Solatek Soil Method (may vary by ALS) | | | |
|--|-----------|---------------------|-----------------|
| Variable | Value | Variable | Value |
| Rinse Water Temp | 90°C | MCS Bake Temp | 310°C |
| Sample Cup Temp | 40°C | Purge Ready Temp | 37°C |
| Sample Needle Temp | 60°C | Purge Temp | 0°C |
| Transfer Line Temp | 150°C | Turbo Cool Temp | -20°C |
| Soil Valve Temp | 100°C | GC Start | Start of Desorb |
| Sample Sweep Time | 0.50 min | GC Cycle Time | 0.00 min |
| Needle Rinse Volume | 7 mL | Dry Purge Time | 5.00 min |
| Needle Sweep Time | 0.75 min | Desorb Preheat Temp | 245°C |
| Sample Preheat Time | 0.00 min | Desorb Time | 2.00 min |
| Preheat Stir | Off | Desorb Temp | 250°C |
| Preheat Stir Mode | Spin | Bake Time | 10.00 min |
| Preheat Stir Speed | 1 | Bake Temp | 260°C |
| Purge Time | 11.00 min | Cryofocuser | Off |
| Purge Stir | On | Standby Temp | 100°C |
| Purge Stir Mode | Spin | Focus Temp | -150°C |
| Purge Stir Speed | 5 | Inject Time | 1.00 min |
| Valve Oven Temp | 150°C | Inject Temp | 180°C |
| Transfer Line Temp | 150°C | Sample Heater | Off |
| Sample Mount Temp | 40°C | Sample Temp | 40°C |
| MCS Temp | 40°C | Purge Temp | 0°C |

| Standard Method Settings: Tekmar P&T 2000/3000/3001/ALS2016 (may vary by concentrator) | | | | |
|---|----------|--|-----------------|------|
| Method Type | 20XX | | | |
| | | | | |
| MCS line temp | 40 | | Purge temp | 35 |
| Purge rdy temp | 35 | | Turbo cool temp | -20 |
| | | | | |
| Sample heater | Off | | Desorb preheat | 245 |
| Prepurge time | 0.0 | | Desorb time | 2.0 |
| Preheat time | 0.0 | | Desorb temp | 250 |
| Sample temp | 40 | | Sample drain | Off |
| Purge time | 11.00 | | Bake time | 10.0 |
| Dry purge | 6.0 | | Bake temp | 260 |
| GC start | Desstart | | BGB off delay | 2.0 |
| Cryo focuser | Off | | MCS Bake | 250 |
| GC cycle time | 0.0 | | Line temp | 120 |
| Cryo standby | 100 | | Valve temp | 120 |
| Cryo focus temp | -150 | | 20XX line | 120 |
| Inj time | 1.0 | | 20XX valve | 120 |
| Cry inj temp | 180 | | | |



SOP Revision Notification / Annual Review Form

SOP Name **GC/MS DETERMINATION OF VOLATILE ORGANICS COMPOUNDS
AND VOLATILE PETROLEUM HYDROCARBONS**

☒ **New Revision** Old Revision # 5 New Revision # 6

Summary of changes: • Removed original author from the cover page.

• Added "The analysis follows the applicable criteria of SW-846 Methods 8000C/8260B or EPA Method 624." to section 3.5.

• Sec 10.7.1 and 10.7.2- updated CCV criteria to include "drift from initial calibration" when a least squares regression equation is used for an analyte.

• Sec 11.8- Updated ICAL criteria to include least squares regression equations and removing the grand mean average which is no longer employed.

• Added verbiage to Sect 13.0 regarding care in reporting analytes that use Quadratic or Quadratic forced that are beyond the linear range for the ICAL.

• Removed method 8000B from Sec 17.0 (References)

• Replaced ICAL checklist with updated format in Sec 18.0

By signing below, I certify that I have been *notified* about the approval of a *new revision* to the above mentioned SOP. I realize it is *my responsibility* to **read, understand and perform** the procedure as set forth in this new revision.

Initials & Date

Initials & Date

Initials & Date

| | | |
|-------|-------|-------|
| _____ | _____ | _____ |
| _____ | _____ | _____ |
| _____ | _____ | _____ |
| _____ | _____ | _____ |

☐ **Annual Review** Current Revision # _____

By signing below, I certify that I have **re-read, understand and agree to perform** the current revision of the above mentioned SOP.

Initials & Date

Initials & Date

Initials & Date

| | | |
|-------|-------|-------|
| _____ | _____ | _____ |
| _____ | _____ | _____ |

APPENDIX D

EXAMPLE CHAIN-OF-CUSTODY FORM



Samples Submitted to: ☐ Pace Analytical
7726 Moller Road
Indianapolis, IN 46268
Phone: 317-875-5894

Chain of Custody Record

Number ☐ Preprinted COC

Instructions on back

| | | | | | | |
|------------------|--------------------------------|---|----------------------|---|--|--|
| Client Name | Environmental Restoration, LLC | Project | Kiser Plating Muncie | Turnaround Time <input type="checkbox"/> Routine (5 working days) <input type="checkbox"/> RUSH* (notify lab) _____ (needed by) | Report Type <input type="checkbox"/> Results Only <input type="checkbox"/> Level II <input type="checkbox"/> Level III <input type="checkbox"/> Level III CLP-like <input type="checkbox"/> Level IV <input type="checkbox"/> Level IV CLP-like <input type="checkbox"/> EDD | |
| Address | 1666 Fabick Drive | Location | Muncie, IN | | | |
| City, State, Zip | St. Louis, MO 63026 | PO # | | | | |
| Contact | Toben Viehweg | Compliance Monitoring? <input type="checkbox"/> Yes <input type="checkbox"/> No | | | | |
| Telephone # | 312-446-6325 | (1) Agency/Program | | | | |

Sampled by (PRINT) Keith Hughes Sampler Signature _____ Sampler Phone # 317-491-4128

Send Report via ☐ Mail ☐ Telephone ☐ Fax (fax #) _____ ☒ e-mail (address) t.viehweg@erllc.com

* Matrix Types: Soil/Solid (S), Sludge, Oil, Wipe, Drinking Water (DW), Groundwater (GW), Surface Water (SW), Waste Water (WW), Other (specify)

** Preservative Types: (1) HNO₃, (2) H₂SO₄, (3) HCl, (4) NaOH, (5) Zinc Acetate, (6) Methanol, (7) Sodium Bisulfate, (8) Sodium Thiosulfate, (9) Hexane, (U) Unpreserved

| Client Sample ID | Matrix* | Grab | Composite | Filtered | Date Collected | Time Collected | No. of Containers | Requested Analyses → Preservative Types ** ↓ | TCLP Metals | TCLP SVOCs | TCLP VOCs | | | | | | | For Lab Use Only |
|------------------|---------|------|-----------|----------|----------------|----------------|-------------------|--|-------------|------------|-----------|--|--|--|--|--|--|------------------|
| | | | | | | | | | | | | | | | | | | |
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| Possible Hazard Identification | <input type="checkbox"/> Hazardous <input type="checkbox"/> Non-Hazardous <input type="checkbox"/> Radioactive | Sample Disposition | <input type="checkbox"/> Dispose as appropriate <input type="checkbox"/> Return <input checked="" type="checkbox"/> Archive | |
| Comments | Relinquished By (signature) | Date/Time | Received By (signature) | Date/Time |
| | Relinquished By (signature) | Date/Time | Received By (signature) | Date/Time |
| | Relinquished By (signature) | Date/Time | Received for Lab By (signature) | Date/Time |
| Sample temperature upon receipt in degrees C = _____ | | | | |

[] Pace Analytical
7726 Moller Road
Indianapolis, IN 46268
Phone: 317-875-5894

Chain of Custody Record

Number Preprinted COC

Instructions on back

| | | | | |
|--|---|--|---|--|
| Client Name Environmental Restoration, LLC | Project Kiser Plating Muncie | Turnaround Time <input type="checkbox"/> Routine (5 working days) <input type="checkbox"/> RUSH* (notify lab) <div style="text-align: center;">_____</div> (needed by) | Report Type <input type="checkbox"/> Results Only <input type="checkbox"/> Level II <input type="checkbox"/> Level III <input type="checkbox"/> Level III CLP-like <input type="checkbox"/> Level IV <input type="checkbox"/> Level IV CLP-like <input type="checkbox"/> EDD | |
| Address 1666 Fabick Drive | Location Muncie, IN | | | |
| City, State, Zip St. Louis, MO 63026 (ERRS Address) | PO # | | | |
| Contact RM's Name | Compliance Monitoring? <input type="checkbox"/> Yes <input type="checkbox"/> No | | | |
| Telephone # Phone Number of ERRS RM | (1)Agency/Program | | | |

Sampled by (PRINT) Keith Hughes Sampler Signature _____ Sampler Phone # 317-491-4128

[illegible]

* **Matrix Types:** Soil/Solid (S), Sludge, Oil, Wipe, Drinking Water (DW), Groundwater (GW), Surface Water (SW), Waste Water (WW), Other (specify)

**** Preservative Types:** (1) HNO₃, (2) H₂SO₄, (3) HCl, (4) NaOH, (5) Zinc Acetate, (6) Methanol, (7) Sodium Bisulfate, (8) Sodium Thiosulfate, (9) Hexane, (U) Unpreserved

[illegible]

| | | | | | | | |
|--------------------------------|------------------------------------|--|--------------------------------------|--------------------|---|---------------------------------|---|
| Possible Hazard Identification | <input type="checkbox"/> Hazardous | <input type="checkbox"/> Non-Hazardous | <input type="checkbox"/> Radioactive | Sample Disposition | <input type="checkbox"/> Dispose as appropriate | <input type="checkbox"/> Return | <input checked="" type="checkbox"/> Archive |
|--------------------------------|------------------------------------|--|--------------------------------------|--------------------|---|---------------------------------|---|

| | | | | |
|--|-----------------------------|-----------|---------------------------------|-----------|
| Comments | Relinquished By (signature) | Date/Time | Received By (signature) | Date/Time |
| | Relinquished By (signature) | Date/Time | Received By (signature) | Date/Time |
| | Relinquished By (signature) | Date/Time | Received for Lab By (signature) | Date/Time |
| Sample temperature upon receipt in degrees C = | | | | |

