VAPOR INTRUSION ASSESSMENT WORK PLAN (REVISION 3)

CTS OF ASHEVILLE, INC. SUPERFUND SITE

235 Mills Gap Road
Asheville, Buncombe County, North Carolina
EPA ID: NCD003149556
CERCLA Docket No. CERCLA-04-2012-3762

Prepared for:

CTS Corporation
905 West Boulevard North
Elkhart, Indiana 46514

Prepared by:

AMEC Environment & Infrastructure, Inc.
1308 Patton Avenue
Asheville, North Carolina 28806

AMEC Project 6252-12-0006

December 9, 2013

amec

December 9, 2013

Ms. Samantha Urquhart-Foster
Superfund Remedial and Site Evaluation Branch
U.S. Environmental Protection Agency
61 Forsyth Street, S.W.
Atlanta, Georgia 30303-8960
Urquhart-Foster.Samantha@epa.gov

Subject: **Vapor Intrusion Assessment Work Plan**
CTS of Asheville, Inc. Superfund Site
235 Mills Gap Road, Asheville, Buncombe County, North Carolina
EPA ID: NCD003149556
CERCLA Docket No. CERCLA-04-2012-3762
AMEC Project 6252-12-0006

Dear Ms. Urquhart-Foster:

Please find attached the Vapor Intrusion Assessment Work Plan (Revision 3; VI Work Plan) for the above-referenced Site. AMEC Environment & Infrastructure, Inc. prepared this VI Work Plan on behalf of CTS Corporation pursuant to the requirement set forth in Section 1.3.4 of the Scope of Work contained in Appendix A of the Administrative Settlement Agreement and Order on Consent for Remedial Investigation/Feasibility Study between the United States Environmental Protection Agency Region 4 (USEPA) and CTS Corporation (effective date of January 26, 2012). In a letter dated November 25, 2013, the USEPA requested that the VI Work Plan be modified to include indoor air sampling. Modifications to the VI Work Plan, Field Sampling and Analysis Plan, and Quality Assurance Project Plan have been made as appropriate.

If you have questions regarding this revised VI Work Plan, please contact us at (828) 252-8130.

Sincerely,

AMEC Environment & Infrastructure, Inc.

Susan E. Kelly, P.E., L.G.
Senior Engineer

Matthew E. Wallace, P.E.
Principal Engineer

NORTH CAROLINA PROFESSIONAL SEAL 24933 12/9/13 ENGINEER MATTHEW E. WALLACE

SEK/MEW:sek

cc: Elizabeth Ahlemann, CTS Corporation
Michael Dolan, Jones Day
Nile Testerman, NCDENR

Correspondence:
AMEC
1308 Patton Avenue
Asheville, North Carolina 28806
Tel 828.252.8130
Fax 828.251.9690
License Number: NC Corporate Engineering F-1253

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LIST OF ACRONYMS

AMEC	AMEC Environment & Infrastructure, Inc.
COPC	constituent of potential concern
FSAP	Field Sampling and Analysis Plan
HASP	Health and Safety Plan
OSWER	USEPA Office of Solid Waste and Emergency Response
ppbv	parts per billion volume
QAPP	Quality Assurance Project Plan
QA/QC	quality assurance/quality control
RI/FS	Remedial Investigation/Feasibility Study
TCE	trichloroethene (also, trichloroethylene)
USEPA	United States Environmental Protection Agency
VI	vapor intrusion
VOC	volatile organic compound

DOCUMENT REVISION LOG

Revision	Date	Description
0	3/09/2012	Initial Issuance
1	7/12/2012	Revisions based on comments received from USEPA in a letter dated June 5, 2012 (“Comments on the Vapor Intrusion Assessment Work Plan”).
2	9/11/2012	Revisions based on comments received from USEPA in a memorandum dated July 23, 2012 (“CTS of Asheville – 2012 Vapor Intrusion QAPP - QAS Review Memorandum 7/23/2012”).
3	12/9/2013	Revisions based on comments received from USEPA in a letter dated November 25, 2013 (“Request for Revision of Vapor Intrusion Assessment Work Plan - CTS of Asheville, Inc. Superfund Site”).

1.0 INTRODUCTION

This document presents the Vapor Intrusion Assessment Work Plan (VI Work Plan) associated with the CTS of Asheville, Inc. Superfund Site (Site) located at 235 Mills Gap Road in Asheville, Buncombe County, North Carolina (Figure 1). The activities described in this VI Work Plan will be performed pursuant to Section 1.3.4 of the Scope of Work contained in Appendix A of the Administrative Settlement Agreement and Order on Consent for Remedial Investigation/Feasibility Study (Settlement Agreement) between the United States Environmental Protection Agency (USEPA) Region 4 and CTS Corporation (effective date January 26, 2012). This VI Work Plan describes the proposed activities that will be undertaken to evaluate potential vapor intrusion at residences located contiguous to the Site and proximate to the currently known contaminated groundwater plume.

1.1 SITE DESCRIPTION

The Site is approximately nine acres on Mills Gap Road in Asheville, Buncombe County, North Carolina and the areal extent of the contamination. The approximate center of the Site is located at north latitude 35°29’36” and west longitude 82°30’25”. The Site formerly contained an approximate 95,000-square foot, single-story brick and metal structure in the southern portion of the Site. The building was demolished in December 2011 and the concrete building pad remains intact. The northeastern portion of the Site contains an asphalt-paved parking area and asphalt-paved driveways are located parallel to the north (front) of the building and southeast (rear) of the building. A six-foot high chain-link fence surrounds the Site and a locked gate at the north end of the Site controls access to the Site from Mills Gap Road. The Site is unoccupied.

1.2 SITE OPERATIONAL HISTORY

International Resistance Company owned and operated a manufacturing facility at the Site from 1952 until 1959, when CTS of Asheville, Inc. purchased the real property, building, and equipment. CTS of Asheville, Inc. manufactured electronic components at the facility from 1959 until April 1986. Arden Electroplating, Inc. leased a portion of the building from approximately December 1, 1985 until November 30, 1986, and the Site was conveyed to Mills Gap Road Associates (MGRA) on December 23, 1987. MGRA reportedly leased portions of the facility to various tenants, and otherwise utilized the

building for business interests. The Site has been vacant/unoccupied since the mid-1990s.

Electronic components utilized in automotive parts and hearing aids were manufactured at the Site until plant operations ceased in April 1986. Small electronic components were electroplated with tin, nickel, zinc, and silver as one step in the process. Wastes generated from the process included sludge containing heavy metals and solvents. Solvents, including trichloroethene (TCE) and acetone, were used in the process to clean and/or degrease metal objects prior to electroplating. Disposal/recycling activities at the facility prior to 1959 are unknown. From 1959 to 1986, solvents and metals were reportedly reclaimed whenever possible. Between 1959 and 1980, metal-bearing rinse waters and alkaline cleaners that could not be reclaimed from the electroplating process were reportedly disposed of through the city sewer system, while concentrated metals and solvent wastes were placed in drums for off-site disposal/recycling. After 1980, all wastes were accumulated in drums on-site prior to off-site disposal or recycling.

1.3 PREVIOUS ASSESSMENT ACTIVITIES

The results of previous assessments determined that unsaturated soil containing VOCs is limited to the former plant area of the Site and that a contaminated groundwater plume is present at and adjacent to the Site. The groundwater plume in the unconsolidated formation (i.e., above bedrock) has generally been delineated to the north and the south, but not to the east and west of the Site. The primary constituents detected in soil and groundwater samples collected during previous Site investigations include VOCs related to chlorinated solvents, such as TCE, and petroleum constituents related to fuel oil.

Previous assessment activities conducted by USEPA included air sampling and air monitoring in the area of the Site. In December 2007, 10 subslab and 12 crawlspace (SUMMA® canister) air samples were collected from 22 residences in the area of the site, as well as ambient air and ‘slam bar’ soil gas samples (T N & Associates, 2008). A Trace Atmospheric Gas Analyzer IIe was also used to screen air quality in the area of the Site. In August 2008, USEPA returned and collected five crawlspace air samples, two indoor air samples, and one soil gas sample (as well as ambient and duplicate air samples) from six residences in the area of the Site (T N & Associates, 2009). Eight of the twenty-one air

samples (not including the trip blank) collected in August 2008 did not contain TCE above laboratory reporting limits. Of the thirteen air samples where TCE was detected above the laboratory reporting limits, one air sample collected from inside the restricted-access fenced area at the springs to the east of the Site contained a concentration of TCE above the then applicable removal action limit of 23 parts per billion by volume (ppbv). The highest analytical result of TCE in the remaining twelve air samples where TCE was detected was 1.6 ppbv.

In October 2012, two crawlspace air samples, one indoor air sample, and one duplicate crawlspace air sample were collected from three residences in the area of the Site (AMEC, 2013). Two ambient air samples and one duplicate ambient air sample were also collected. Concentrations of TCE, cis-1,2-dichloroethene, and/or vinyl chloride were detected above the laboratory reporting limits in the collected air samples. The detected concentrations of TCE ranged from 0.055 to 0.12 ppbv in the crawlspace/indoor air samples. The detected concentrations of TCE ranged from 0.027 to 0.12 ppbv in the ambient air samples.

1.4 OBJECTIVES OF VI ASSESSMENT

The objective of the Vapor Intrusion Assessment is to determine whether concentrations of Site-related VOCs are present in crawlspaces and/or indoor air at residences previously assessed by USEPA that are contiguous to the Site and/or proximate to the currently known contaminated groundwater plume. The detected analytes, if any, will be compared to risk-based screening values to indicate the potential for the occurrence of vapor intrusion to pose a potential risk to the residential receptors. The purpose of comparing the measured air concentrations to the screening levels is to identify constituents of potential concern that will then be further evaluated by a risk assessment. An exceedance of a screening value does not indicate unacceptable risk or hazard for receptors. As additional data is collected during implementation of the RI/FS, this VI Work Plan may be modified to include additional sampling locations/events.

2.0 PROPOSED VAPOR INTRUSION ASSESSMENT

The proposed VI Assessment is described in the following sections. The Sampling and Analysis Plan for implementation this VI Work Plan consists of a Field Sampling and Analysis Plan (FSAP) and Quality Assurance Project Plan (QAPP). The FSAP (Appendix A) describes the data gathering methods, sampling objectives, sample locations and frequency, and sampling equipment and procedures. The QAPP (Appendix B) describes the project objectives and organization, functional activities, and the quality assurance and quality control (QA/QC) protocols that will be used to achieve the desired data quality objective for the project.

2.1 PROPOSED SAMPLE LOCATIONS

In accordance with Section 1.3.4 of the Settlement Agreement Scope of Work, this VI Assessment will evaluate vapor intrusion at residences previously assessed by USEPA that are immediately contiguous to the Site and/or proximate to the currently known groundwater plume. Crawlspace and indoor air samples will be collected from two residences at the locations shown in Figure 2. Four ambient air samples associated with the crawlspace/indoor air samples will also be collected. The locations of the ambient air samples will be dependent on the predominant wind direction at the time of sampling, and the proximity to previously identified surface waters containing Site-related VOCs.

2.2 ACCESS AGREEMENTS

Vapor intrusion assessment access authorization agreements for the 261 Mills Gap Road, 271 Mills Gap Road and 275 Mills Gap Road residences were provided to the Respondent by the USEPA on November 25, 2013. The residence at 261 Mills Gap Road is currently unoccupied, so crawlspace/indoor air samples will not be collected from this residence at this time.

2.3 PROPERTY OWNER NOTIFICATION

Prior to commencing the air sampling event, property owners will be notified by USEPA of the upcoming sampling activities. USEPA will coordinate the timing and logistical issues associated with the sampling activities.

2.4 PROPERTY RECONNAISSANCE

Prior to collecting air samples, the interior of each residence will be surveyed to collect information about the structure (e.g., configuration, presence/absence of a vapor barrier, heating/cooling systems, etc.) and to assess factors that could influence the air sampling results (e.g., products or chemicals containing VOCs). An Occupied Dwelling Questionnaire, such as the one included in Appendix C, will be completed in coordination with the occupant of the residence prior to initiating the air sampling activities.

2.5 COLLECTION OF AIR SAMPLES

Prior to collection of air samples, products or chemicals potentially containing Site-related VOCs will be attempted to be identified in the structure. Crawlspace air samples will be collected in the center of the crawlspace of the structure, or in the general location of previous samples collected by USEPA, if possible or applicable. Indoor air samples will be collected with the sample intake positioned at a height of two to three feet above the floor of the residence in an area that has the potential for frequent use. Ambient air samples will be collected either from on the ground or from a height of not more than six feet above the ground. The air samples will be collected using individually-certified, 6-Liter, electropolished, stainless steel (SUMMA®) canisters equipped with flow controllers. The samples will be collected over a 24-hour period.

2.6 ANALYSIS OF AIR SAMPLES

The air samples will be submitted for analysis of Site-related VOCs according to USEPA Method TO-15 SIM (selective ion monitoring).

2.7 HEALTH AND SAFETY

A Site Health and Safety Plan (HASP) has been developed specific to the Site activities and has been submitted to the USEPA under separate cover. The HASP applies to AMEC employees and AMEC subcontractors. Field teams will have a copy of the HASP during field activities. Personnel involved in the air sampling activities will be required to read, understand, and conform to the requirements of the HASP.

If the Field Operations Leader or Project Manager determines that sampling activities cannot be conducted due to the proximity of unauthorized persons or other unforeseen conditions or situations, the sampling activities will cease until such time as they can safely be resumed. USEPA will be notified of such occurrences.

2.8 REPORTING

A Vapor Intrusion Assessment Report will be prepared and submitted to USEPA. The Report will provide a description of the sampling and analysis activities, a tabulation of the analytical results, and a vapor intrusion risk evaluation, as described below.

A screening-level assessment of potential human health risk associated with detected analytes in the air samples (after data validation and QA/QC evaluation), will be performed. This preliminary risk evaluation will be performed in accordance with the USEPA's Draft Guidance for Evaluating the Vapor Intrusion to Indoor Air Pathway from Groundwater and Soils (USEPA, 2002) and Risk Assessment Guidance for Superfund, Volume 1: Human Health Evaluation Manual (Part F, Supplemental Guidance for Inhalation Risk Assessment; USEPA, 2009).

For screening purposes, detected indoor air concentrations will be directly compared to ambient air concentrations (background) and USEPA's indoor air screening levels. Detected crawlspace concentrations will be compared to the USEPA indoor air screening level concentrations divided by a crawlspace to indoor air attenuation factor of 0.53, which is the 95 percent upper confidence limit of the arithmetic mean for crawlspace to indoor attenuation factors assembled by USEPA (Table 19 in USEPA, 2012). The resulting screening values will be compared to the crawlspace air sampling results to assess whether potential indoor air exposures should be further evaluated through additional data collection and/or quantitative risk assessment. This crawlspace attenuation factor is based on data presented in "EPA's Vapor Intrusion Database: Evaluation and Characterization of Attenuation Factors for Chlorinated Volatile Organic Compounds and Residential Buildings" (March 2012). In USEPA's March 2012 report, empirical crawlspace attenuation factors were calculated by dividing an indoor air concentration measured in a building by the measured crawlspace concentration in the same building. Per this reference, the mean crawlspace attenuation factor for the national data set is 0.46 with a standard

deviation of 0.28. The calculated attenuation factors in the study ranged from 0.057 to 0.92. These attenuation data are primarily based on studies conducted in the northeastern and western United States. Crawlspace air samples will also be compared to ambient air concentrations.

The building attenuation factor, which expresses the ratio of the indoor air concentration arising from vapor intrusion to the crawlspace soil gas concentration, is dependent on numerous site-specific building construction conditions. These site-specific factors include construction and integrity of the building foundation, pressure differences between the subsurface and the building interior, the indoor air exchange rate, interior ventilation and air flow, and the size, geometry, and compartmentalization of the building (USEPA, 2012). Typically homes in the southeastern United States are not as “air-tight” in construction as buildings in the northeastern United States. Therefore, the crawlspace attenuation factor of 0.53, which is the 95 percent upper confidence limit of the arithmetic mean for crawlspace to indoor attenuation factors assembled by USEPA (Table 19 in USEPA, 2012), is recommended as both applicable and protective for screening purposes.

The objective of the risk evaluation is to:

- Identify constituents of potential concern (COPCs) in air through comparison to risk-based screening values published by USEPA, including residential air Regional Screening Levels and Office of Solid Waste and Emergency Response generic screening levels for the vapor intrusion pathway.
- Calculate cumulative hazard indices for non-carcinogenic COPCs and cumulative excess cancer risk estimates for potentially carcinogenic COPCs using residential exposure assumptions.
- Make a recommendation concerning whether additional sampling is warranted or whether corrective action to minimize residential exposures is indicated.

The Vapor Intrusion Assessment Report will be submitted to USEPA within 45 days of receipt of the analytical results. Draft transmittal letters to property owners will be submitted to USEPA with the Vapor Intrusion Assessment Report. The draft transmittal letters will be prepared in accordance with “Communicating Environmental Data to Property Owners and Tenants” (USEPA, 2010).

3.0 SCHEDULE

The proposed schedule for the implementation of the Vapor Intrusion Work Plan is presented below.

Activity	Time to Complete
USEPA approval of VI Work Plan	30 days after submittal (assumed 30-day review/approval period)
Commence implementation of VI Assessment	Within 30 days of USEPA approval of VI Work Plan
VI Assessment field activities	Estimated 3 days to complete
Receive laboratory data	Within 21 days of sample collection
Vapor Intrusion Assessment Report	Within 45 days after receipt of laboratory data

4.0 REFERENCES

AMEC, 2013. Vapor Intrusion Assessment Report (Revision 1), CTS of Asheville, Inc. Superfund Site EPA ID: NCD003149556, February 12, 2013.

T N & Associates, Inc., USEPA Region 4 START, 2008, Subsurface Soil and Groundwater Sampling Report, Revision 1, Mills Gap, April 23, 2008.

T N & Associates, Inc., USEPA Region 4 START, 2009, Vapor Sampling Letter Report, Revision 2, Mills Gap, June 16, 2009.

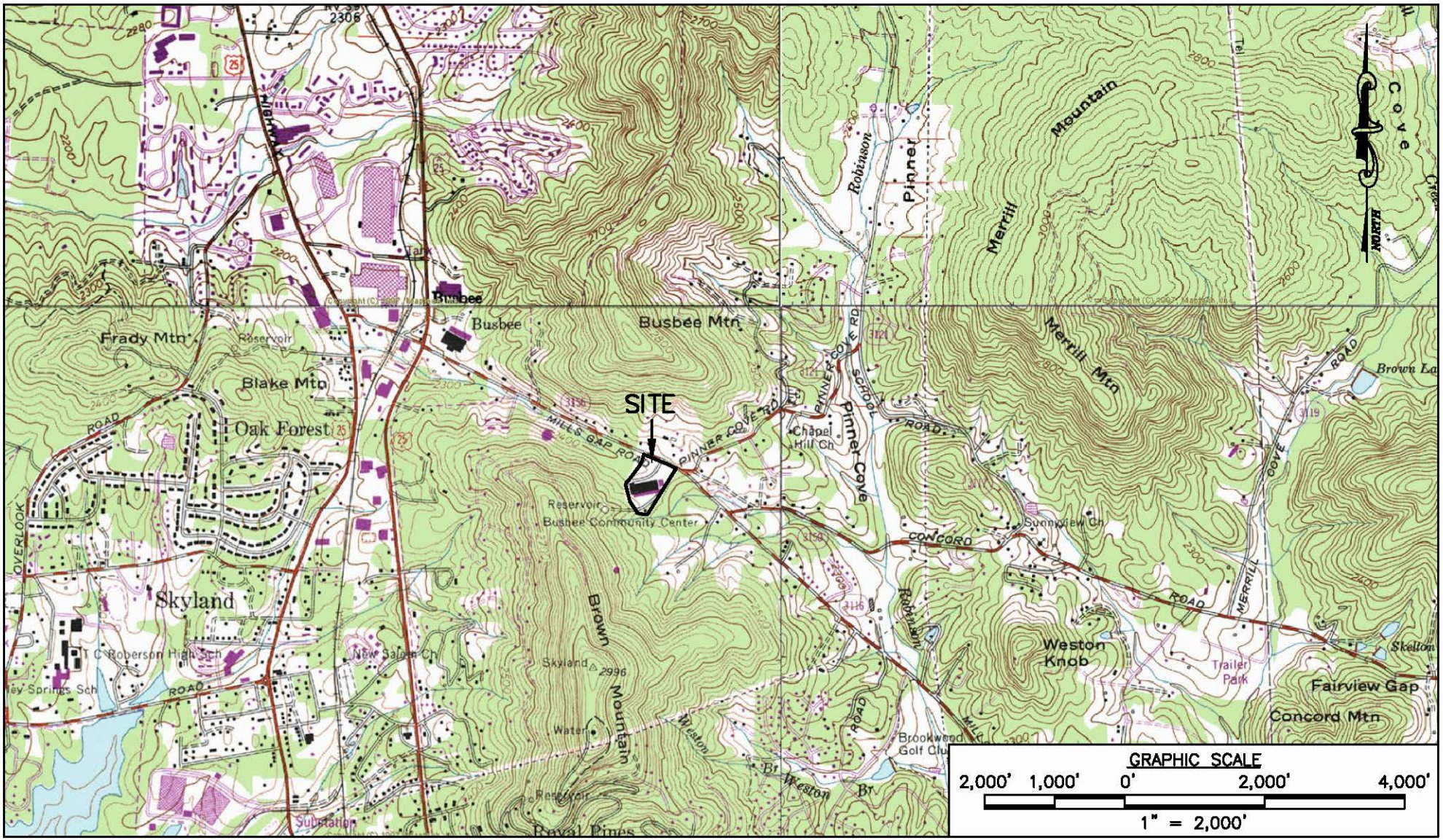
USEPA, 2002. OSWER Draft Guidance for Evaluating the Vapor Intrusion to Indoor Air Pathway from Groundwater and Soils (Subsurface Vapor Intrusion Guidance), EPA530-D-02-004, November 2002.

USEPA, 2009. Risk Assessment Guidance for Superfund Volume I: Human Health Evaluation Manual (Part F, Supplemental Guidance for Inhalation Risk Assessment), Final, EPA-540-R-070-002, January 2009.

USEPA, 2010. Communicating Environmental Data to Property Owners and Tenants (Standard Operating Procedure, Version #1), Interim Final, October 2010.

USEPA, 2012. EPA's Vapor Intrusion Database: Evaluation and Characterization of Attenuation Factors for Chlorinated Volatile Organic Compounds and Residential Buildings, EPA 530-R-10-002, March 16, 2012.

FIGURES

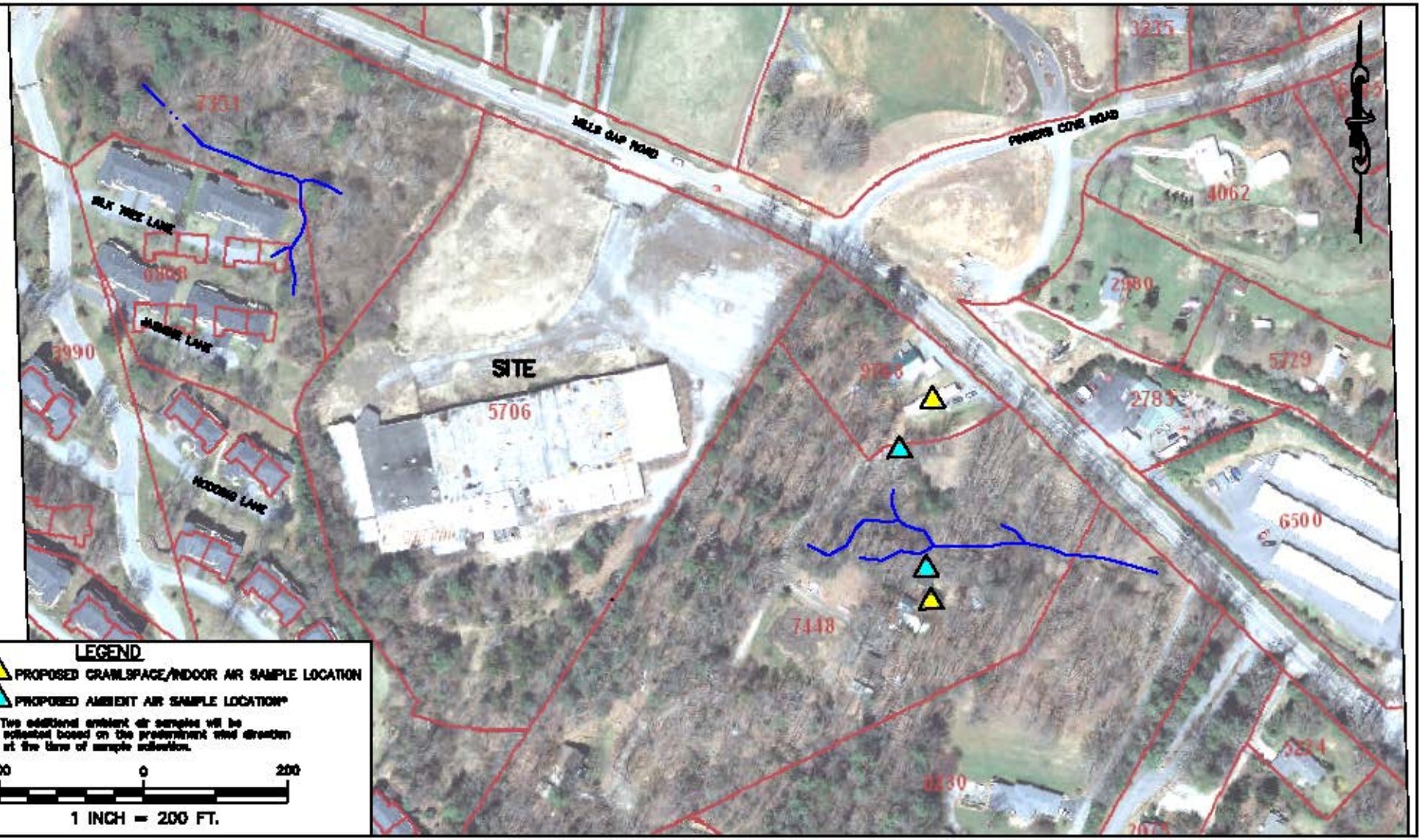


TOPOGRAPHIC SITE MAP
CTS OF ASHEVILLE, INC. SUPERFUND SITE
ASHEVILLE, NORTH CAROLINA

amec

DRAWN: SEK	ENG CHECK: --	DATE: DECEMBER 2013	PROJECT: 6252-12-0006
DFT CHECK: MEW	APPROVAL: MEW	SCALE: 1" = 2,000'	FIGURE: 1

REFERENCE: USGS QUADRANGLES: ASHEVILLE (1961), OTEEN (1962), FRUITLAND (1978) AND SKYLAND (1978)



PROPOSED AIR SAMPLE LOCATIONS
CTS OF ASHEVILLE, INC. SUPERFUND SITE
ASHEVILLE, NORTH CAROLINA

amec

DRAWN: SEK	ENG CHECK: --	DATE: DECEMBER 2013	PROJECT: 6252-12-0006
DFT CHECK: MEW	APPROVAL: MEW	SCALE: 1" = 200'	FIGURE: 2

REFERENCE: 2010 AERIAL PHOTOGRAPH FROM BUNCOMBE COUNTY GIS WEBSITE.

APPENDIX A

FIELD SAMPLING AND ANALYSIS PLAN

VAPOR INTRUSION ASSESSMENT WORK PLAN: FIELD SAMPLING AND ANALYSIS PLAN (REVISION 3)

CTS OF ASHEVILLE, INC. SUPERFUND SITE

235 Mills Gap Road
Asheville, Buncombe County, North Carolina
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CERCLA Docket No. CERCLA-04-2012-3762

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1308 Patton Avenue
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LIST OF ACRONYMS

AMEC	AMEC Environment & Infrastructure, Inc.
COC	chain-of-custody
FSAP	Field Sampling and Analysis Plan
FOL	Field Operations Leader
GPS	Global Positioning System
Hg	mercury
IDW	investigation derived waste
PID	photoionization detector
QAPP	Quality Assurance Project Plan
USEPA	United States Environmental Protection Agency
VI	vapor intrusion
VOC	volatile organic compound

DOCUMENT REVISION LOG

Revision	Date	Description
0	3/09/2012	Initial Issuance
1	7/12/2012	Revisions based on comments received from USEPA in a letter dated June 5, 2012 ("Comments on the Vapor Intrusion Assessment Work Plan").
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3	12/09/2013	Revisions based on comments received from USEPA in a letter dated November 25, 2013 ("Request for Revision of Vapor Intrusion Assessment Work Plan - CTS of Asheville, Inc. Superfund Site").

1.0 INTRODUCTION

On behalf of CTS Corporation, AMEC Environment & Infrastructure, Inc. (AMEC) has prepared this Field Sampling and Analysis Plan (FSAP) for the CTS of Asheville, Inc. Superfund Site (Site) located in Asheville, Buncombe County, North Carolina. The activities described in this FSAP will be performed pursuant to the Administrative Settlement Agreement and Order on Consent for Remedial Investigation/Feasibility Study between the United States Environmental Protection Agency (USEPA) Region 4 and CTS Corporation, effective January 26, 2012.

This FSAP presents the procedures and methods associated with the Vapor Intrusion Assessment Work Plan (VI Work Plan). This FSAP, in conjunction with the Quality Assurance Project Plan (QAPP), are included in the VI Work Plan and provide the framework upon which the Vapor Intrusion Assessment will be conducted.

2.0 SAMPLING OBJECTIVE

The objective of the Vapor Intrusion Assessment is to determine whether concentrations of Site-related volatile organic compounds (VOCs) are present in crawlspaces or indoor spaces at residences previously assessed by USEPA that are contiguous to the Site and/or proximate to the currently known contaminated groundwater plume. Concentrations of detected analytes, if any, will be compared to risk-based screening values to indicate the potential for the occurrence of vapor intrusion to pose a risk to the residential receptors. As additional data is collected during implementation of the Remedial Investigation, the VI Work Plan might be modified to include additional sampling locations/events.

3.0 AIR SAMPLING APPROACH

Air sampling will be conducted at two residences adjacent to the Site previously assessed by USEPA and/or proximate to the currently known contaminated groundwater plume.

Crawlspace and indoor air samples will be collected at the following residences:

- 271 Mills Gap Road
- 275 Mills Gap Road

Access agreements have been obtained by USEPA allowing for access by the Respondent's representative(s) from the property owners. Property owners/tenants will be notified of the sampling activities by USEPA prior to beginning the sampling event. Air samples will be collected from the crawlspaces and indoor areas of the structures.

Four ambient air samples associated with the crawlspace/indoor air samples will also be collected. The locations of the ambient air samples will be dependent on the predominant wind direction at the time of sampling, and the proximity to previously identified surface waters containing Site-related VOCs.

The air samples (crawlspace, indoor and ambient) will be submitted for analysis of Site-related VOCs, as described in the VI Work Plan QAPP. The sampling activities are expected to take up to three days to complete. Quality assurance/quality control samples will be collected/submitted as described in the QAPP.

Sample collection activities will be documented in field logbooks and on field data record forms (FDRs). Use of logbooks is described in the QAPP. Examples of FDRs that are anticipated to be used during the sample collection are contained in Appendix C of the QAPP.

The samples will be submitted for analysis with a maximum turnaround time of 21 days from the laboratory's receipt of the samples. A Vapor Intrusion Assessment Report will be prepared and submitted to USEPA, along with draft transmittal letters to property owners with the respective sampling data.

4.0 SAMPLE DESIGNATION, HANDLING, AND ANALYSIS

Procedures for sample designation, handling, and analysis are included in the Vapor Intrusion Assessment Work Plan QAPP.

5.0 SAMPLING EQUIPMENT AND PROCEDURES

The following data collection or sampling procedures are proposed for the air sampling activities:

- Data collection related to physical properties of structures
- Recording predominant direction of wind, barometric pressure, and temperature
- Deployment and retrieval of SUMMA® canisters for collection of air samples
- Surveying sample locations

5.1 COLLECTION OF PHYSICAL PROPERTIES OF STRUCTURES

Prior to collecting air samples, the interior of each residence will be surveyed to collect information about the structure (e.g., configuration, presence/absence of a vapor barrier, heating/cooling systems, etc.) and to assess factors that could influence the air sampling results (e.g., products or chemicals containing VOCs). An Occupied Dwelling Questionnaire, such as the one included in Appendix C of the VI Work Plan, will be completed in coordination with the occupant of the residence prior to initiating the air sampling activities.

Background indoor air sources of volatile chemicals in residential structures include consumer products, supplies used for personal hobbies, household cleaners, paints, and building supplies. These background sources will be attempted to be identified and removed from the residence prior to collection of crawlspace and indoor air samples.

The air in the vicinity of the location intended for sampling will be screened with a calibrated photoionization detector (PID) to assess the potential presence of VOCs. PID readings will be recorded on the Questionnaire.

5.2 RECORDING PREDOMINANT DIRECTION OF WIND

The predominant direction of wind at the sampling locations will be recorded on the FDR during deployment and retrieval of the air sampling equipment. The predominant direction of the wind will be determined based on visual observations in the area of the sampling location. Based on the topography of the area surrounding the Site, the predominant direction of the wind might be different at the individual sampling locations.

The Field Operations Leader will be notified if there is not a discernable predominant wind direction, and forecast information from weather agencies might be consulted to determine if weather patterns will produce a likely predominant wind direction during the air sampling equipment deployment period.

5.3 COLLECTION OF AIR SAMPLES

Air samples will be collected using individually-certified, 6-Liter, electropolished, stainless steel (SUMMA®) canisters equipped with flow regulators in accordance with the time integrated sample collection procedure for VOCs in USEPA Science and Ecosystem Support Division's (SESD's) Operating Procedure "Ambient Air Sampling," effective January 5, 2011. Air samples will not be collected during periods of rain or snow. Modifications to the sampling procedure will be documented in the field logbook. Clean, non-powdered, disposable gloves (e.g. nitrile) will be worn when deploying and collecting the canisters used for collecting the air samples. A new pair of gloves will be donned immediately prior to deploying and collecting the canisters.

5.3.1 Collection of Crawlspace Air Samples

Additional information on the procedure for the collection of the crawlspace air samples is as follows:

Selection and preparation of sample collection point:

- Observe the area for the apparent presence of items or materials that may potentially produce or emit VOCs and interfere with analytical laboratory analysis of the collected sample.
- Record relevant information on the FDR.
- Using a calibrated PID, screen air in the vicinity of the sample collection location to assess the potential presence of VOCs. Record PID readings on the FDR.
- If PID readings are recorded, the Field Operations Leader will determine whether the sampling should proceed.

Preparation of 24-hour SUMMA® canister and collection of sample:

- Place SUMMA® canister on floor of crawlspace if possible. As an alternate, canister can be placed on a stable surface or affixed to a wall or floor support with

nylon rope. Avoid placing canisters near windows or other potential sources of drafts and air supply vents.

- Record SUMMA® canister and flow controller identification numbers on FDR and chain-of-custody (COC).
- Record sample identification on canister identification tag, and record on FDR and COC.
- Remove brass plug from canister fitting.
- Install pressure gauge/flow controller on canister valve fitting and tighten. If pressure gauge has additional (second) fitting, install brass plug from canister fitting into gauge fitting and tighten.
- Open and close canister valve.
- Record gauge pressure on FDR and COC. Remove brass plug from gauge fitting and store for later use.
- Open canister valve to initiate sample collection.
- Record air temperature, date and local time (24-hour basis) of valve opening on FDR and COC.
- Take digital photograph(s) of SUMMA® canister and surrounding area.

Termination of 24-hour sample collection:

- Revisit SUMMA® canister approximately at end of sample collection period (e.g., 24 hours after initiation of sample collection) and record gauge pressure on FDR and COC.
- Record air temperature, date and local time (24-hour basis) of valve closing on FDR and COC.
- Close canister valve.
- Remove pressure gauge and flow controller from canister.
- Reinstall brass plug on canister fitting and tighten.
- Remove SUMMA® canister from sample collection area.

Preparation and shipment of sample to analytical laboratory:

- Pack SUMMA® canister in shipping container, note presence of brass plug installed in tank fitting.
- Complete COC and place requisite copies in shipping container.
- Close shipping container and affix custody seal to container closure.

5.3.2 Collection of Indoor Air Samples

Additional information on the procedure for the collection of the indoor air samples is as follows:

Selection and preparation of indoor air sample collection point:

- Identify the areas that have the potential for frequent use.
- Observe the area for the apparent presence of items or materials that may potentially produce or emit VOCs and interfere with analytical laboratory analysis of the collected sample.
- Record relevant information on the FDR.
- Using a calibrated PID, screen air in the vicinity of the sample collection location to assess the potential presence of VOCs. Record PID readings on the FDR.
- If PID readings are recorded, the Field Operations Leader will determine whether the sampling should proceed.

Preparation of 24-hour SUMMA® canister and collection of sample:

- Place SUMMA® canister on floor if possible. As an alternate, canister can be placed on a stable surface. Avoid placing canisters near windows or other potential sources of drafts and air supply vents. Sample intake should be approximately two to three feet above the floor.
- One sample should be collected from near the middle of the structure. If two samples are to be collected, then one of the sample locations should be the master bedroom or the bedroom of the youngest child.
- Record SUMMA® canister and flow controller identification numbers on FDR and COC.
- Record sample identification on canister identification tag, and record on FDR and COC.
- Remove brass plug from canister fitting.
- Install pressure gauge/flow controller on canister valve fitting and tighten. If pressure gauge has additional (second) fitting, install brass plug from canister fitting into gauge fitting and tighten.
- Open and close canister valve.
- Record gauge pressure on FDR and COC.
- Remove brass plug from gauge fitting and store for later use.
- Open canister valve to initiate sample collection.
- Record air temperature, date and local time (24-hour basis) of valve opening on FDR and COC.
- Take digital photograph(s) of SUMMA® canister and surrounding area.

Termination of 24-hour sample collection:

- Revisit SUMMA® canister approximately at end of sample collection period (e.g., 24 hours after initiation of sample collection) and record gauge pressure on FDR and COC. Gauge pressure should read >1 and <10 inches of mercury (Hg).
- Record air temperature, equipment serial numbers, date and local time (24-hour basis) of valve closing on FDR and COC.
- Close canister valve.
- Remove pressure gauge and flow controller from canister.
- Reinstall brass plug on canister fitting and tighten.
- Remove SUMMA® canister from sample collection area.

Preparation and shipment of sample to analytical laboratory:

- Pack SUMMA® canister in shipping container, note presence of brass plug installed in tank fitting.
- Complete COC and place requisite copies in shipping container.
- Close shipping container and affix custody seal to container closure.

5.3.3 Collection of Ambient Air Samples

Ambient (outdoor) air samples will be collected in the vicinity of the residential structures. Two ambient air samples will be collected between a surface water feature containing Site-related VOCs and the adjacent residence(s) where a crawlspace/indoor air sample is collected (see Figure 2 of VI Work Plan). Additional information on the procedure for the collection of the ambient air samples is as follows:

Selection and preparation of ambient sample collection area:

- Choose an area for sample collection that is upwind of the residence(s) being assessed, if possible. Collect sample away from wind breaks, if possible.
- For locations between a residence and a surface water feature identified as having concentrations of Site-related VOCs, choose a location approximately one-half the distance between the residential structure and the surface water feature (see Figure 2 of the VI Work Plan).
- Observe the area for the apparent presence of items or materials that may potentially produce or emit VOCs and interfere with analytical laboratory analysis of the collected sample.
- Record relevant information on FDR.
- Using a calibrated PID, screen ambient air in the location intended for sampling to assess the potential presence of VOCs. Record PID readings on the FDR.

Preparation of SUMMA® canister and collection of ambient air sample

- Place SUMMA® canister on the ground or within approximately six feet above the ground. Canister can be placed on a stable surface, or suspended from a structure/tree with nylon rope.
- Record SUMMA® canister and flow controller identification numbers on FDR and COC.
- Record sample identification on canister identification tag, and record on FDR and COC.
- Remove brass plug from canister fitting.
- Install pressure gauge/flow controller on canister valve fitting and tighten. If pressure gauge has additional (second) fitting, install brass plug from canister fitting into gauge fitting and tighten.
- Open and close canister valve.
- Record gauge pressure on FDR and COC. Remove brass plug from gauge fitting and store for later use.
- Open canister valve to initiate sample collection.
- Record air temperature, date and local time (24-hour basis) of valve opening on FDR and COC.
- Take digital photograph(s) of SUMMA® canister and surrounding area.

Termination of ambient sample collection

- Revisit SUMMA® canister approximately at end of sample collection period (e.g., 24 hours after initiation of sample collection) and record gauge pressure on FDR and COC.
- Record air temperature, date and local time (24-hour basis) of valve closing on FDR and COC.
- Close canister valve.
- Remove pressure gauge/flow controller from canister.
- Reinstall brass plug on canister fitting and tighten.
- Remove SUMMA® canister from sample collection area.

Preparation and shipment of sample to analytical laboratory

- Pack SUMMA® canister in shipping container, note presence of brass plug installed in tank fitting.
- Complete COC and place requisite copies in shipping container.
- Close shipping container and affix custody seal to container closure.

5.3.4 Collection of Quality Assurance/Quality Control Samples

The collection of quality assurance/quality control samples, as described in the QAPP, will include the submittal of field duplicate and trip blank samples to the analytical laboratory for analyses of Site-related VOCs. Duplicate samples will be collected using two SUMMA® canisters connected with a stainless steel "tee" fitting to produce one air inlet.

5.4 SURVEYING

The coordinates and location descriptions of previously collected air samples are contained in reports provided by USEPA. If a sampling location is modified (i.e., the sample has to be collected from a different location in the structure), the modified location will be noted in the field log book and will be surveyed as described below. Ambient air sampling locations and new air sampling locations will be surveyed as described below.

Sampling locations will be surveyed using a Trimble® Global Positioning System (GPS) or similar instrument. As specified in SESD Operating Procedure "Global Positioning System," effective date April 20, 2011, sample locations will be located with three meter accuracy. If a location is in an area where a GPS signal cannot be received (e.g. areas with tree canopy or inside the structure), the GPS locations will be located from the nearest point where a signal is received and deviations will be noted in the field book.

6.0 DECONTAMINATION PROCEDURES

Field equipment requiring decontamination will not be employed during implementation of the Vapor Intrusion Assessment. Air canisters, and associated flow controllers and pressure gauges, will be individually cleaned and certified by the laboratory prior to use.

7.0 MANAGEMENT OF INVESTIGATION DERIVED WASTE

The procedures associated with the management of investigative derived waste (IDW) have been developed in accordance with the SESD Operating Procedure "Management of Investigation Derived Waste," effective October 15, 2010. IDW that will potentially be generated during implementation of the air sampling program include personal protective equipment and disposable items. The IDW to be generated during implementation of the

air sampling is expected to be relatively free of contamination. IDW will be placed in trash bags and disposed of in a municipal solid waste dumpster.

8.0 REFERENCES

USEPA, 2010. Management of Investigation Derived Waste, Science and Ecosystem Support Division, Athens, GA; SESDPROC-202-R2, October 15, 2010.

USEPA, 2011. Ambient Air Sampling, Science and Ecosystem Support Division, Athens, GA; SESDPROC-303-R4, January 5, 2011.

USEPA, 2011. Global Positioning System, Science and Ecosystem Support Division, Athens, GA; SESDPROC-110-R3, April 20, 2011.

APPENDIX B

QUALITY ASSURANCE PROJECT PLAN

VAPOR INTRUSION ASSESSMENT WORK PLAN: QUALITY ASSURANCE PROJECT PLAN (REVISION 3)

CTS OF ASHEVILLE, INC. SUPERFUND SITE

235 Mills Gap Road
Asheville, Buncombe County, North Carolina
EPA ID: NCD003149556
CERCLA Docket No. CERCLA-04-2012-3762

Prepared for:

CTS Corporation
905 West Boulevard North
Elkhart, Indiana 46514

Prepared by:

AMEC Environment & Infrastructure, Inc.
1308 Patton Avenue
Asheville, North Carolina 28806

AMEC Project 6252-12-0006

December 9, 2013

Vapor Intrusion Assessment Work Plan:
Quality Assurance and Project Plan (Revision 3)

CTS of Asheville, Inc. Superfund Site
235 Mills Gap Road
Asheville, Buncombe County, North Carolina
EPA ID: NCD003149556
CERCLA Docket No. CERCLA-04-2012-3762

Prepared For: CTS Corporation
Prepared by: AMEC Environment & Infrastructure, Inc.

December 9, 2013

Matthew G. Wallace

Matthew Wallace, P.E.
AMEC Environment & Infrastructure, Inc.
Project Manager

12/9/13
Date

Chris Ricardi

Christian Ricardi, NRCC-EAC
AMEC Environment & Infrastructure, Inc.
Quality Assurance Manager

12/9/13
Date

Samantha Urquhart-Foster
USEPA Region 4
Remedial Project Manager

Date

USEPA Region 4
Quality Assurance Manager

Date

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B ALS Environmental Quality Assurance Manual
C Example Field Data Records
D ALS Environmental Standard Operating Procedure for Cleaning and Certification of SUMMA Canisters and Other Specially-Prepared Canisters
E ALS Environmental Standard Operating Procedure for Evaluation and Pressurization of Specially Prepared Stainless Steel Canisters
F ALS Environmental Standard Operating Procedure for Determination of Volatile Organic Compounds in Air Samples Collected in Specially Prepared Canisters and Gas Collection Bags by Gas Chromatography/ Mass Spectrometry (GC/MS)

LIST OF ACRONYMS

AMEC	AMEC Environment & Infrastructure, Inc.
COC	chain-of-custody
DQO	data quality objective
EDD	electronic data deliverable
FDR	field data record
FOL	Field Operations Leader
FSAP	Field Sampling and Analysis Plan
GSL	Generic Screening Level
HASP	Health and Safety Plan
LCS	laboratory control sample
LD	laboratory duplicate
MDL	method detection limit
OSHA	Occupational Safety and Health Act
PQL	practical quantitation limit
PM	Project Manager
QA/QC	quality assurance/quality control
QAM	Quality Assurance Manager
QAPP	Quality Assurance Project Plan
RI/FS	Remedial Investigation/Feasibility Study
RPD	relative percent difference
RSL	Regional Screening Level
SDG	sample delivery group
SIM	selective ion monitoring
TED	Technical Environmental Database
USEPA	United States Environmental Protection Agency
VOC	volatile organic compound

DISTRIBUTION LIST

Samantha Urquhart-Foster (1 electronic)
Remedial Project Manager
USEPA, Region 4
61 Forsyth Street, SW
Atlanta, Georgia 30303-8960
404-562-8760
urquhart-foster.samantha@epamail.epa.gov

Nile Testerman (1 electronic)
Project Manager
NCDENR, Superfund Section
207 West Jones Street
Raleigh, North Carolina 27603
919-707-8339
nile.testerman@ncdenr.gov

Matthew Wallace (1 electronic)
Project Manager
AMEC Environment & Infrastructure, Inc.
1308 Patton Avenue
Asheville, North Carolina 28806
828-252-8130
matthew.wallace@amec.com

Susan Kelly (1 paper, 1 electronic)
Field Operations Leader
AMEC Environment & Infrastructure, Inc.
1308 Patton Avenue
Asheville, North Carolina 28806
828-252-8130
susan.kelly@amec.com

Christian Ricardi (1 electronic)
Quality Assurance Manager
AMEC Environment & Infrastructure, Inc.
511 Congress Street
Portland, Maine 04101
207-775-5401
christian.ricardi@amec.com

Kathleen Aguilera (1 electronic)
Project Manager
ALS Environmental
2655 Park Center Drive, Suite A
Simi Valley, California 93065
805-526-7161
kaquilera@alsglobal.com

DOCUMENT REVISION LOG

Revision	Date	Description
0	3/09/2012	Initial Issuance
1	7/12/2012	Revisions based on comments received from USEPA in a letter dated June 5, 2012 ("Comments on the Vapor Intrusion Assessment Work Plan").
2	9/11/2012	Revisions based on comments received from USEPA in a memorandum dated July 23, 2012 ("CTS of Asheville – 2012 Vapor Intrusion QAPP - QAS Review Memorandum 7/23/2012").
3	12/09/2013	Revisions based on comments received from USEPA in a letter dated November 25, 2013 ("Request for Revision of Vapor Intrusion Assessment Work Plan - CTS of Asheville, Inc. Superfund Site").

1.0 INTRODUCTION

On behalf of CTS Corporation (CTS), AMEC Environment & Infrastructure, Inc. (AMEC) has prepared this Quality Assurance Project Plan (QAPP) for the CTS of Asheville, Inc. Superfund Site (Site) located in Asheville, Buncombe County, North Carolina. The activities described in this QAPP will be performed pursuant to the Administrative Settlement Agreement and Order on Consent for Remedial Investigation/Feasibility Study between the United States Environmental Protection Agency (USEPA) Region 4 and CTS Corporation, effective January 26, 2012 (Settlement Agreement).

This QAPP has been designed to be a project document that is applicable to the evaluation of vapor intrusion. This QAPP, in conjunction with the Field Sampling and Analysis Plan (FSAP), are included in the Vapor Intrusion Assessment (VI Work Plan) and provide the framework upon which the sampling activities will be conducted. The QAPP has been prepared to document how the sampling activities will be completed and includes investigation procedures, sampling methods, analytical methods, sample management, documentation procedures and quality assurance (QA) review procedures.

2.0 PROJECT MANAGEMENT

This section provides the overall approach to manage activities described in the VI Work Plan and includes the following:

- Project organization and responsibilities
- Problem definition
- Project description
- Data quality objectives
- Method performance objectives
- Special training, requirements, and certification
- Documentation and records management

2.1 PROJECT ORGANIZATION AND RESPONSIBILITIES

The various responsibilities of key project personnel are presented in this section and a project organizational chart is presented in Appendix A.

2.1.1 AMEC Environment & Infrastructure, Inc.

AMEC will execute the Vapor Intrusion Assessment from its Asheville, North Carolina office with support from other AMEC offices. Project personnel and duties are described in the following sections.

2.1.1.1 Project Manager

The Project Manager, Mr. Matthew Wallace, PE (North Carolina), will be responsible for the scope, cost, and technical considerations related to the project; staff and project coordination; and implementation of review of overall project quality related to the collection, completeness, and presentation of data. The Project Manager oversees the technical work conducted by the Field Operations Leader, quality assurance activities by the Quality Assurance Manager, and health and safety activities by the Site Health and Safety Supervisor.

2.1.1.2 Field Operations Leader

The Field Operations Leader (FOL), Ms. Susan Kelly, LG, PE (North Carolina), will be responsible for executing the planned work elements, issuing specific instructions for

performing assigned work elements, and performing and directing the work so it is conducted in compliance with project-specific objectives and applicable QA procedures. The FOL will coordinate with the Project Manager and Quality Assurance Manager to review general work plans and specific work elements. The FOL maintains field documentation and deliverables in the project files during the performance of the assigned tasks. For field sampling activities, the FOL will be responsible for performing and/or overseeing the field work, preparing proper documentation, and ensuring proper handling of samples from sampling activities. The FOL has the authority to issue a stop work order if field sampling operations are not being conducted in accordance with the requirements specified in the Work Plan/FSAP/QAPP or when worker safety becomes an issue.

2.1.1.3 Quality Assurance Manager

The Quality Assurance Manager, Mr. Christian Ricardi, NRCC-EAC of AMEC's Portland, Maine office, will be responsible for reviewing the project QA program as it relates to the collection and completeness of data from field and laboratory operations. Mr. Ricardi's primary responsibilities include review of quality assurance/quality control (QA/QC) protocols, ascertaining quality of environmental data collected to verify that it meets proposed data quality objectives, and identifying and verifying corrective actions, if any become necessary.

2.1.1.4 Project Chemist

A Project Chemist will responsible for reviewing laboratory reports for accuracy and completeness, performing data validation using general procedures described in USEPA Region 4's Data Validation Standard Operating Procedures for Organic Analysis (USEPA, 2008) modified for evaluation of USEPA Method TO-15/TO-15 SIM (selective ion monitoring). The Project Chemist will submit the validated laboratory reports with QA/QC Evaluation Sheets to the Quality Assurance Manager.

2.1.1.5 Site Health and Safety Supervisor

The Site Health and Safety Supervisor, Ms. Susan Kelly, LG, PE, is responsible for developing, implementing, and updating the Site Health and Safety Plan (HASP) to be consistent with anticipated conditions that may be encountered during field operations. Ms. Kelly will also serve as the FOL during implementation of the VI Work Plan.

2.1.2 Analytical Laboratory

Laboratory analyses will be performed by ALS Environmental of Simi Valley, California. Personnel organization, responsibility, and training for the laboratory can be found in ALS Environmental's Quality Assurance Manual (QAM), which is included in Appendix B.

2.2 PROJECT DESCRIPTION

The project involves collecting air samples from crawlspaces and indoor locations at residences previously assessed by USEPA that are contiguous to the Site and/or proximate to the currently known contaminated groundwater plume. Ambient air samples will also be collected to evaluate ambient outdoor air quality conditions. The collected air samples, and associated QA/QC samples will be submitted for analysis of Site-related VOCs, according to USEPA Method TO-15 SIM. Concentrations of detected constituents, if any, will be compared to risk-based screening values to indicate the potential for the occurrence of vapor intrusion to pose a potential risk to the residential receptors. As additional data is collected during implementation of the Remedial Investigation, the VI Work Plan might be modified to include additional sampling locations/events.

2.3 DATA QUALITY OBJECTIVES

Data collected at a site needs to be of sufficient quality and quantity to support defensible decision making. Data quality objectives (DQOs) are identified before the sampling and analysis begin. DQOs will be used to ascertain the type, quality, and quantity of data necessary to address problems. The USEPA guidance document, Guidance on Systematic Planning Using the Data Quality Objectives Process (USEPA, 2006), outlines the following seven-step process for establishing DQOs:

1. **State the Problem.** Concisely describe the problem to be studied.
2. **Identify the Goal of the Study.** State how environmental data will be used in meeting objectives and solving the problem, identify study questions, and define alternative outcomes.
3. **Identify Information Inputs.** Identify data and information needed to answer study questions.
4. **Define the Study Boundaries.** Specify the conditions (time periods, spatial areas, and situations) to which the decision will apply and within which the data will be collected.

5. **Develop the Analytical Approach.** Define the conditions by which the decision maker will choose among alternative risk management actions. This is usually specified in the form of an "if…then…" statement.
6. **Specify Performance or Acceptance Criteria.** Define in statistical terms the decision maker's acceptable error rate based on the consequence of making an incorrect decision.
7. **Develop the Plan for Obtaining Data.** Evaluate the results of the previous steps and develop the most resource-efficient design for data collection that meets all of the DQOs.

2.3.1 State the Problem

Soil and groundwater contamination has been identified at the CTS of Asheville, Inc. Superfund Site. The VOC groundwater plume extends beyond the Site boundaries and is in the vicinity of residential dwellings. Groundwater discharges at springs located east and west of the Site. VOCs might pose a risk to nearby residential receptors via the inhalation of vapors emanating from the groundwater plume and/or the springs/surface waters adjacent to the Site.

2.3.2 Identify the Goals of the Study

The principal study question the Vapor Intrusion Assessment addresses is:

- Do Site-related contaminants detected in the crawlspace and/or indoor air samples, if any, indicate the potential for the occurrence of vapor intrusion to pose a potential risk to the residential receptors?

Alternative actions include:

- Take no action.
- Resample the crawlspace, indoor and/or ambient air.
- Collect additional indoor air samples.
- Mitigate or reduce the source of the air contamination.
- Remove occupant(s) from residence.

2.3.3 Identify Information Inputs

The primary information input needed to support the decision making process will be the results of analyses performed on the collected air samples, as well as the environmental conditions present during sample collection. Prior to collecting air samples, the interior of each residence will be observed to collect information about the structure (e.g., configuration, presence/absence of a vapor barrier, heating/cooling systems, etc.) and to

document factors that could influence the air sampling results (e.g., products or chemicals containing VOCs). An Occupied Dwelling Questionnaire, such as the one included in Appendix C of the VI Work Plan, will be completed in coordination with the occupant of the residence prior to initiating the air sampling activities. Two crawlspace and two indoor air samples will be collected.

Four ambient air samples will be collected to determine if there are anthropogenic sources of contamination in the area where the crawlspace and indoor air samples will be collected. The results of the ambient air samples will be compared to the crawlspace and indoor air sample results where VOCs are detected to evaluate whether the crawlspace and/or indoor air sample results are the result of anthropogenic source(s) or from subsurface vapor intrusion.

Two of the ambient air samples will be collected at the locations depicted in Figure 2 of the VI Work Plan. The two pre-determined ambient air sample locations are approximately half of the distance between the nearest residence in an area of one to three residences where crawlspace and indoor air samples will be collected and a surface water feature (e.g., spring or creek) where Site-related VOCs have been detected. The other two ambient air samples will be collected upwind of the area of each of the residences where crawlspace samples will be collected. The predominant wind direction will be determined so that placement of two ambient air sample canisters can be established. Two ambient air samples will be collected upwind of the structures where crawlspace air samples are collected, as described in Section 5.3.2 of the FSAP.

Meteorological data will be collected to document the weather conditions during the time period when the samples are collected. Meteorological data, including wind speed and direction, temperature, and barometric pressure, will be obtained from the State Climate Office of North Carolina website (Climate Retrieval and Observations Network of the Southeast Database) that publishes hourly meteorological data measured at the Asheville Regional Airport. The Asheville Regional Airport is located approximately 4.5 miles southwest of the Site.

The air samples will be submitted for Site-related VOCs according to USEPA Method TO-15 SIM. The Site-related VOCs include trichloroethene and its breakdown/daughter compounds, as follows:

- trichloroethene
- cis-1,2-dichloroethene
- trans-1,2-dichloroethene
- vinyl chloride

Performance and acceptance criteria are described in Section 2.4 of the QAPP.

2.3.4 Define the Study Boundaries

The target populations for the study include:

- Two air samples collected in residential crawlspaces and indoor air locations (population is the 6 Liters of air collected at each location by the sampling container over a 24-hour period of time)
- Four ambient air samples collected (population is the 6 Liters of air collected at each location by the sampling container over a 24-hour period of time)

Samples will be collected from the crawlspaces at residences previously assessed by USEPA that are contiguous to the Site and/or proximate to the currently known contaminated groundwater plume. Sample locations are depicted in Figure 2 of the VI Work Plan. Samples will be collected from those residences where permission to collect air samples is granted by the owner of the residence. Ambient (outdoor) air samples associated with the crawlspace air samples will also be collected.

The air samples will be collected in SUMMA® canisters over a 24-hour period. The sampling activities are expected to take up to three days to complete.

Practical constraints that might interfere with the investigation include:

- Access to sampling location is not granted from property owner
- Physical limitations to the proposed sampling location (i.e., sampling personnel cannot physically enter crawlspace)
- Weather (air samples will not be collected during rain or snow conditions)
- Vegetation (restricting deployment of ambient air samples)

The smallest unit on which decisions will be made are the analytical results of individual air samples.

2.3.5 Develop the Analytic Approach

The analytical results of constituents detected in the crawlspace and indoor air samples, if any, will be compared to their associated RSL and/or GSL for indoor air. For screening purposes, the analytical results of the indoor air samples will be compared to the indoor air screening values. If crawlspace air samples are collected without a corresponding indoor air sample, the indoor air screening values will be divided by a crawlspace to indoor air attenuation factor of 0.53 for evaluation of the results. USEPA Method TO-15 SIM has quantitation limits that are less than the RSLs and/or GSLs, where established, so that potential contaminants can be detected if they are present at concentrations greater than their associated RSL/GSL (Table 1).

2.3.6 Specify Performance or Acceptance Criteria

Data is subject to random and systematic errors at different stages of the collection process and typically include the following components:

- Sampling Error: Sometimes called Statistical Sampling Error, is influenced by the inherent variability of the population over space and time, the sample collection design, and the number of samples taken. It is usually impractical to measure the entire population space, and limited sampling may miss some features of the natural variation of the measurement of interest. Sampling design error occurs when the data collection design does not capture the complete variability within the population space, to the extent appropriate for making conclusions. Sampling error can lead to random error (i.e., random variability or imprecision) and systematic error (bias) in estimates of population parameters.
- Measurement Error: Sometimes called Physical Sampling Error, is influenced by imperfections in the measurement and analytical system. Random and systematic measurement errors are introduced in the measurement process during physical sample collection, sample handling, sample preparation, sample analysis, data reduction, transmission, and storage.

The vapor intrusion assessment is a systematic study of individual locations and assessment of statistical error is not planned. Measurement error is addressed by establishing standardized methods of sample collection and through the validation of analytical data.

Data from off-Site laboratory analysis will be reviewed and validated as described in Section 5.0. Results will be evaluated based on criteria in the referenced analytical method and USEPA validation guidelines. Results may be accepted without qualification or with validation qualifiers (e.g., J, UJ). Results that don't meet minimum criteria for acceptance (i.e., qualified as rejected during validation) will be unacceptable for decision making purposes. Project quality goals (i.e., acceptance criteria) are presented in Table 2.

2.3.7 Develop the Plan for Obtaining Data

A systematic/judgmental sampling design will be implemented, as crawlspace, indoor and ambient air samples will be collected from properties that have been previously assessed by USEPA that are contiguous to the Site and/or proximate to the currently known contaminated groundwater plume. Prior to collecting the air samples, the interior of each residence will be surveyed to collect information about the structure (e.g., configuration, presence/absence of a vapor barrier, heating/cooling systems, etc.) and to assess factors that could influence the air sampling results (e.g., products or chemicals containing VOCs). Prior to collecting the air samples, the interior of each residence will be surveyed to collect information about the structure (e.g., configuration, presence/absence of a vapor barrier, heating/cooling systems, etc.) and to assess factors that could influence the air sampling results (e.g., products or chemicals containing VOCs). The following steps are included to obtain data:

- Identify sample locations and obtain permission to collect sample.
- Survey sample locations and remove potential indoor sources.
- Collect samples for laboratory analysis.
- Obtain analytical results from laboratory and complete data validation.
- Compare results to project objectives identified in Section 2.3.2.
- Complete actions for individual locations based on analytical data.

2.4 METHOD PERFORMANCE OBJECTIVES

Performance objectives are defined for field data and fixed laboratory data. Method performance objectives for work performed are expressed in terms of precision, accuracy, representativeness, comparability, completeness, and sensitivity. Target analytes and detection limits are summarized in Table 1. Project QA/QC acceptance criteria for air

samples are summarized in Table 2. The following sections describe the method performance parameters and calculations, as appropriate.

2.4.1 Precision

Precision is described as the agreement among a set of duplicate or replicate measurements. Precision is measured using relative percent difference (RPD) for two data points, as follows:

$$RPD = \frac{|X_1 - X_2| x100}{(X_1 + X_2)/2}$$

where: RPD = relative percent difference between duplicate results

X_1 and X_2 = results of original sample and duplicate analyses

$|X_1 - X_2|$ = absolute difference between duplicates X_1 and X_2

2.4.1.1 Field Precision

Field precision is assessed through the collection and measurement of field duplicates (one extra sample in addition to the original field sample). Field duplicates will be collected at a frequency of one per ten investigative samples. Precision will be measured through the calculation of RPD. The resulting information will be used to assess sample homogeneity, spatial variability of samples, sample collection reproducibility, and analytical variability. The field precision goal is a RPD less than 50 percent.

2.4.1.2 Laboratory Precision

The precision of the analysis can be inferred through laboratory duplicate samples. The laboratory analyzes one or more of these duplicate samples at a rate of one per 20 samples. The precision of laboratory analyses will be assessed by calculating the RPD for each pair of laboratory duplicate samples. The laboratory precision goal is a RPD less than 25 percent.

2.4.2 Accuracy

Accuracy is the degree of agreement between a measurement or observation and an accepted value.

2.4.2.1 Field Accuracy

Field accuracy will be achieved by adhering to sampling, handling, preservation, and holding time requirements. Trip blanks will be used to assess the potential for contamination of samples due to migration of contaminants (e.g., VOCs) during sample shipment, handling, and/or storage.

2.4.2.2 Laboratory Accuracy

Laboratory accuracy is assessed by analyzing laboratory control samples (LCS). The results are expressed as a percent recovery. Surrogate recoveries may also be used to assess accuracy. Method blanks will be used to assess possible contamination from laboratory procedures. LCSs and method blanks will be analyzed at least once with each analytical batch, with a minimum of one for every 20 samples. The percent recovery (% recovery) is calculated with the following equation:

$$\%\ recovery = \left(\frac{X - B}{T} \right) x\ 100$$

where: X = measured amount in sample after spiking
B = background amount in sample
T = amount of spike added

The laboratory accuracy acceptance criteria are presented in Table 2.

2.4.3 Representativeness

Representativeness is a qualitative measure of the degree to which sample data accurately and precisely represent a characteristic environmental condition. Representativeness is demonstrated in the project planning documents by providing full descriptions of the sampling techniques and the rationale used for selecting sampling locations. Representativeness is reassessed during the data usability process.

A 24-hour sample collection time will be used to obtain samples that are representative of daily conditions (i.e., the sample canisters will be deployed for a 24-hour period). Prior to deployment of the sample canisters, the air in the vicinity of the location intended for sampling will be screened with a calibrated photoionization detector (PID) to assess the potential presence of VOCs. The presence of elevated VOC concentrations from other

sources (i.e., not from vapor intrusion) could result in the collected sample to be diluted, which would adversely affect the analytical results of the sample (i.e., actual VOC concentrations might be too diluted to be detected by the analytical measurement equipment). If VOCs are detected by the PID, an alternate sample location will be identified where VOCs are not indicated by the PID.

2.4.4 Completeness

Completeness is a measure of the quantity of valid data obtained from a measurement system compared to the quantity that was planned under normal conditions. Percent completeness is calculated with the following equation:

$$\% \text{ Completeness} = \frac{\text{Valid Data Obtained}}{\text{Total Data Planned}} \times 100$$

Valid analytical results used to meet completeness objectives are those results that provide defensible estimates of the true concentration of an analyte in a sample. These valid results include data that is not qualified and data for which QC results indicate qualification is necessary but which may still be used to meet project objectives. Invalid results are those data for which there is an indication that the prescribed sampling or analytical protocol was not followed.

The goal of the Vapor Intrusion Assessment is to obtain field samples and usable laboratory data from residential structures identified in the VI Work Plan that have granted access for sampling. After the completion of the sampling activities, an assessment of sampling completeness will be performed to identify residences where samples were not obtained, if any. A second evaluation of completeness will be done upon completion of data validation to determine if usable data were obtained from the laboratory for the collected samples. If usable data for the Site-related VOCs are not obtained from each sampled location, an evaluation will be conducted to determine the impact of missing data on the assessment of potential contamination at the missing locations. Recollection of samples might be necessary, as determined after assessment of available data from other sample locations. A completeness goal of 100 percent is proposed for this project.

2.4.5 Comparability

Comparability is the degree of confidence with which one data set can be compared to another. Throughout sampling on this project, standard methodologies as discussed in the FSAP and in this QAPP will be used for both sampling and analysis activities to ensure comparability. The intention is to use the same laboratory (ALS Environmental) for standard analyses and there should not be a need to assess the comparability of data from different laboratories.

2.4.6 Sensitivity

Sensitivity is the capability of a test method or instrument to quantify sample results at levels consistent with the project objectives. The analytical method selected for this investigation will provide detection limits sufficient to determine the presence or absence of contamination in air samples at low levels. Although there is not a single definition of this term, the following terms and definition of detection limits will be used:

- Method detection limit (MDL) is a statistically determined concentration. It is the minimum concentration of an analyte that can be measured and reported with 99 percent confidence that the analyte concentration is greater than zero as determined in the same or a similar matrix. Because of the uncertainty of accuracy and precision in this range, sample results greater than the MDL but less than the practical quantitation limit will be reported as estimated and flagged with a "J."
- Practical quantitation limit (PQL) is the concentration of the target analyte that the laboratory has demonstrated the ability to measure within specified limits of precision and accuracy during routine laboratory operating conditions. This value is variable and highly matrix-dependent. It is the minimum concentration that the laboratory will report as unqualified.

Laboratory method detection and practical quantification limits are presented in Table 1. The laboratory PQLs and MDLs were compared to applicable RSLs and GSLs, where established, for the constituents analyzed. With the exception of trichloroethene (TCE), the PQLs are less than the associated RSLs and/or GSLs, where established (i.e., a RSL has not been established for cis-1,2-dichloroethene). The PQL for TCE is 0.025 micrograms per cubic meter ($\mu g/m^3$) and the GSL is 0.022 $\mu g/m^3$; however, the MDL for TCE is 0.0067 $\mu g/m^3$, so an estimated concentration of TCE can be determined if TCE is present in an air sample.

2.5 SPECIAL TRAINING/CERTIFICATION

Training of field personnel will be provided by the FOL. Routine training will be completed at the beginning of each field event if required. The FOL will review applicable procedures with each field personnel to verify that the project requirements and procedures are understood and implemented properly. Field personnel will review and follow manufacturer's operating procedures for field equipment, such as the PID.

Personnel conducting work at the Site will be appropriately trained in health and safety procedures. If appropriate, personnel conducting work covered by this QAPP will have obtained at a minimum, the 40-hour hazardous waste-site worker training program and the 8-hour annual refresher course in compliance with regulations stated in 29 CFR Part 1910.120. Certificates or documentation representing completion of training will be maintained in personnel files. The Project Manager or FOL will verify that personnel have the necessary training and certifications prior to the implementation of the project.

A Site HASP has been developed specific to the Site activities discussed in this QAPP. The HASP applies to AMEC employees and AMEC subcontractors, only. Personnel implementing the air sampling activities will be required to read, understand, and conform to the requirements of the HASP. If new information arises, the HASP will be updated, as necessary, to ensure compliance with the Occupational Safety and Health Act (OSHA) and safe working conditions.

2.6 DOCUMENTS AND RECORDS

Critical records for the work will be maintained in AMEC's Asheville, North Carolina, office. File maintenance will be under the direct control of the Project Manager. Project records will be organized with a project-specific file and document numbering system in accordance with AMEC protocols.

The anticipated project records include:

- Project log book(s)
- Field data records (FDRs)
- Safety records, as specified in the Site HASP

- Chain of custody records
- Laboratory reports

The hard copy records will be stored in AMEC's Asheville, North Carolina office.

The anticipated electronic records include:

- Laboratory electronic data deliverables (EDDs)
- Final validated laboratory analytical results
- Meteorological data obtained from internet sources
- Survey data
- Photographs (digital)
- Scanned hard copy information (e.g., log books, FDRs, etc.)

The electronic records will be stored on AMEC's Asheville, North Carolina office server, which is backed up daily. Electronic data will be available to other AMEC office servers, as necessary for data validation, presentation, etc.; however, the original data documents will be stored on the Asheville server.

The off-Site laboratory (ALS Environmental) will submit analytical results in hard copy and electronic formats. The electronic data will be submitted as EDDs in accordance with the format described in Table 3. The analytical results will be imported into the AMEC Technical Environmental Database (TED). The TED is an Oracle-based relational database designed with using Microsoft structured query language. The TED is used to manage and store a variety of records generated during field investigations, including sample location information and analytical data. The TED can provide output files, such as Excel, for use in data validation and subsequent importation of data qualification actions. A variety of data output formats are available including sample results summary tables, summaries of data qualification actions, trend analysis for long term monitoring programs, and queries used to generate figures and tables in contamination and risk assessments. The TED data are permanently stored on a secure AMEC server that is backed up daily.

The primary documents to be produced for the project are the Vapor Intrusion Assessment Report and the draft transmittal letters. The Vapor Intrusion Assessment Report will include:

- A description of the work performed, including sampling and laboratory procedures
- An evaluation of the analytical results with respect to the RSLs and GSLs
- A screening-level assessment of potential human health risk associated with detected analytes in the air samples
- A map depicting the sampling locations
- A table summarizing the analytical results of the samples
- A data validation summary with associated documentation and the laboratory analytical results

The draft transmittal letters will be prepared in accordance with "Communicating Environmental Data to Property Owners and Tenants" (USEPA, 2010).

If this QAPP, or the associated VI Work Plan or FSAP, is updated/modified, the updated version(s) will be distributed to those persons identified in the distribution list. If the modifications are minor, then only the portions of the document with the modifications will be disturbed; otherwise, the entire document(s) will be distributed.

Retention of files will be in accordance with the Settlement Agreement, which requires the preservation and retention of records and documents during the pendency of the Settlement Agreement and for a minimum of fifteen years after completion of construction of any remedial action.

3.0 MEASUREMENT AND DATA ACQUISITION

The following sections describe the design and implementation of measurement procedures and discuss the methods to be used for sampling, analysis, data handling, and QC in support of the tasks performed.

3.1 SAMPLING PROCESS DESIGN

The VI Work Plan describes the proposed sampling plan, including planned sampling locations, rational for the sampling locations, and measurement methods. Procedures for air sampling, surveying, decontamination, and management of investigation derived waste are described in the FSAP.

Samples will be collected from the crawlspaces at residences previously assessed by USEPA that are contiguous to the Site and/or proximate to the currently known contaminated groundwater plume. Samples will be collected from those residences where permission to collect air samples is granted by the owner of the residence. Ambient air samples associated with the crawlspace air samples will also be collected. The air samples will be collected in 6-Liter SUMMA® canisters over a 24-hour period and submitted for analysis of Site-related VOCs according to USEPA Method TO-15 SIM.

3.1.1 Sampling Method

The sampling method for the collection of crawlspace and ambient air samples is described in Section 5.3 of the FSAP.

Field equipment for the project includes:

- A PID for detecting potential VOCs in the area of the sampling activities
- SUMMA® canisters
- Pressure gauges for SUMMA® canisters
- Flow controllers for SUMMA® canisters

Field supplies for the project include:

- FDRs and log books
- Personal protective equipment (e.g., nitrile gloves)
- Measuring tapes (engineers scale)

- Shipping containers (cardboard boxes)
- Packing tape
- Black ink pens
- Digital camera
- Calibration gases (supplied with rented equipment)

Support supplies/equipment include:

- Vehicles for personnel and sample transport
- Supplies to deploy air canisters, if needed

3.1.2 Field Sampling Documentation

Documentation of field activities will be completed using a combination of logbooks, FDRs, and sample custody records. Field logbooks will be completed to provide a general record of activities and events that occur during each field task. FDRs have been designed for exploration or sample collection tasks, to provide a complete record of data obtained during the activity. Examples of FDRs that will be used during the air sampling activities are included as Appendix C.

Deviations from the procedures specified in the QAPP and the FSAP will be documented in the field logbook and applicable FDRs. Such deviations may be dictated by Site-specific conditions encountered during the sampling activity.

3.1.2.1 Field Logbooks

The field logbook provides a daily hand written account of all field activities. Logbooks will be permanently bound and entries will be made in permanent black or blue ink, and corrections will be made with a single line with the author's initials and date. Each page of the logbook will be dated and signed by the person completing the log. Partially completed pages will have a line drawn through the unused portion at the end of each day. The following information will generally be entered into the field logbook during implementation of the Vapor Intrusion Assessment:

- Project name and number
- Date and time of each entry
- Weather conditions anticipated for the day, or as weather conditions change

- Site personnel and their responsibilities
- Descriptions of important tasks or subtasks
- A description of samples collected (if not documented on a FDR)
- A summary of problems encountered during the day, including cause of problem and corrective actions implemented, if appropriate

3.1.2.2 Field Data Records

Field data records contain sample collection and/or exploration details. Examples of FDR forms anticipated to be used during implementation of the Work Plan are contained in Appendix C. FDRs will be completed in the field by field personnel at the time testing/sampling is done. The goal of the FDR is to document exploration and sample collection methods, materials, dates and times, and sample locations and identifiers. Field measurements and observations associated with a given exploration or sample collection task will be recorded on the FDR. FDRs will be maintained throughout the field program in files that become a permanent record of field program activities. FDRs anticipated for the Vapor Intrusion Assessment include Photograph Logs and Air Sampling Records.

3.2 SAMPLE HANDLING AND CUSTODY

The following sections describe how samples will be identified, contained, packaged, transported, and tracked during sampling and analysis activities. The FOL will maintain the field logbook and will be responsible for sample custody in the field.

3.2.1 Sample Designation

Samples will be designated on the basis of sample type (e.g., field sample or QA/QC sample) and associated location, if applicable. Sample identification numbers will be assigned to the sampling locations in a sequential manner, beginning with “-01”. The sample identification number will remain the same for a particular sampling location throughout the duration of the Remedial Investigation, if additional samples are collected at the same location at a later time.

Field samples will be designated with the air sample type (i.e., crawlspace or ambient) and the predetermined location identification number. Crawlspace air samples will be identified as “CAS”, indoor air samples will be identified as “IAS”, and ambient air samples will be identified as “AAS.”

The QC samples will be cross-referenced on the sample FDRs or in the field logbook. The QC samples will have a prefix identifying their purpose, followed by the sequential number, as follows:

- FD-01 (field duplicate)
- TB-01 (trip blank)

Each sample submitted for analysis at the laboratory will be identified with a unique identification number (sample ID). These sample ID will be tracked from collection through laboratory analysis and into the final reports. The sample IDs will be recorded on a FDR during the sampling activities. The sample information will be recorded on FDRs or in the field logbook with sample designation information while in the custody of the sampling team. A sample label, or a tag, will be completed and attached to each sample container for every sample collected. Labels will be completed using an ink pen, and will contain at least the following information:

- Project name or number
- Date and time of sample collection
- Sample identification number
- Sampler's initials
- Analysis to be conducted

3.2.2 Sample Collection and Preservation

Air samples will be collected using 6-Liter SUMMA® canisters supplied by the laboratory. The canisters will be individually cleaned and leak checked by the laboratory. ALS Environmental's standard operating procedures related to cleaning and pressurizing the SUMMA® are included as Appendix D ("Standard Operating Procedure for Cleaning and Certification of SUMMA Canisters and Other Specially-Prepared Canisters") and Appendix E ("Standard Operating Procedure for Evaluation and Pressurization of Specially Prepared Stainless Steel Canisters"). Flow controllers and vacuum gauges will also be supplied by the laboratory. The flow controller will be adjusted by the laboratory so that the canisters will collected a sample over a 24-hour period under vacuum. The samples will be submitted for Site-related VOCs according to USEPA Method TO-15 SIM, which

has a holding time of 30 days. Sampling procedures are described in Section 5.3 of the FSAP.

3.2.3 Sample Packaging and Shipment

Sample containers will be placed in a shipping container for shipment to the laboratory. Custody seals, which are preprinted, adhesive-backed seals designed to break if disturbed, will be placed on the shipping container prior to shipment to provide security. The custody seals will be signed and dated before leaving AMEC's possession. Upon receipt by the laboratory, the sample custodian will confirm that the custody seals are intact or notify the Project Manager or FOL if custody seals have been broken.

Regulations for packaging, marking/labeling and shipping hazardous waste materials and waste are issued by U.S. Department of Transportation (USDOT). Air carriers which transport hazardous material, such as Federal Express, may also require compliance with the current edition of the International Air Transport Association (IATA) Dangerous Goods Regulations. Current IATA Regulations will ensure compliance with USDOT protocol.

3.2.4 Chain-of-Custody Records

The chain-of-custody (COC) record will be placed inside the shipping container. An example of the ALS Environmental's COC is on page 27 (Figure 6-1) of ALS Environmental's QAM (Appendix B). The sampling personnel will retain a copy of the COC. The COC will include at least the following information:

- Name of person collecting the samples
- Date and time samples were collected
- Sample ID
- Canister and flow controller ID
- Canister pressure (start and stop)
- Analyses requested
- Signature of the sampling person relinquishing samples to a non-sampling person (such as a Federal Express agent or laboratory courier), with the date and time of transfer

In addition, if samples are known to require expedited turnaround in the laboratory due to project time constraints or analytical concerns such as extraction time or sample retention

period limitations, the person completing the COC record will note these constraints in the remarks section of the custody record and will notify the Laboratory Project Manager of the expedited turnaround requirement.

3.2.5 Laboratory Custody Procedures

Information regarding the laboratory's sample receipt, handling and custody procedures are presented in ALS Environmental's QAM (Section 6.0). Below is a brief overview of lab custody procedures.

Upon arriving at the laboratory, samples are logged in by a designated sample custodian giving each sample a unique laboratory designation/ID. Sample receipt protocols and storage conditions include the following:

- Verify samples received are listed in the COC. Notify Laboratory Project Manager if not listed.
- Verify that sample holding times have not been exceeded. Notify Laboratory Project Manager if hold times have been exceeded.
- Examine shipping records for accuracy and completeness.
- Sign COC and attach the waybill.
- Note any other problems with the samples on the receipt form, specifically with preservation and contact the Laboratory Project Manager if problems are identified.
- Log samples into the Master Logbook and into the Laboratory Information Management System, and attach the laboratory sample numbers to each sample container.
- Measure the vacuum of the received canisters using a calibrated gauge.
- Place the samples into proper laboratory storage.

The Laboratory Project Manager will send a copy of the laboratory sample receipt form via email to the AMEC Project Manager, or an acceptable representative, and AMEC will verify that the samples were received in an acceptable condition. The laboratory will also generate an intra-lab COC that will be maintained while the samples are being analyzed and remain in lab custody. This procedure ensures that the sample integrity is maintained through adequate protection from contamination from outside sources or from highly contaminated samples.

Holding times are the responsibility of the laboratory for samples received within 48 hours of sampling or if less than half of the holding time has passed. If a holding time is exceeded, the laboratory will identify and document the root cause of the failure, and will contact the Laboratory Project Manager.

3.3 ANALYTICAL METHOD

Site-related VOCs in air samples and associated QC samples will be analyzed by USEPA Method TO-15 SIM. The laboratory's standard operating procedure for Method TO-15 is included as Appendix F. The air samples are collected in specially-prepared stainless steel canisters. A known volume of sample is directed from a canister through a solid multi-sorbent concentrator where VOCs are trapped. VOCs are then released by thermal desorption and carried onto a gas chromatographic column for separation and identification using a mass spectrometer. The mass spectrometer is operated using the selective ion monitoring (SIM) mode, which allows for detection of specific analytes with increased sensitivity relative to full scan mode. Responses of detected analytes are compared to responses of standards with known concentrations to establish the analyte concentration present in the sample.

3.4 FIELD QUALITY CONTROL

The field quality control program ensures that samples collected are representative of the media being sampled and that the data generated are valid. Field quality control will be accomplished through accurate record keeping in the field logbook and FDRs and collection and analysis of QC samples including field duplicates and trip blanks. Field quality control criteria are summarized in Table 2.

QC blank data will be reviewed during data validation to assess possible impacts of field or lab contamination on sample results. Problems that require corrective action may be encountered in the field. Findings that require corrective action will be communicated to the Project Manager and documented in the field logbook. The Project Manager will confirm that corrective actions have been implemented and that the problem has been resolved. If more easily addressed problems are encountered in the field, such problems will be addressed and the corrective action noted in the field logbook. If an error is made

on an accountable document assigned to one individual, that individual will make corrections by crossing a line through the error, entering the correct information, and initialing and dating the correction. The erroneous information will not be obliterated. The person who made the entry will correct any subsequent error discovered on an accountable document.

The following sections describe quality control samples that will be collected during implementation of the VI Work Plan.

3.4.1 Field Duplicates

Field duplicates are two samples taken from the same location at the same sampling time, but submitted to the laboratory as a separate sample and analyzed separately. Duplicate samples will be collected using two SUMMA® canisters connected with a stainless steel "tee" fitting to produce one air inlet. Field duplicates will be collected at a frequency of ten percent. At least one duplicate sample will be collected from a crawlspace air sample location, one duplicate will be collected from an indoor air location and one duplicate sample will be collected from an ambient (outdoor) air sample location. The acceptance criteria for field duplicate samples is an RPD of less than or equal to 50 percent.

3.4.2 Trip Blanks

A trip blank is utilized to detect possible VOC contamination of samples to be analyzed for VOCs. A trip blank canister will be individually cleaned and certified by the laboratory and will accompany the sample containers during transit, during sampling activities, and during storage with the collected samples prior to analysis. One trip blank sample will be analyzed per sampling event. The analytical results of trip blank samples will be evaluated during the data validation process to determine the representativeness of the field sample results and to determine if data qualification actions are necessary.

3.5 LABORATORY QUALITY CONTROL

Laboratory performance will be monitored by the inclusion of various internal QC checks that allow an evaluation of method control (batch QC) and the effect of the sample matrix on the data being generated (matrix-specific QC). The overall data quality objectives are to implement procedures for the laboratory analysis and reporting of the data that are

indicative of the degree of quality consistent with their intended use. Laboratory batch QC samples consist of method and instrument blanks, laboratory control samples, and calibration verification samples. Matrix specific QC samples consist of field and laboratory duplicates. ALS Environmental's general QC procedures are included in Section 9.0 of their QAM (Appendix B). ALS Environmental's QC procedures for Method TO-15 are included ALS Environmental's standard operating procedure for Method TO-15 (Appendix F).

3.6 INSTRUMENT/EQUIPMENT TESTING, INSPECTION, AND MAINTENANCE

Maintenance and inspection of field and laboratory equipment are described in the following sections.

3.6.1 Field Equipment

Preventative maintenance of field measurement instrumentation and equipment will be performed according to the procedures presented in the manufacturer's instructions. The field staff and/or subcontractors are responsible for ensuring instrumentation is operating properly prior to use. If problems are encountered, they will be communicated to the FOL and documented in the field logbook. The faulty instrumentation/equipment will be scheduled for repair and then sequestered and tagged until repaired and qualified for re-use.

3.6.2 Laboratory Equipment

Testing, inspection, and maintenance of laboratory instruments/equipment will be conducted in accordance with the procedures specified in Section 12 of ALS Environmental's QAM (Appendix B) and Section 10.0 of ALS's standard operating procedure for Method TO-15 (Appendix F).

3.7 INSTRUMENT/EQUIPMENT CALIBRATION AND FREQUENCY

General guidance regarding calibration and frequency of calibration of field and laboratory equipment are described in the following sections.

3.7.1 Field Equipment

A thermometer will be used to measure outdoor and indoor air at the beginning and end of sample collection at each sample location. A PID will be used to measure the

concentration of potential VOCs in the vicinity of the sampling locations. Proper maintenance, calibration, and operation of the equipment will be the responsibility of the sampling personnel. The equipment used during the field investigations will be maintained, calibrated, and operated according to the manufacturer's guidelines and recommendations. Calibration of the equipment will be documented in the field logbook.

The PID will be calibrated using two-point calibration. One point will be zero air calibration gas and the second point will be a standard reference gas of known concentration. The PID's computer has a set of known/pre-programmed gases and concentrations, so isobutylene at 100 ppm will be selected for the second calibration point. The calibration will be verified after the two-point procedure. The calibration will be acceptable when the fresh air concentration is within 5 ppm for the zero air calibration, and the gas concentration is within 10 percent of the standard gas concentration.

3.7.2 Laboratory Equipment

The calibration of laboratory instruments and support equipment is necessary to ensure that the analytical system is operating correctly and functioning within the guidelines of precision, accuracy, and sensitivity. The frequency and type of calibration for laboratory equipment/procedures and control limits/acceptance criteria are presented in Section 9.0 of ALS Environmental's QAM (Appendix B) and Sections 11.0 and 12.0 of ALS Environmental's standard operating procedure for Method TO-15 (Appendix F).

Reference standards are used to calibrate the equipment. Physical reference standards include weights for scales and balances, and certified thermometers for calibrating working thermometers. Chemical reference standards include reference materials traceable to recognized standards suppliers, and are generally associated with normal instrument calibrations. The standards must be verified by quantitation against a second known standard before the data is reported, and must meet specified QC criteria for calibration verification.

At minimum, the laboratory equipment must be calibrated and maintained at intervals prescribed by the method. An instrument is said to be calibrated when an instrument response can be directly related to the concentration of an analyte graphically through the use of a calibration curve. The low standard of the curve will be established by the

laboratory as the PQL. Results above the highest standard will be diluted into the calibration range and reanalyzed.

3.8 INSPECTION/ACCEPTANCE OF SUPPLIES AND CONSUMABLES

Supplies and consumables that are anticipated to be used during the Vapor Intrusion Assessment include SUMMA® canisters, flow controllers, vacuum gauges, calibration gas, and personal protective equipment (gloves). The FOL is responsible for confirming that the materials meet the required specifications, are intact and in good condition, are available in adequate supply, and are stored appropriately until use. Certification of the SUMMA® canisters, flow controllers, and vacuum gauges will be provided by the laboratory and maintained in the project file. The laboratory's standard operating procedures related to the SUMMA® canisters are included in Appendices D and E.

The PID will be rented from an environmental equipment supplier. Calibration gases will be purchased from an environmental equipment supplier and maintained at the AMEC Asheville, North Carolina office until use. Calibration gas certifications will be reviewed to ensure that they are at the concentration ranges specified for the project and that they are within their expiration dates. Certification records will be maintained in the project files. Calibration acceptance criteria are indicated on the calibration record in Appendix C.

3.9 NON-DIRECT MEASUREMENTS

For the purposes of the Vapor Intrusion Assessment, non-direct measurements will include information/data from previous investigation reports, information regarding the environment of the sample location (i.e., information from the Occupied Dwelling Questionnaire), and meteorological data. Non-directly measured data will be retained in the project files. The data may be of unknown quality and will be assessed by the Project Manager and Quality Assurance Manager if the non-direct data is used in the decision making process.

3.10 DATA MANAGEMENT

The objective of data management is to establish procedures to be used during field investigations for documenting, tracking, and presenting investigative data. Data

generated during the field investigations, as well as previously existing data, form the basis for developing conclusions and recommendations. Efficient utilization and comprehensive consideration of available data requires that the data be properly organized for review. Organization of the data will be planned prior to collection to assure the generation of identifiable and useable data. This section describes procedures necessary to provide for collecting sufficient data to accurately validate raw data and to transfer validated data to a data management system through which it can be evaluated with minimal effort. This section also describes the operating practices to be followed by personnel while collecting and reporting data.

3.10.1 Investigation Data

The following data will be collected and maintained in the project file:

- Air sampling records (FDR and logbooks including observations, temperature, and PID readings)
- Meteorological data obtained from the State Climate Office of North Carolina
- Photographs
- COC records

The flow of data for the project will be as follows:

- Field data records, including COCs, will be forwarded to the FOL, if collected by others.
- Air samples will be sent to the selected laboratory.
- Laboratory results, including EDDs and hard copies, will be sent to the FOL; the FOL will forward the laboratory results, including EDDs to the Quality Assurance Manager.
- The Data Reviewer will perform data validation with oversight by the Quality Assurance Manager. The validated data will be transferred in electronic format to the FOL.

3.10.2 Off-Site Laboratory Data

The laboratory will provide a hardcopy analytical report to document the chemical testing and report analytical results. Data deliverables will include sample result and QC summary forms and all supporting raw data needed to verify sample results. For USEPA Method TO-15, forms similar to those defined under the USEPA Contract Laboratory Program (CLP) deliverables will be required. The laboratory can use customized reporting forms providing they contain equivalent information as CLP forms.

At a minimum, the data packages from the laboratory will include the following:

- Data package narrative
  - summary of analytical methods used
  - correlation of field sample identifications and laboratory sample identifications
  - data qualifier definitions
  - deviations from established QA/QC procedures with corrective action
- Sample results
  - project name
  - field sample identification
  - laboratory sample ID
  - unit of measurement
  - batch number
  - collection/extraction/analysis dates
  - detection limits
  - dilution factors
- Sample documentation
  - original chain-of-custody
  - shipping documents
  - receipt forms
- Quality Assurance/Quality Control
  - spike recoveries (LCS)
  - internal standard summary
  - initial calibration summaries
  - GC/MS tuning summaries
  - continuing calibration summaries
  - QC blank summaries
  - measures of precision (laboratory duplicates)
  - control limits for accuracy and precision
  - surrogate recoveries
- Raw data including instrument printouts (run sequence/acquisition files, chromatograms and quantitation reports), instrument logbook pages, and sample preparation logs.

Sample results will be provided by the laboratory in both electronic and hard copy format. Hard copy and EDDs from the laboratory will be transmitted to the Quality Assurance Manager and Project Chemist. The electronic data will be provided in a format described in Table 3. Electronic lab results will be imported into the AMEC TED. Files of the unvalidated electronic data are provided to the Project Chemist for used during data

validation. The project chemist will make any necessary data qualification and changes based on the data validation review, and qualified results are entered back into the TED database. During data validation a quality assurance review of sample results will be completed to ensure that the data in the database match the hard copy provided by the laboratory.

Final validated laboratory data will be maintained in the TED database to allow easy retrieval of information and electronic transfer of the data to other parties. Once final data are entered into the TED and validation is completed, data reports will be generated as needed to support contamination assessments and report preparation. A data validation report will be prepared and will be organized by sample collection task and might include multiple sample delivery groups (SDGs). The validation report(s) will include the following information:

- Introduction
  - description of sampling task
  - identity of the laboratory used for analysis
  - a summary of analytical methods
  - a table summarizing summary of SDGs and samples are included in the report
  - a description of the data validation process
  - a table summarizing project QC limits
- Validation actions and observations
  - a discussion of data validation actions, qualifications, and observations
  - a table summarizing all data qualification actions
  - A tabulation of validated samples results
- References cited

Upon completion of the field investigation and subsequent validation of off-Site laboratory data, an EDD will be prepared with relevant field information and laboratory data in the format specified for environmental data in the Region 4 Science and Ecosystem Support Division (SESD) database (Data Archival and ReTrieval – DART or Equis Pro interface). This database is the USEPA Region 4 repository for storing Superfund data, which includes location, geological, and analytical data. Data will be submitted in accordance with the SESD's Environmental Data Submission Guideline SESDGUID-106-RO (USEPA, 2010).

4.0 ASSESSMENT AND OVERSIGHT

Internal and external checks (assessments) have been built into this project to assure the following:

- Elements of this QAPP have been properly implemented as prescribed
- The quality of the data generated is adequate and satisfies the DQOs that have been identified in this QAPP
- Corrective actions, when needed, are implemented in a timely manner and their effectiveness is confirmed

Formal audits are not planned for this program. The FOL will provide training and oversight to field crews and review field records on a daily basis to verify that sample collection procedures and record keeping steps are being completed in accordance with the FSAP and QAPP. The USEPA may complete reviews and audits of the field sampling events at any time during the monitoring program.

If deviations from the Work Plan, this QAPP, or the FSAP are identified, the information will be verbally reported to the Project Manager and noted in the field logbook. Based on the severity of the deviation, the Project Manager might request formal documentation of the deviation in the form of a memorandum to the project file. The Project Manager will determine the timeframe required for corrective action, if corrective action is necessary. Corrective actions will be completed and an assessment of the potential impact on data quality will be made. Project reviews and summaries of issues requiring corrective actions will be summarized in the Site logbook. If potential impact to data quality is identified, a summary of the issues, corrective actions, and impacts to data will be provided to the Quality Assurance Manager for use during the validation of the analytical data. Impacts to data use will be identified in data quality reports prepared for each sampling round.

5.0 DATA VALIDATION AND USABILITY

Data validation involves reviewing and accepting, qualifying, or rejecting data based on requirements in the referenced analytical methods, data validation guidelines, and QC goals established for this project in Section 2.4. Data validation will be conducted based on procedures in the USEPA Region 4 Data Validation Standard Operating Procedures for Organic Analysis (USEPA, 2008), in conjunction with Method TO-15 SIM and the ALS Environmental TO-15 standard operating procedure (Appendix F). Project QC limits identified in Table 2 will be used as to evaluate sample results during validation. Validation will be performed by the Project Chemist, under the direction of the Quality Assurance Manager.

Data validation will consist of a systematic review of the analytical results and associated QC methods and results. In any area not specifically addressed by USEPA guidelines, best professional judgment will be utilized and described in the Usability Assessment portion of the data validation report.

In general, data validation will include a check of data completeness for data in each data package, a transcription check for sample results, and a review of laboratory reporting forms. Specifically, this review will include the following:

- Data package completeness
- Required reporting summary forms to determine whether the QC requirements were met and to determine the effect of QC requirements on the precision, accuracy, and/or sensitivity of the data
- Additional QA/QC parameters, such as field duplicates, to assess the technical usability of the data
- Application of standard data quality qualifiers to the data

In addition, each data validation effort will include a comprehensive review of the following data quality indicators:

- Sample collection, preservation, and holding times (to assess potential for degradation that could affect accuracy)
- Blanks (to assess cross-contamination)
- Surrogate/system monitoring compounds (to assess method accuracy)
- Laboratory Control Samples (to assess accuracy of the method)

- Instrument tuning and calibration
- Compound quantitation limits and method detection limits (to assess sensitivity compared to project-specific requirements)
- Field duplicate relative percent differences (to assess precision of the method relative to field sampling techniques, the specific sample matrix, and representativeness of the sample aliquot to the area sampled, if applicable)

Analytical results may be qualified by the data validator based on actions described in the USEPA validation guidelines or professional judgment. Results may be accepted without qualification or with validation qualifiers (e.g., U, J, UJ, N). Results that don't meet minimum criteria for acceptance (i.e., qualified as rejected during validation) will be unacceptable for decision making purposes. At a minimum, data rejection criteria identified in the USEPA validation documents will be applied to results.

The following validation qualifiers may be applied to sample results:

- U = target analyte is not detected above the associated detection limit
- J = the reported sample concentration is an estimate value
- UJ = the reported quantitation limit is an estimated value
- N = there is uncertainty in the identification of the reported analyte
- R = constituent rejected and unusable for detect and non-detects

The results of the data validation and any corrective actions implemented will be recorded on a QA/QC worksheet, which will be initialed and dated by the data reviewer. The Quality Assurance Manager or appropriate designee will provide secondary review of the QA/QC worksheet and will also initial and date the worksheet. The initialed and dated QA/QC worksheet will be attached to the final analytical laboratory report that is retained in the project files.

Full validation, including raw data verification and calculation checks, will be completed on the laboratory data. Results will be qualified using general procedures described in the USEPA validation guideline and the judgment of the project chemist. Upon completion of the validation task, a report will be prepared. Validation reports will be organized by sample collection task and may include be designed to report on multiple sample delivery groups.

The validation report(s) will include the following information:

- Identity of the laboratory used for analysis
- A summary of analytical methods
- A summary of samples that are included in the sample set
- A discussion of data validation actions, qualifications, and observations
- A tabulation of validated samples results

Qualifiers applied to the data during validation will be entered into the electronic data deliverables in the database.

6.0 RECONCILIATION OF DATA TO PROJECT OBJECTIVES

At the end of the project there will be an assessment of field records, field data, laboratory analytical data usability, and project completeness to determine if project objectives defined in the FSAP and QAPP have been met. The FOL and Project Manger will review field records and reports to verify completeness of field records and identify any issues regarding project procedures, collection of field data that did not meet quality objectives, the completeness of the samples collected, or corrective actions. A review of the data validation report associated with the laboratory data will also be completed by the Quality Assurance Manager in cooperation with the FOL and Project Manger to identify data that is qualified. An assessment of impacts from field issues or data qualification actions will be performed and documented in a data usability report. Impacts might include identification of entire samples or a subset of analytes where data are considered unusable. In other cases, uncertainties in the accuracy of usable data might be identified.

In the Vapor Intrusion Assessment Report, a data quality and completeness summary will be provided that identifies data gaps or analytical data quality issues that are identified in field operation reports or analytical data validation reports. The following items will be included in the data usability summary:

- Field investigation scope modifications
- Field investigation data quality issues
- Field investigation data gaps (data that was planned for collection and not obtained)
- Field and analytical data completeness
- Analytical data validation qualification actions and impacts on data usability
- Analytical detection limits and impacts on data usability

The need for recollection of sample or other actions related to the data usability will be determined on a case by case basis with input from the USEPA Project Manager.

7.0 REFERENCES

USEPA, 2006. Guidance on Systematic Planning Using the Data Quality Objectives Process (EPA QA/G-4), EPA/240/B-06/001, February 2006.

USEPA, 2008. Data Validation Standard Operating Procedures for Organic Analyses; USEPA Region 4, Science and Ecosystem Support Division Quality Assurance Section, MTSB; Athens, Georgia; August 2008.

USEPA, 2010. Communicating Environmental Data to Property Owners and Tenants (Standard Operating Procedure, Version #1), Interim Final, October 2010.

USEPA, 2010. Environmental Data Submission Guideline, Science and Ecosystem Support Division, Athens, GA; SESDGUID-106-RO, December 17, 2010.

TABLES

TABLE 1
Target Compounds and Reporting Limits
CTS of Asheville, Inc. Superfund Site
Asheville, North Carolina
AMEC Project 6252-12-0006

Analyte	CAS Number	PQL	MDL	GSL (a)	Basis of GSL Concentration	RSL (a)	Basis of RSL Concentration
cis-1,2-Dichloroethene	156-59-2	0.025	0.0055	3.5	nc	NL	NA
trans-1,2-Dichloroethene	156-60-5	0.025	0.0047	7	nc	6.3	nc
Trichloroethene	79-01-6	0.025	0.0067	0.022	c	0.43	c
Vinyl Chloride	75-01-4	0.025	0.0050	0.28	c	0.16	c

Prepared By: SEK 12/9/13
Checked By: MEW 12/9/13

Notes:
CAS - Chemical Abstracts Service
PQL - Practical Quantitative Limit
MDL - Method Detection Limit
RSL - Regional Screening Level (USEPA, November 2013)
GSL - Generic Screening Level (USEPA Office of Solid Waste and Emergency Response, Table 2c, November 2002)
nc - noncarcinogenic risk
c- carcinogenic risk
NL - analyte does not have a corresponding RSL.
Concentrations are in micrograms per cubic meter ($\mu g/m^3$)
(a) GSLs and RSLs based on noncarcinogenic (nc) hazard have been divided by ten to address additivity

TABLE 2
Project Quality Control Limits
CTS of Asheville, Inc. Superfund Site
Asheville, North Carolina
AMEC Project 6252-12-0006

Analyte	Primary Ion	Secondary Ion	Surrogate %Recovery	LCS %Recovery	Field Duplicate RPD	Lab Duplicate RPD
cis-1,2-Dichloroethene	96	98	70 - 130	62 - 130	50	25
trans-1,2-Dichloroethene	96	98	70 - 130	60 - 128	50	25
Trichloroethene	130	132	70 - 130	51 - 127	50	25
Vinyl Chloride	62	64	70 - 130	56 - 127	50	25

Notes:
LCS - laboratory control sample
RPD - relative percent difference

Prepared By: SEK 8/31/12
Checked By: CSR 8/31/12

TABLE 3
Laboratory Electronic Data Deliverable Format
CTS of Asheville, Inc. Superfund Site
Asheville, North Carolina
AMEC Project 6252-12-0006

Equis "EZEDD01" Field Name	data type	Required for "EDD"	Description	"TED" Table	"TED" Column
project_code	1 Text20	X	This field contains the internal project_code used by TED to identify a unique site. This will be provided to the lab on a per project basis.	Location	Site_id
sample_name	2 Text30	X	This field contains the sample number as written in the Analysis Request and Chain of Custody (AR/COC) form sent to the laboratory with the field samples for analysis. This is a unique number assigned to each sample by sampling personnel. For laboratory samples enter "LAB QC".	sample_collection	field_sample_id
sys_sample_code	3 Text20				
sample_date	4 Date	X	mm/dd/yyyy. Date sample was collected in the field. Date information must be identical with the date from the AR/COC form. Leave blank for lab samples. Year may be entered as yyyy.	sample_collection	field_sample_date
sample_time	5 Time				
analysis_location	6 Text2				
lab_name_code	7 Text10	X	Laboratory that performed the analysis.	sample_analysis	lab_id
lab_sample_id	8 Text20	X	Unique sample ID internally assigned by the laboratory.	sample_analysis	lab_sample_id
sample_type_code	9 Text10	X	Specifies sample type. For field samples, enter FS (regular environmental sample), otherwise, use values listed in the LOV. For example, normal field samples must be distinguished from laboratory method blank samples, etc.	sample_collection	qc_code
Lab_Del_Group	10 Text20	X	Tracking code used by the laboratory. Commonly called Sample Delivery Group (SDG).	sample_analysis	lab_sample_delivery_group
Lab_Batch_Number	11 Text20		Tracking number used by the laboratory to identify a group of samples analyzed in the same batch. This field, in conjunction with laboratory blank ID, is used to link the relationship between field samples and laboratory blank and other QC samples.		
lab_anl_method_name	12 Text35	X	Test method used in the analysis of the analyte.	sample_analysis	analysis_method

TABLE 3
Laboratory Electronic Data Deliverable Format
CTS of Asheville, Inc. Superfund Site
Asheville, North Carolina
AMEC Project 6252-12-0006

Equis "EZEDD01" Field Name	data type	Required for "EDD"	Description	"TED" Table	"TED" Column
cas_rn	13 Text15	X	Unique analyte identifier. Use assigned CAS number when one is identified for an analyte. Tentatively Identified Compounds (TICs) and a number of other analytes are not assigned a standard CAS number. The laboratory is required to assign a UNIQUE identifier for all chemical_names.	sample_analysis_results	casno
chemical_name	14 Text60	X	Name of analyte or parameter analyzed.		
result_value	15 Text20	X	Must only be a numeric value. It is stored as a string of characters so that significant digits can be retained. Must be identical with values presented in the hard copy. Analytical result is reported left justified. Reported as the reporting_detection_limit for non-detects.	sample_analysis_results	lab_result
lab_qualifiers	16 Text7	X	Qualifier flags assigned by the laboratory.	sample_analysis_results	lab_qualifier
result_unit	17 Text15	X	This format assumes that the result value and detect limit have the same units.	sample_analysis_results	result_uom
result_type_code	18 Text10	X	Parameter list type. Valid Values = Targer analytes (TCL, TAL or TRG); Surrogates (SUR); and TICs	sample_analysis_results	result_type
detect_flag	19 Text2	X	Enter “Y” for detected analytes or “N” for non-detected analytes.	sample_analysis_results	report_hit_flag
reporting_detection_limit	20 Text20	X	Must only be a numeric value. Use the value of the Reported Detection Limit (RDL), Practical Quantitation Limit (PQL), or Contract Required Quantitation Limit. Value is stored as a string to retain significant figures. Unit of measure must be identical with result_unit value.	sample_analysis_results	detection_limit
dilution_factor	21 Text6	X	Must be a numeric entry. The factor by which the sample was diluted as part of the preparation process. If no dilution was done, enter the value 1. Value is stored as a string to retain significant figures.	sample_analysis	dilution_factor
sample_matrix_code	22 Text10	X	Code which distinguishes between different type of sample matrix. For example, soil samples must be distinguished from ground water samples, etc. Valid codes for HESE are "G" (gas), "L" (liquid), "S" (solid), and "P" (free or raw liquid product).	sample_collection	matrix

TABLE 3
Laboratory Electronic Data Deliverable Format
CTS of Asheville, Inc. Superfund Site
Asheville, North Carolina
AMEC Project 6252-12-0006

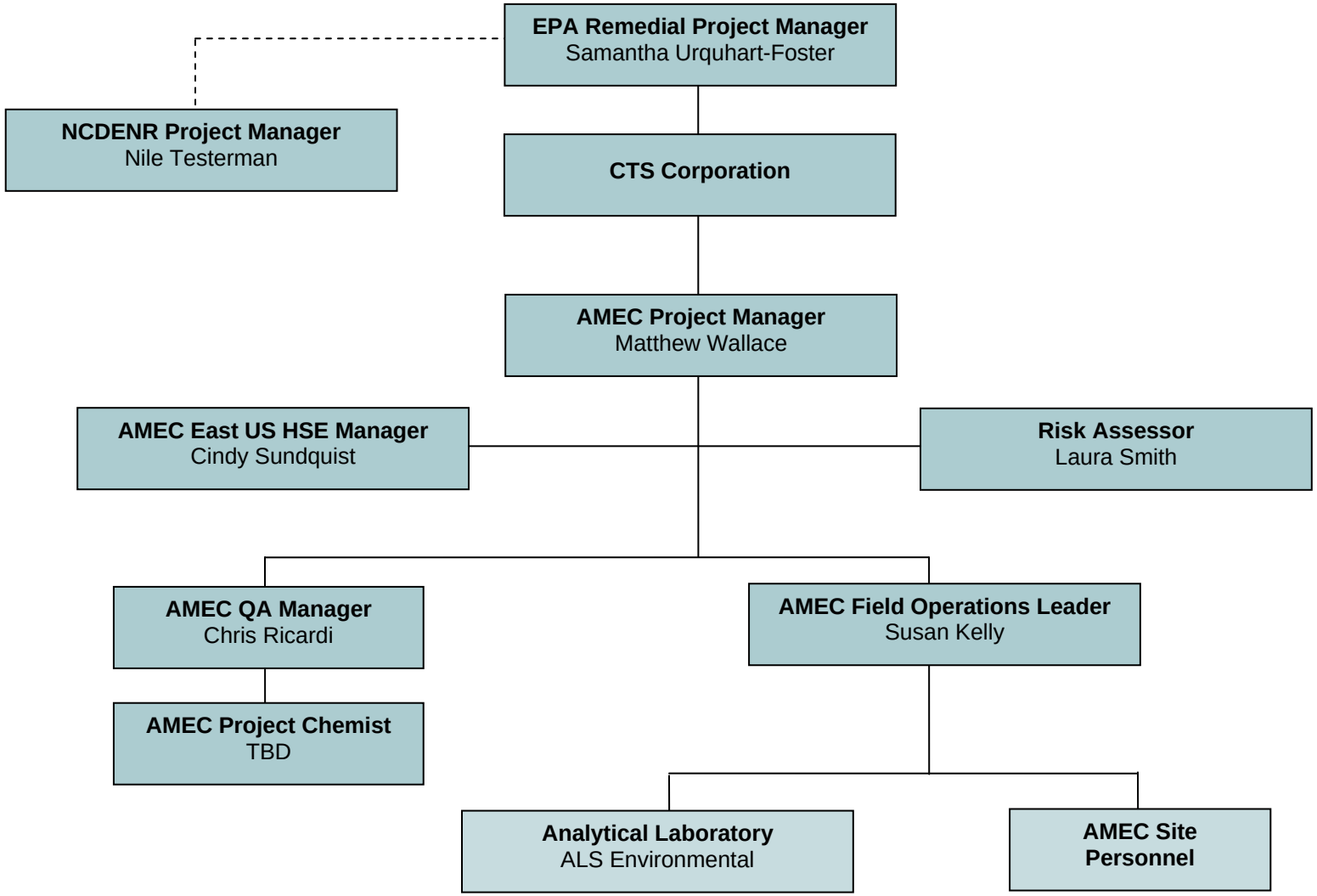
Equis "EZEDD01" Field Name	data type	Required for "EDD"	Description	"TED" Table	"TED" Column
total_or_dissolved (or fraction)	23 Text1	X	Must be “T” for total metal concentration, “D” for dissolved or filtered metal concentration, or “N” for organic (or other) parameters for which neither “total” nor “dissolved” is applicable. Also, HESE requires "C" for TCLP and "S" for SPLP fractions.	sample_analysis	fraction
basis	24 Text10				
analysis_date	25 Date	X	mm/dd/yyyy. Date sample was analyzed.	sample_analysis	analysis_date
analysis_time	26 Time				
method_detection_limit	27 Text20				
lab_prep_method_name	28 Text35		Description of sample preparation or extraction method.	sample_analysis	prep_method_name
prep_date	29 Date	X	mm/dd/yyyy. This field is used to determine whether holding times for field samples have been exceeded.	sample_analysis	extraction_date
prep_time	30 Time				
test_batch_id	31 Text20	X	Default is 1 for primary results. Other valid values are 2, 3, 4, 5, and RE. Primary use is for reanalyses and dilutions where more than one result may be reported.	sample_analysis	run_id
result_error_delta	32 Text20				
TIC_retention_time	33 Text8				
qc_level	34 Text10		Laboratory QC level associated with the analysis	sample_analysis	qc_level
result_comment	35 Text255		Any comments related to the analysis.	sample_analysis_results	comments
sample_quantitation_limit (may be REQUIRED FIELD for certain projects)	36 Text20		Must only be a numeric value. Use the value of the Sample Quantitation Limit (SQL). Value is stored as a string to retain significant figures. Unit of measure must be identical with result_unit value.	sample_analysis_results	TBD

Note: All "X" marked fields are minimum data required to load data to "TED".

APPENDIX A

ORGANIZATION CHART

Organization Chart



APPENDIX B

ALS ENVIRONMENTAL
QUALITY ASSURANCE MANUAL

QUALITY ASSURANCE MANUAL

ALS Environmental
Simi Valley Facility
2655 Park Center Drive, Suite A
Simi Valley, California 93065
805-526-7161 (T)
805-526-7270 (F)
www.alsglobal.com

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QUALITY ASSURANCE MANUAL

SOPID:	ALSSV-QAM	Rev. Number:	26.0	Effective Date:	12/22/2012

Approved By: _____ Date: 12/11/12
Technical Director (Wet / Gen) – Sue Anderson

Approved By: _____ Date: 12/11/12
Technical Director (SVOA / IH) – Madeleine Dangazyan

Approved By: _____ Date: 12/11/12
Technical Director (VOA GC Air) – Wade Henton

Approved By: _____ Date: 12/11/12
Technical Director (VOA GC/MS Air) – Chris Parnell

Approved By: _____ Date: 12/11/12
QA Manager – Chaney Humphrey

Approved By: _____ Date: 12/11/12
Laboratory Director – Kelly Horiuchi

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1) Introduction and Company Quality Assurance Policy

The purpose of this Quality Assurance Manual is to outline the quality system for the Simi Valley location of ALS Environmental. Columbia Analytical Services, Inc. was purchased in November of 2011 and at that time was registered as Columbia Analytical Services, Inc. dba ALS Environmental. The laboratory shall be referred to as ALS Environmental-Simi Valley throughout this QA Manual.

ALS Environmental is a professional analytical services laboratory which performs chemical and microbiological analyses on a wide variety of sample matrices, including drinking water, groundwater, surface water, wastewater, soil, sludge, sediment, tissue, industrial and hazardous waste, air, and other material.

We recognize that quality assurance requires a commitment to quality by everyone in the organization - individually, within each operating unit, and throughout the entire laboratory. Laboratory management is committed to ensuring the effectiveness of its quality systems and to ensure that all tests are carried out in accordance to customer requirements. Key elements of this commitment are set forth in the *SOP for Laboratory Ethics and Data Integrity* and in this Quality Assurance Manual (QAM). ALS Environmental is committed to operate in accordance with these requirements and those of regulatory agencies, accrediting authorities, and certifying organizations. The laboratory also strives for improvement through varying continuous improvement initiatives and projects.

Quality Management Systems are established, implemented and maintained by management. Policies and procedures are established in order to meet requirements of accreditation bodies and applicable programs as well as client's quality objectives. The laboratory's management is committed to complying with the National Environmental Laboratory Accreditation Conference (NELAC) Quality Systems Standards (2003 and 2009), ISO/IEC 17025:2005, and the Department of Defense (DoD) Quality Systems Manual for Environmental Laboratories. Systems are designed so that there will be sufficient Quality Assurance (QA) activities conducted in the laboratory to ensure that all analytical data generated and processed will be scientifically sound, legally defensible, of known and documented quality, and will accurately reflect the material being tested. Quality Systems are applicable to all fields of testing in which the laboratory is involved. All personnel involved with environmental testing and calibration activities must familiarize themselves with the quality documentation and implement the policies and procedures in their work.

Quality Control (QC) procedures are used to continually assess performance of the laboratory and quality systems. ALS Environmental maintains control of analytical results by adhering to written standard operating procedures (SOPs), using analytical control parameters with all analyses, and by observing sample custody requirements. All analytical results are calculated and reported in units consistent with project specifications to allow comparability of data.

This QAM is applicable to the facility listed on the title page and the off-site extraction facility located at 2360 Shasta Way, Unit G, Simi Valley California. The information in this QAM has been organized according to requirements found in the National Environmental Laboratory Accreditation Program (NELAP) Quality Systems Standards (2003 and 2009), the EPA Requirements for Quality Assurance Project Plans, EPA QA/R-5, USEPA, 2001;

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and *General Requirements for the Competence of Testing and Calibration Laboratories*, ISO/IEC 17025:2005.

2) Program Description

The purpose of the QA program at ALS Environmental is to ensure that our clients are provided with analytical data that is scientifically sound, legally defensible, and of known and documented quality.

2.1 Quality Management Systems

The laboratory has developed a Quality Management System to ensure all products and services meet our client's needs. The system is implemented and maintained by the Quality Assurance Manager (QA Manager) with corporate oversight by the Corporate Quality Assurance Manager (CQAM). These systems are based upon ISO 17025:2005 standards, upon which fundamental programs (AIHA, NELAC 2003, 2009 and DoD QSM) are based. Implementation and documentation against these standards are communicated in corporate policy statements, this QAM, and SOPs. Actual procedures, actions and documentation are defined in both administrative and technical SOPs. Figure 2-1 shows the relationships of the quality systems and associated documentation. Quality systems include:

- Standard Operating Procedures
- Sample Management and Chain of Custody procedures
- Statistical Control Charting
- Standards Traceability
- Ethics Training
- Document Control
- Corrective Action Program
- Management Reviews
- Demonstration of Capability

The effectiveness of the quality system is assessed in several ways, including:

- Internal and External Audits covering all aspects of the organization
- Annual Management Reviews
- Analysis of Customer Feedback
- Internal and External Proficiency Testing

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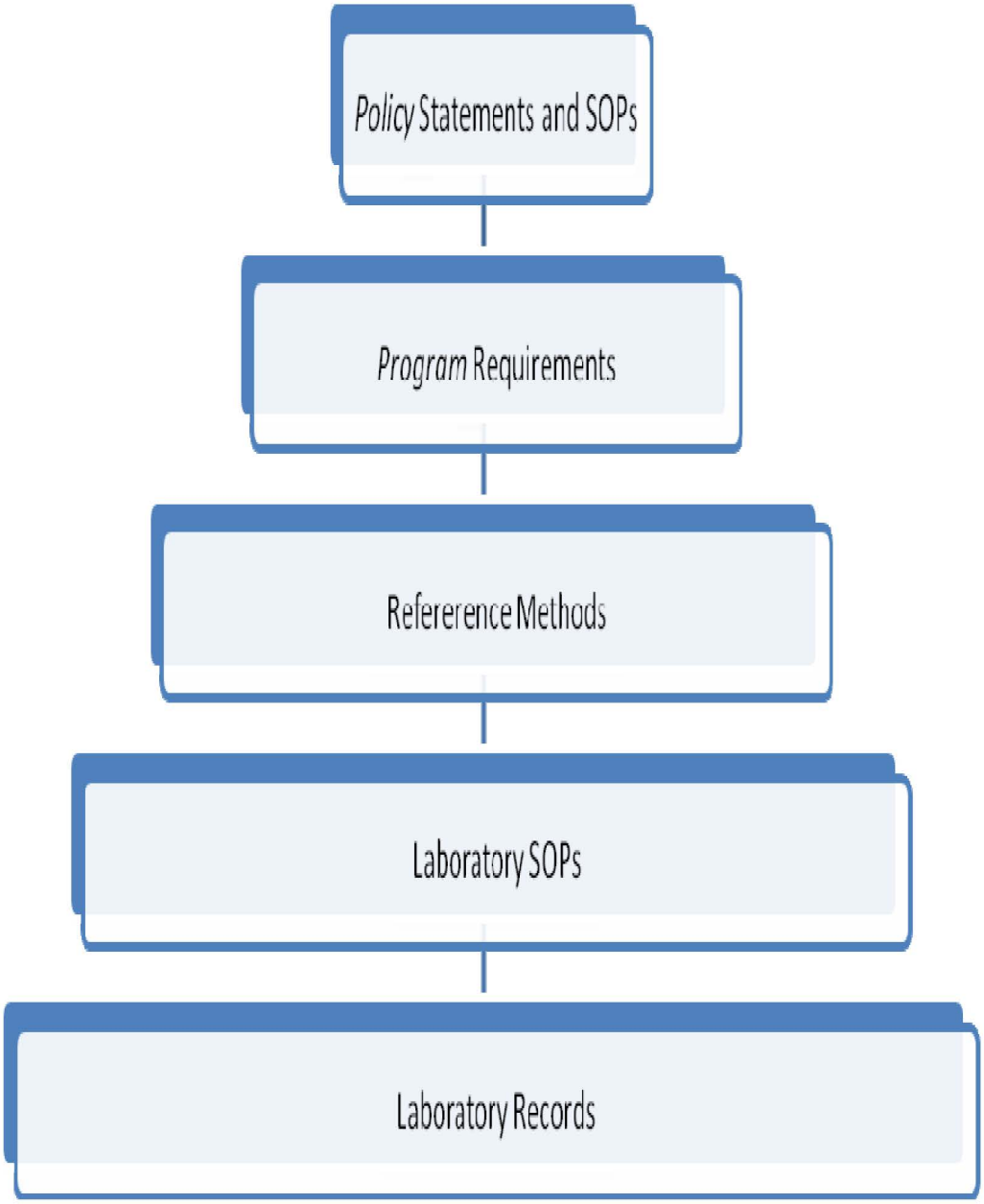
Figure 2-1

Relationships of Quality Management Systems and Documentation



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2.2 Facilities, Equipment, and Security

ALS Environmental-Simi Valley maintains approximately 20,000 square feet of laboratory and administrative workspace. The laboratory has been designed and constructed to provide safeguards against cross-contamination of samples and is arranged according to work function, which enhances the efficiency of analytical operations. The ventilation system is designed to meet any needs of analyses performed in the separate work areas. ALS Environmental-Simi Valley minimizes laboratory contamination sources by employing janitorial staff to ensure good housekeeping. In addition, the segregated laboratory areas are designed for safe and efficient handling of a variety of sample types. These specialized areas (and access restrictions) include:

- Sample Management Office; Shipping and Receiving
- Records Archival
- Volatile Organics Laboratory (GC and GC/MS)
- Semi-Volatiles Laboratory (GC, GC/MS and HPLC)
- Ultra Low Level Volatile Organics GC/MS
- General/Wet Chemistry Laboratory
- R&D Laboratory
- Canister Conditioning and Maintenance
- Flow Controller and Critical Orifice Calibration Station
- Sample Storage Walk-in Refrigerator
- Sample, Standards, and Media Storage
- Waste Disposal
- Laboratory Deionized Water System
- Laboratory Management, Client Service, Report Generation and Administration
- Information Technology (IT)

The designated areas for sample receiving, refrigerated sample storage, dedicated sample container preparation and shipping provide for the efficient and safe handling of a variety of sample types. Figure 2-2 shows the facility floor plan. The laboratory is equipped with state-of-the-art analytical and administrative support equipment. The equipment and instrumentation are appropriate for the procedures in use. Appendix C lists the major equipment, illustrating the laboratory's overall capabilities and depth.

ALS Environmental-Simi Valley also maintains a satellite extraction facility located at 2360 Shasta Way, Unit G, Simi Valley, California. The approximately 2,000 square foot building contains five fume hoods and is designed with the purpose of performing semi-volatile organics extraction of air, liquid and solid matrices. The extraction facility is equipped with sufficient bench space, glassware washing equipment and materials, flammable solvent storage, sample/extract storage refrigerators and an electric kiln. Refer to Figure 2-3 for the floor plan of the facility.

2.3 Technical Elements of the Quality Assurance Program

The laboratory's technical procedures are based upon procedures published by various agencies or organizations (See Section 16). The Quality Assurance Program provides laboratory organization, procedures, and policies by which the

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laboratory operates. The necessary certifications and approvals administered by external agencies are maintained by the QA department. This includes method approvals and audit administration. In addition, internal audits are performed to assess compliance with policies and procedures. SOPs are maintained for technical and administrative functions. A document control system is used for SOPs, as well as laboratory notebooks, and this QA Manual. A list of QA Program documents is provided in Appendix A and SOPs in Appendix F.

Acceptable calibration procedures are defined in the SOP for each test procedure. Calibration procedures for other laboratory equipment (balances, thermometers, etc.) are also defined. Quality Control (QC) procedures are used to monitor the testing performed. Each analytical procedure has associated QC requirements to be achieved in order to demonstrate data quality. The use of method detection limit studies, control charting, technical training and preventive maintenance procedures further ensure the quality of data produced. Proficiency Testing (PT) samples are used as an external means of monitoring the quality and proficiency of the laboratory. PT samples are obtained from qualified vendors and are performed on a regular basis. In addition to method proficiency, documentation of analyst training is performed to ensure proficiency and competency of laboratory analysts and technicians. Sample handling and custody procedures are defined in SOPs. Procedures are also in place to monitor the sample storage areas. The technical elements of the QA program are discussed in further detail in later sections of this QA manual.

2.4 Operational Assessments and Service to the Client

The laboratory uses a number of systems to assess its daily operations. In addition to the routine quality control (QC) measurements, the senior laboratory management examines a number of other indicators to assess the overall ability of the laboratory to successfully perform analyses for its clients including; on-time performance, customer complaints, training reports and non-conformity reports. A frequent, routine assessment must also be made of the laboratory's facilities and resources in anticipation of accepting an additional or increased workload.

ALS Environmental utilizes a number of different methods to ensure that adequate resources are available for service demands. Senior staff meetings, tracking of outstanding proposals and an accurate, current synopsis of incoming work all assist the senior staff in properly allocating sufficient resources. All Requests for Proposal (RFP) documents are reviewed by Project Managers, Business Development and appropriate managerial staff to identify any project specific requirements that differ from the standard practices of the laboratory. Any requirements that cannot be met are noted and communicated to the client, as well as requesting the client to provide any project specific Quality Assurance Project Plans (QAPPs) if available. Status/production meetings are also conducted regularly with the laboratory and project managers to inform the staff of the status of incoming work, future projects, or project requirements.

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When a customer requests a modification to an SOP, policy, or standard specification the Project Manager will discuss the proposed deviation with the Laboratory Director and department manager to obtain approval for the deviation. The QA Manager may also be involved. All project-specific requirements must be on-file and with the service request upon logging in the samples. The modification or deviation must be documented. A Project-Specific Communication Form, Form V, LIMS comments, or similar, may be used to document such deviations.

The laboratory shall afford clients cooperation to clarify the client's request and to monitor the laboratory's performance in relation to the work performed, provided that the laboratory ensures confidentiality to other clients. The laboratory maintains and documents timely communication with the client for the purposes of seeking feedback and clarifying customer requests. Feedback is used and analyzed to improve the quality of services. The *SOP for Handling Customer Feedback* (ADM-FDBK) is in place for these events.

2.5 Document Control and Records

Procedures for control and maintenance of documents are described in the *SOP for Document Control* (CE-GEN005). The requirements of the SOP apply to all laboratory logbooks (standards, maintenance, run logbooks, etc), certificates of analysis, SOPs, QAMs, quality assurance project plans (QAPPs), Environmental Health & Safety (EHS) manuals, and other controlled ALS Environmental documents.

The contents of this manual are reviewed, revised (as needed) and approved for use at least annually by authorized personnel (QA Manager, Laboratory Director and Team Leaders) where the scope of the review ensures that it continuously reflects current policies and practices and incorporates all applicable requirements. Additionally, the date the review was completed is indicated by the date of the last approval signature on the title page.

Each controlled copy of a controlled document will be released only after a document control number is assigned and the recipient is recorded on a document distribution list. Filing and distribution is performed by the QA Manager, or designee, and ensures that only the most current version of the document is distributed and in use. A document control number is assigned to logbooks. Completed logbooks that are no longer in use are archived in a master logbook file. Logbook entries are standardized following the *SOP for Making Entries Onto Analytical Records* (CE-QA007). The entries made into laboratory logbooks are reviewed and approved at a regular interval (quarterly).

A records system is used which ensures all laboratory records (including raw data, reports, and supporting records) are retained and available. The archiving system is described in the *SOP for Data and Record Archiving* (ADM-ARC).

External documents relative to the management system are managed by the QA Manager. To prevent the use of invalid and/or outdated external documents, the laboratory maintains a master list of current documents and their availability. The list is reviewed before making the documents available. External documents are not issued to personnel.

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2.6 Subcontracting

Analytical services are subcontracted when the laboratory needs to balance workload or when the requested analyses are not performed by the laboratory. Subcontracting, to capable qualified laboratories is only done with the knowledge and approval of the client. Subcontracting to another ALS Environmental laboratory is preferred over external-laboratory subcontracting. Established procedures are used to qualify external subcontract laboratories. These procedures are described in the *SOP for Qualification of Subcontract Laboratories* (CE-QA004). The Corporate Quality Assurance staff is responsible for maintaining a list of qualified subcontract laboratories.

2.7 Procurement

The quality level of reagents and materials (grade, traceability, etc.) required is specified in the analytical SOPs. Department supervisors ensure that the proper materials are purchased. Inspection and verification of material ordered is performed at the time of receipt by receiving personnel. The receiving staff labels the material with the date received. Expiration dates are assigned as appropriate for the material. Storage conditions and expiration dates are specified in the analytical SOP. The *SOP for Handling Consumable Materials* (ADM-CONSUM) provides default expiration requirements. Supplies and services that are critical in maintaining the quality of laboratory testing are procured from pre-approved vendors. The policy and procedure for purchasing and procurement are described in the *SOP for Procurement and Control of Laboratory Services and Supplies* (CE-GEN007). Also, refer to section 8.6 for a discussion of reference materials.

Receipt procedures include technical review of the purchase order/request to verify that what was received is identical to the item ordered. The laboratory checks new lots of reagents for unacceptable levels of contamination prior to use in sample preservation, sample preparation, and sample analysis by following the *SOP for Checking New Lots of Chemicals for Contamination* (ADM-CTMN).

2.8 Review of Requests, Tenders and Contracts (Procedures for Accepting New Work)

Requests for new work are reviewed prior to signing any contracts or otherwise agreeing to perform the work. The specific methods to be used are agreed upon between the laboratory and the client. A capability review is performed to determine if the laboratory has or needs to obtain certification to perform the work, to determine if the laboratory has the resources (personnel, equipment, materials, capacity, skills, expertise) to perform the work, and if the laboratory is able to meet the client's required reporting and QC limits. The results of this review are communicated to the client and any potential conflict, deficiency, lack of appropriate accreditation status, or concerns of the ability to complete the client's work are resolved. Any differences between the request or tender and the contract shall be resolved before any work commences. The client should be notified at this time if work is expected to be subcontracted. Each contract shall be acceptable both to the laboratory and the client. Records are maintained of pertinent discussions with a client relating to the client's requirements or the results of the work. If a contract needs to be amended after work has commenced, the contract review process is repeated and any amendments are

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communicated to all affected personnel. Changes in accreditation status affecting ongoing projects must be reported to the client.

Figure 2-2
ALS Environmental-Simi Valley Laboratory Floor Plan

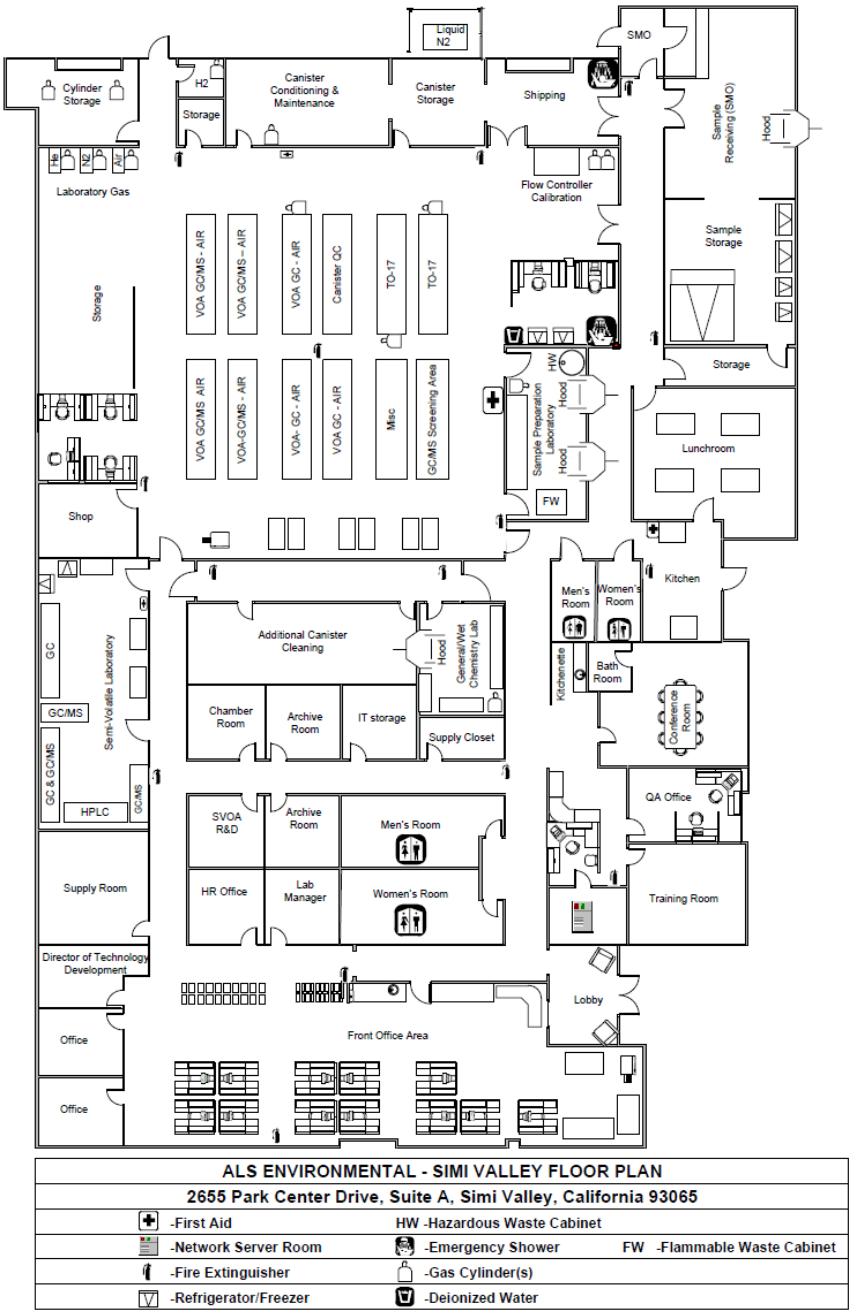

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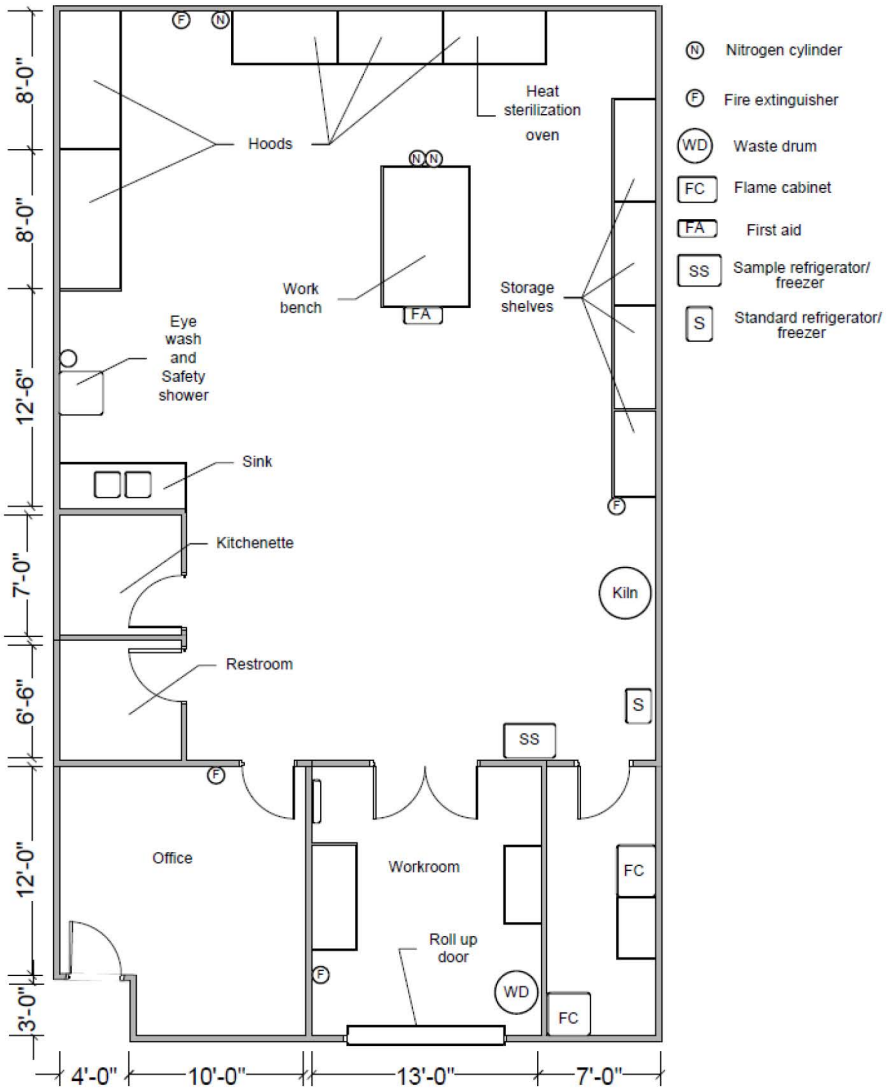
Figure 2-3
ALS Environmental-Simi Valley Extraction Laboratory Floor Plan

Extraction Laboratory for
ALS Environmental

2360 Shasta Way, Unit G
Simi Valley, CA. 93065



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3) Professional Conduct, Data Integrity, and Ethics

One of the most important aspects of the success of ALS Environmental is the emphasis placed on the integrity of the data provided and the services rendered. This success is reliant on both the professional conduct of all employees within ALS Environmental as well as established laboratory practices.

3.1 Professional Conduct

To promote quality, ALS Environmental requires certain standards of conduct and ethical performance among employees. The following examples of documented ALS Environmental policy are representative of these standards, and are not intended to be limiting or all-inclusive:

- Under no circumstances is the willful act of fraudulent manipulation of analytical data condoned. Such acts are to be reported immediately to senior management for appropriate corrective action.
- Unless specifically required in writing by a client, alteration, deviation or omission of written contractual requirements is not permitted. Such changes must be in writing and approved by senior management.
- Falsification of data in any form will not be tolerated. While much analytical data is subject to professional judgment and interpretation, outright falsification, whenever observed or discovered, will be documented, and appropriate remedies and punitive measures will be taken toward those individuals responsible.
- It is the responsibility of all ALS Environmental employees to safeguard sensitive company information, client data, records, and information; and matters of national security concern should they arise. The nature of our business and the well being of our company and of our clients is dependent upon protecting and maintaining proprietary company/client information. All information, data, and reports (except that in the public domain) collected or assembled on behalf of a client is treated as confidential. Information may not be given to third parties without the consent of the client. Unauthorized release of confidential information about the company or its clients is taken seriously and is subject to formal disciplinary action. All employees sign a confidentiality agreement upon hire to protect the company and client's confidentiality and proprietary rights.

3.2 Prevention and Detection of Improper, Unethical or Illegal Actions

It is the intention of ALS Environmental to proactively prevent and/or detect any improper, unethical or illegal action conducted within the laboratory. This is performed by the implementation of a program designed for not only the detection but also prevention. Prevention consists of educating all laboratory personnel of their roles and duties as employees, company policies, inappropriate practices, and their corresponding implications as described here.

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In addition to education, appropriate and inappropriate practices are included in SOPs such as manual integration, data review and specific method procedures. Electronic and hardcopy data audits are performed regularly, including periodic audits of chromatographic electronic data. Requirements are described in the *SOP for Internal Audits* (CE-QA001) and details are listed in laboratory administrative SOPs. All aspects of this program are documented and retained on file according to the company policy on record retention.

The *SOP for Laboratory Ethics and Data Integrity* (CE-GEN001) also contains information on the ALS Environmental ethics and data integrity program, including mechanisms for reporting and seeking advice on ethical decisions.

3.3 Laboratory Data Integrity and Ethics Training

Each employee receives in-depth (approximately 2-4 hour) core Data Integrity/Ethics Training. New employees are given a QA and Ethics orientation within the first month of hire, followed by the core training within 1 year of hire. On an ongoing basis, all employees receive annual ethics refresher training. Topics covered are documented in writing and all training is documented. It is the responsibility of the QA Manager to ensure that the training is conducted as described.

Key topics covered are the organizational mission and its relationship to the critical need for honesty and full disclosure in all analytical reporting, how and when to report data integrity issues and record keeping. Training includes discussion regarding all data integrity procedures, data integrity training documentation, in-depth data monitoring and data integrity procedure documentation.

Data integrity training provides assurance that a highly ethical approach to testing is a key component of all laboratory planning, method implementation, and training. There are four elements to the laboratory's procedures for data integrity. These include:

1) Data integrity training (conducted initially and at least annually);
2) Signed data integrity documentation for all employees;
3) In-depth periodic monitoring of data integrity;
4) Data integrity procedure documentation (*SOP for Laboratory Ethics and Data Integrity* (CE-GEN001)).

There is specific emphasis on the importance of proper written narration on the part of the analyst with respect to those cases where analytical data may be useful, but are in one sense or another partially deficient. A signature attestation sheet of data integrity training including their understanding of their obligations related to data integrity and as specified in the training is generated for attendees and maintained on file for review. Trainees are required to understand that any infractions of the laboratory data integrity procedures will result in a detailed investigation that could lead to very serious consequences including immediate termination, or civil/criminal prosecution.

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The training session includes many concepts and topics, numerous examples of improper actions (defined by DoD as deviations from contract-specified or method-specified analytical practices and may be intentional or unintentional), legal and liability implications (company and personal), causes, prevention, awareness, and reporting mechanisms.

3.4 Management and Employee Commitment

ALS Environmental makes every attempt to ensure that employees are free from any commercial, financial, or other undue pressures that might affect their quality of work. Related policies are described in the *SOP for Laboratory Ethics and Data Integrity* (CE-GEN001). This includes:

- ALS Environmental Open Door Policy – Employees are encouraged to bring any work related problems or concerns to the attention of local management or their Human Resources representative. However, depending on the extent or sensitivity of the concern, employees are encouraged to directly contact any member of upper management.
- FAIRCALL – An anonymous and confidential reporting system available to all employees that is used to communicate misconduct and other concerns. The program shall help minimize negative morale, promote a positive work place, and encourage reporting suspected misconduct without retribution. Associated upper management is notified and the investigations are documented.
- Use of flexible work hours. Within reason and as approved by supervisors, employees are allowed flexible work hours in order to help ease schedule pressures which could impact decision-making and work quality.
- Operational and project scheduling assessments are continually made to ensure that project planning is performed and that adequate resources are available during anticipated periods of increased workloads. Procedures for subcontracting work are established, and within the ALS Environmental laboratory network additional capacity is typically available for subcontracting, if necessary.
- Gifts and Favors (Code of Conduct Agreement) – To avoid possible conflict of interest implications, employees do not receive unusual gifts or favors to, nor accept such gifts or favors from, persons outside the Company who are, or may be, in any way concerned with the projects on which the Company is professionally engaged.

All employees are required to sign and adhere to the requirements set forth in the *Code of Conduct Agreement, Confidentiality Agreement,* and *Ethics and Data Integrity Agreement.* The *Ethics and Data Integrity Agreement* is signed by all employees on an annual basis (see Appendix A).

4) Organization and Responsibilities

The ALS Environmental-Simi Valley staff, consisting of approximately 36 employees, includes chemists, technicians and support personnel. They represent diverse educational backgrounds, experience, and provide the comprehensive skills that the laboratory requires. As seasonal workload increases, temporary employees may be hired to perform specific tasks.

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ALS Environmental is committed to providing an environment that encourages excellence. All employees share the responsibility for maintaining and improving the quality of our analytical services. The responsibilities of key personnel within the laboratory are described below. Table 4-1 lists the ALS Environmental-Simi Valley personnel assigned to these key positions. Managerial staff members are provided the authority and resources needed to perform their duties. An organizational chart of the laboratory, as well as the resumes of key personnel, can be found in Appendix B.

- The role of the **Laboratory Director** is to provide technical, operational, and administrative leadership through planning, allocation and management of personnel and equipment resources. The Laboratory Director provides leadership and support for the QA program and is responsible for overall laboratory efficiency and the financial performance of the Simi Valley facility. The Laboratory Director has the authority to stop work in response to quality problems. The Laboratory Director also provides resources for implementation of the QA program, reviews and approves this QA Manual, reviews and approves standard operating procedures (SOPs), and provides support for business development by identifying and developing new markets through continuing support of the management of existing client activities.

- The **Quality Assurance Manager** (QA Manager) has the authority and responsibility for implementing, maintaining, and improving the quality system. This includes coordination of QA activities within the laboratory, ensuring that all personnel understand their contributions to the quality system, ensuring communication takes place at all levels within the laboratory regarding the effectiveness of the quality system, evaluating the effectiveness of training; and monitor trends and continually improve the quality system. Audit and surveillance results, control charts, proficiency testing results, data analysis, corrective and preventive actions, customer feedback, and management reviews can all be used to support quality system implementation. The QA Manager is responsible for ensuring compliance with NELAC standards (and ISO, DoD QSM, etc. as applicable). The QA Manager works with laboratory staff to establish effective quality control and assessment plans and has the authority to stop work in response to quality problems. The QA Manager is responsible for maintaining the QA Manual and performing an annual review of it; reviewing and approving SOPs and ensuring the annual review of technical SOPs; maintaining QA records such as metrological records, archived logbooks, PT results, etc.; document control; conducting PT sample studies; approving nonconformity and corrective action reports; maintaining the laboratory's certifications and approvals; and performing internal QA audits.

  The QA Manager reports directly to the Laboratory Director and also reports indirectly to the Corporate Quality Assurance Manager. It is important to note that when evaluating data, the QA Manager does so in an objective manner and free of outside, or managerial, influence.

- The Corporate Quality Assurance Manager (CQAM) is responsible for the overall QA program at all the ALS Environmental USA laboratories. The CQAM is responsible for oversight of QA Managers regulatory compliance efforts (NELAC, ISO, DoD, etc). The CQAM or designee performs annual internal audits at each laboratory; maintains a

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database of laboratory certification/accreditation programs; approves company-wide SOPs; provides assistance to the laboratory QA staff and laboratory managers; etc.

- In the case of absence of the Laboratory Director or QA Manager, deputies are assigned to act in that role. Default deputies for these positions are a Project Manager or Volatile Organics Technical Manager (for the Laboratory Director) and the CQAM or Laboratory Director (for the QA Manager).
- In the event that work is stopped in response to quality problems, only the Laboratory Director or QA Manager have the authority to resume work.

- The **Environmental Health and Safety Coordinator** (EH&S) is responsible for the administration of the laboratory health and safety policies. This includes the formulation and implementation of safety policies, the supervision of new-employee safety training, the review of accidents, incidents and prevention plans, the monitoring of hazardous waste disposal and the conducting of departmental safety inspections. The EH&S Coordinator is also designated as the Chemical Hygiene Officer. The EH&S Coordinator has a dotted-line reporting responsibility to ALS's Environmental EH&S Director.
- The **Data Validation Coordinator/Reporting Supervisor** is responsible for data review, data package preparation, review and coordination, and preparation of case narratives (based on the information provided by the laboratory).
- The **Client Services Manager** is responsible for the Client Services Department defined for the laboratory (i.e. Project Managers, data reporting, etc.) and the sample management office/bottle preparation sections. The Client Services Department provides a complete interface with clients from initial project specifications to final deliverables. Sample management handles all activities associated with receiving, storage, and disposal of samples. The Client Services Manager has the authority to stop subcontractor work in response to quality problems.
- The **Project Manager** is a scientist assigned to each client to act as a technical liaison between the client and the laboratory. The Project Manager is responsible for ensuring that the analyses performed by the laboratory meet all project, contract, and regulatory-specific requirements. This entails coordinating with the ALS Environmental laboratory and administrative staff to ensure that client-specific needs are understood and that the services ALS Environmental provides are properly executed and satisfy the requirements of the client.
- The Analytical Laboratory is divided into operational units based upon specific disciplines. Each department is responsible for establishing, maintaining and documenting a QC program meeting department needs. Each **Department Manager and Supervisor** has the responsibility to ensure that QC functions are carried out as planned, and to guarantee the production of high quality data. Department managers and bench-level supervisors have the responsibility to monitor the day-to-day operations to ensure that productivity and data quality objectives are met. Each department manager has the authority to stop work in response to quality problems in their area. Analysts have the responsibility to carry out testing according to

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prescribed methods, SOPs, and quality control guidelines particular to the laboratory in which he/she is working.

- The **Sample Management Office** plays a key role in the laboratory QA program by performing and/or assisting in the proper preparation and shipment of sampling media. In addition, personnel are responsible for the verification of sample receipt information, performing sample acceptance and log-in and distribution of documentation per laboratory defined procedures and the initial storage of samples in the proper environment and location and performing proper sample disposal. Responsibilities also include monitoring and recording of critical thermal preservation equipment temperatures and calibration of associated thermometers against NIST traceable thermometers.
- **Information Technology** (IT) staff is responsible for the administration of the Laboratory Information Management System (LIMS) and other necessary support services. Other functions of the IT staff include laboratory network maintenance, IT systems development and implementation, education of analytical staff in the use of scientific software, Electronic Data Deliverable (EDD) generation, and data back-up, archival and integrity operations.

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Table 4-1
Summary of Technical Experience and Qualifications

Personnel	Years of Experience	Project Role
Kelly Horiuchi, B.A.	(b)(4) [redacted]	Laboratory Director / Project Manager
Chaney Humphrey, B.S.		Quality Assurance Manager
Robin Gill		Data Validation Coordinator / Reporting Supervisor
Ku-Jih Chen, B.S.		Principle Chemist
Sue Anderson, B.S.		General (WET) Chemistry Technical Manager / Project Manager
Samantha Henningsen, B.S.		Project Manager
Kathleen Aguilera, B.A.		Client Services Manager / Project Manager
Wade Henton, B.S.		Volatiles (GC) Team Leader / Technical Manager
Chris Parnell, B.S.		Operations Manager / Volatiles (GC/MS) Technical Manager
Wida Ang, B.S.,M.S.		Volatiles (GC/MS) Team Leader
Madeleine Dangazyan, B.S.		Semi-Volatiles / Industrial Hygiene Technical Manager / Team Leader
Manny Zamora		Sample Management Team Leader
Jeff Christian, B.S.		Director of Operations – Western U.S.

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5) Information Management

The generation, compilation, reporting, and archiving of electronic data is a critical component of laboratory operations. In order to generate data of known and acceptable quality, the quality assurance systems and quality control practices for electronic data systems must be complete and comprehensive and in keeping with the overall quality assurance objectives of the organization. ALS Environmental management provides the tools and resources to implement electronic data systems and establishes information technology standards and policies.

5.1 Software Quality Assurance Plan

ALS Environmental has defined practices for assuring the quality of the computer software used throughout all laboratory operations to generate, compile, report, and store electronic data. These practices are described in the *Software Quality Assurance Plan (SQAP)*. The purpose of the SQAP is to describe the policies and practices for the procurement, configuration management, development, validation and verification, data security, maintenance, and use of computer software. The policies and practices described in the plan apply to purchased computer software as well as to internally developed computer software. Key components of this plan are policies for software validation and control.

5.2 IT Support

The local ALS Environmental Information Technology (IT) department is established to provide technical support for all computing systems. The IT department staff continually monitors the performance and output of operating systems. The IT department oversees routine system maintenance and data backups to ensure the integrity of all electronic data described in the *SOP for Tape Backup and Tape Archiving* (ADM-TapeBU). A software inventory is maintained. Additional IT responsibilities are described in the SQAP and the *SOP for Software and Data Quality Assurance* (ADM-SftwreQA).

In addition to the local IT department, ALS Environmental corporate IT provides support for network-wide systems. ALS Environmental also has personnel assigned to information management duties such as development and implementation of reporting systems; data acquisition, and Electronic Data Deliverable (EDD) generation.

5.3 Information Management Systems

ALS Environmental has various systems in place to address specific data management needs. The Laboratory Information Management System (LIMS) is used to manage sample information and invoicing. Access is controlled by password. This system defines sample identification, analysis specifications, and provides a means of sample tracking. This system is used during sample login to generate the internal service request.

Included on the service request is a summary of client information, sample identification, required analyses, work instructions, and deliverable requirements. The LIMS is used to track the status of a sample and is important in maintaining internal chain of custody.

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Where possible, instrument data acquired locally is immediately moved to a server (Microsoft Windows2003® domain). This provides a reliable, easily maintained, high-volume acquisition and storage system for electronic data files. With password entry, users may access the system from many available computer stations, improving efficiency and flexibility. The server is also used for data reporting, EDD generation, and administrative functions. Access to these systems is controlled by password. A standardized EDI (electronic data interchange) format is used as a reporting platform, providing functionality and flexibility for end users. With a common standardized communication platform, the EDI provides data reporting in a variety of hardcopy and electronic deliverable formats.

5.4 Backup and Security

Laboratory data is either acquired directly to the centralized acquisition server or acquired locally and then transferred to the server. All data is eventually moved to the centralized data acquisition server for reporting and archiving. Differential backups are performed on all file server information once per day, Sunday through Thursday. Full backups are performed each Friday night. Tapes are physically stored in a locked media cabinet within a locked, temperature controlled computer room, with every other full backup also securely stored offsite.

Access to sample information and data is on a need-to-know basis. Access is restricted to the person's areas of responsibility. Passwords are required on all systems. No direct external, non-ALS Environmental access is allowed to any of our network systems.

The external e-mail system and Internet access is established via a single gateway to discourage unauthorized entry. ALS Environmental uses a closed system for company e-mail. Files, such as electronic deliverables, are sent through the external e-mail system only via a trusted agent or comparable service. The external messaging system operates through a single secure gateway. E-mail attachments sent in and out of the gateway are subject to a virus scan. Because the Internet is not regulated, we use a limited access approach to provide a firewall for added security. Virus screening is performed continuously on all network systems with Internet access.

6) Sample Management

Standard operating procedures have been established for all aspects of sample management within the laboratory including sample receiving, handling, acceptance, log-in, protection, storage, retention, transportation, and disposal. The procedures include provisions necessary to protect the integrity of the sample (as received) and to protect the interests of the laboratory as well as the client. These procedures ensure that samples are handled properly and that all associated documentation is complete and consistent. The sample handling factors that must be taken into account to ensure accurate, defensible analytical results include but are not limited to:

- Amount of sample taken (sampling)
- Type of container used
- Existence and type of sample preservation

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- Holding Time
- Proper custodial documentation
- Sample storage, tracking and/or transfer
- Retention
- Disposal

A record of all procedures to which a sample is subjected while in the possession of the laboratory including acceptance, rejection, login, identification, preservation checks, storage, tracking, and disposal are documented and maintained. In addition, all indirect procedures which support each record of a sample and protects the integrity of a sample is documented and maintained (i.e., refrigerator and freezer temperature checks, thermometer calibrations, etc.).

6.1 Sampling

The quality of analytical results is highly dependent upon the quality of the procedures used to collect, preserve and store samples. ALS Environmental-Simi Valley does not provide sampling services. The laboratory only provides materials needed for sample collection; therefore, ALS Environmental-Simi Valley recommends that clients follow sampling guidelines described in the specific reference methods including 40 CFR 136 and/or USEPA SW-846, NIOSH, OSHA, ASTM, CARB and SCAQMD as appropriate.

When transporting samples to the laboratory, the most expedient but lawful route of transport should be utilized. Also, the hazardous potential of the samples needs to be considered when shipping samples via air freight or passenger airlines.

6.2 Preservation

ALS Environmental-Simi Valley uses sample preservation, container, and holding time recommendations published in a number of referenced documents including, but not limited to USEPA SW 846, USEPA 600/4-79-020, USEPA 600/r-93-100 (inorganic substances), 600/4-91-010, and EPA/625/R-96/010b (air samples) and the US EPA Methods Update Rule effective 4/11/07. The complete citation for each of these and other references can be found in Section 16 of this document. The appropriate container, preservation and holding time information are summarized in Table 6-1. Additional information on this is addressed in each corresponding method SOP.

6.3 Shipping of Containers and Samples

ALS Environmental-Simi Valley provides sample containers to clients via media requests for all matrices (soil, water, air) with the appropriate preservatives (as applicable). These containers include Tedlar bags, Summa canisters, silica-gel tubes, etc. ALS Environmental-Simi Valley keeps client-specific shipping requirements on file and utilizes all major transportation carriers to guarantee that sample shipping requirements (same-day, overnight, etc.) are met. ALS Environmental-Simi Valley also provides its own courier service that makes scheduled courier runs in the greater Los Angeles metropolitan area. The procedures for all requirements directed toward media requests follow the requirements detailed in the *SOP for Media Request Fulfillment* (MED-Media_Req).

6.4 Sample Receiving and Acceptance

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It is the policy of ALS Environmental-Simi Valley to check and record the condition of each sample (i.e. pressure, temperature, etc.) delivered to the Sample Management Office (SMO) and received by the Sample Management Custodian or alternates against certain acceptance criteria as documented in the *SOP for Sample Receiving, Acceptance and Log-In* (SMO-SMPL_REC). This policy is available to all sample management personnel for reference. Any samples, which deviate from these outlined areas, will be clearly flagged with the nature and substance of the deviation. Assessment and condition checks utilized by ALS Environmental-Simi Valley for the acceptance or rejection of samples are based on Table 6-1, applicable Quality Assurance Project Plan (QAPP), permit, program or rule where appropriate. This verification of sample integrity is conducted by the Sample Custodian and may be dependent on the matrix (i.e., temperature, preservation, and headspace) being submitted.

Any abnormalities or departures from specified condition requirements (as described herein) as observed during the initial assessment are recorded. When there is any doubt as to the suitability of a sample for testing, including signs of damage, when a sample does not conform to the description provided, or when the test method required is not specified in sufficient detail the appropriate Project Manager (PM) is notified. The Project Manager is to consult with the client, whenever possible, regarding specific integrity issues documented during sample receipt for further instructions before proceeding and retain a written record of discussion. There may be instances where the client is unavailable, in which case the PM shall document all attempts at contacting the client.

There may be a need to inform the client that a sample(s) is rejected and cannot be accepted for analysis into the laboratory. This situation includes, but is not limited to loss of sample or insufficient amount (subsampling may be performed if it would not cause loss of sample integrity, but the procedure must be indicated with the test results). Subsampling as in the case of air samples is not appropriate.

The procedures for sample documentation, handling acceptance requirements and deviations from the sample acceptance policy are discussed in detail in the *SOP for Sample Receiving, Acceptance and Log-In* (SMO-SMPL_REC). This procedure is also in place to ensure samples are received and properly logged into the laboratory, and that all associated sample documentation, including Chain-of-Custody (COC) records are complete and consistent with the samples received. All associated documentation, including chain of custody forms, memos, transmittal forms, and phone logs, are kept with each project file.

6.5 Sample Log-in

Each sample is logged into the laboratory in such a way as to ensure traceability and cross-reference with regards to the unique laboratory job number, sample identifications and client sample identifications. The laboratory identification is retained throughout the life of the sample in the laboratory. The identification system is designed and operated to ensure that samples cannot be confused physically or in laboratory documentation. Additional information is provided in the *SOP for Sample Receiving, Acceptance and Log-In* (SMO_SMPL_REC).

6.6 Sample Custody

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A sample is in someone's "custody" if:

1. It is in one's actual physical possession;
2. It is in one's view, after being in one's physical possession;
3. It is in one's physical possession and then locked up so that no one can tamper with it;
4. It is kept in a secured area, restricted to authorized personnel only.

Chain-of-Custody (COC) records are used to establish the legal custody of samples, showing the continuous possession of samples from sample collection and transportation to final destination at the laboratory. Custody of each sample is maintained from receipt through disposal (internally utilizing LIMS). When environmental samples are shipped to other laboratories for analysis, the sample management office follows formalized procedures for maintaining the chain of custody, which is written in SOPs for Sample Receiving, Acceptance and Login and Laboratory Storage, Analysis and Tracking.

When samples are removed from the fixed lab and transported to the off-site extraction facility for sample preparation, internal chain of custody procedures still apply. When sample preparation is completed, sample extracts are returned to the laboratory.

Laboratory security and access is important in maintaining the integrity of samples received at ALS Environmental-Simi Valley. Access to the building is limited to the reception area and sample receiving doors, which are manned during business hours and locked at all other times. In addition, the sample storage area within the laboratory is a controlled access area.

The laboratory is equipped with an alarm system which is monitored by a private security firm who provides nighttime and weekend security.

6.7 Sample Storage, Analysis and Tracking

The procedures and requirements for documenting the storage, analysis and tracking as well as maintaining integrity of samples are detailed in the *SOP for Laboratory Storage, Analysis and Tracking* (ADM-LabSAT).

6.8 Sample Retention and Waste Disposal

Upon completion of all analyses, the laboratory samples are retained in accordance with the requirements specified in the method SOPs and the *SOP for Waste Disposal* (DSP-Waste). The samples are disposed according to approved disposal practices or returned to the client (if applicable). All samples are characterized according to hazardous/non-hazardous waste criteria and are segregated accordingly. This evaluation is generally based on results from analyses performed on the sample by ALS Environmental-Simi Valley or an approved subcontract laboratory. It should be noted that all wastes produced at the laboratory, including the laboratory's own various hazardous waste streams, are treated in accordance with all applicable local, State and Federal laws. Complete documentation is maintained for samples from initial receipt through final disposal. This ensures an accurate record of the samples from "cradle to grave."

6.9 Intra-laboratory / Inter-laboratory Transfer of Samples

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When environmental samples are shipped to another laboratory for analysis, samples are properly packed for shipment and preserved if necessary. Sample bottles are wrapped in protective material and placed in a plastic bag (preferably Ziploc®) to avoid any possible cross-contamination of samples during the transportation process. Blue or wet ice is used for temperature preservation, where necessary.

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Table 6-1
Sample Preservation and Holding Times for Performed Methods

Determination (Method)	Matrix	Container	Preservation	MAXIMUM HOLDING TIME
Solid / Water Sample Analysis				
Bromide (EPA 9056)	S,W	P, FP, G	Cool, 4˚C	28 Days
Chloride (EPA 9056)	S,W	P, FP, G	None Required	28 Days
Fluoride (9056)	S,W	P	Cool, 4˚C	28 days
Hydrogen Ion - pH (EPA 9040B/9045C)	S,W	P, FP, G	None Required	Analyze immediately
Nitrate (EPA 9056)	S,W	P, FP, G	Cool, 4˚C	48 hours
Nitrite (EPA 9056)	S,W	P, FP, G	Cool, 4˚C	48 hours
Orthophosphate (EPA 9056)	S	P,G	Cool, 4˚C	48 hours
Chromium VI (EPA 3060A/7196A)	S	P,G	Cool, 4˚C	30 days / 7 days after digestion
Chromium VI (EPA 3060A/7199)	S	P,G	Cool, 4˚C	30 days / 7 days after digestion
Chromium VI (EPA 7196A)	W	P, FP, G	Cool, 4˚C	24 hours
Chromium VI (EPA 7199)	W	P, FP, G	Cool, 4˚C; and/or Ammonium Sulfate buffer to pH = 9.0-9.5	24 hours; 28 days when preserved to pH of 9.0-9.5
Formaldehyde, Acetaldehyde (EPA 8315A Procedure 1 Modified)	S,W	Glass w/Teflon-Lined Lid	Cool, 4˚C	Aqueous – prep. - 72 hours, analysis – 30 days; Soil – prep. minimum, analysis – 30 days
Orthorhombic Cyclooctasulfur (In-House Method)	Solid Wallboard	Ziploc Bag, G	None Required	-
H2S/Sulfur Emission (In-House Method)	Solid Wallboard	Ziploc Bag, G	None Required	-
Copper Corrosion (In-House Method)	Solid Wallboard	Ziploc Bag, G	None Required	-

* W = Water or Aqueous solution; S = Soil or Sediment; P = Polyethylene, G = Glass, FP = fluoropolymer

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Table 6-1 (continued)
Sample Preservation and Holding Times for Performed Methods[a]

Determination (Method)	Matrix	Container	Preservation	MAXIMUM HOLDING TIME	Sample Vol.[d]
Amines (In-House Method)	Air	Treated Alumina Tubes	Sample Receipt-NA; Storage 4˚C±2˚C	30 days	100L
Ammonia (OSHA ID-188/ID-164)	Air	H_2SO_4 Treated Carbon Bead Tubes	Sample Receipt-NA; Storage 4˚C±2˚C	28 days	TWA: 24L STEL: 7.5L
BTU by ASTM D 3588 (SULFUR, ASTM D 5504; C1-C6+, EPA TO-3M; FIXED GASES, 3C)	Gaseous Fuels	Tedlar Bag, Mylar Bag, Summa Canister	N/A	Sulfur (Bag – 24 hours; Canister[c] – 7 days) C1-C6+ (Bag – 72 hours; Canister[b] -30 days[c]) 3C (Bag – 72 hours; Canister[b] -30 days[c])	Bags – 500mL; Canisters – ≥1.0L
C_1-C_6+ (Modified EPA TO-3)	Air	Tedlar Bag, Mylar Bag, Summa Canister	N/A	Bag – 72 hours; Canister[b] – 30 days[c]	Bags – 500mL; Canisters – ≥1.0L
Carboxylic Acids (In-House Method)	Air	Treated Silica Gel Tubes	Sample Receipt-NA; Storage 4˚C±2˚C	30 days until extraction; 14 days for analysis	100L
Chromium VI (Modification of EPA 7196/7199 or CARB SOP MLD039)	Air	HCO_3 Treated Filters	Sample Receipt, 4˚C±2˚C Storage Freezer	21 days until extraction; 24 hours for analysis	210L
Total Gaseous Non-methane Organics (TGNMO) (EPA 25C)	Air	Tedlar Bag, Mylar Bag, Summa Canister	N/A	Bag – 72 hours; Canister[b] – 30 days[c]	Bags - 500mL; Canisters – ≥1.0L
Fixed Gases (EPA 3C & ASTM D 1946)	Air	Tedlar Bag, Mylar Bag, Summa Canister	N/A	Bag – 72 hours; Canister[b] – 30 days[c]	Bags – 500mL; Canisters – ≥1.0L
Helium & Hydrogen (EPA 3C Modified)	Air	Summa Canister	N/A	Canister[b] – 30 days[c]	Bags – 500mL; Canisters – ≥1.0L
Argon (EPA 3C Modified)	Air	Tedlar Bag, Mylar Bag, Summa Canister	N/A	Bag – 72 hours[c]; Canister[b] – 30 days[c]	Bags – 500mL; Canisters – ≥1.0L
Air-Phase Petroleum Hydrocarbons (MADEP APH)	Air	Summa Canister	N/A	28 days	6.0L

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Table 6-1 (continued)
Sample Preservation and Holding Times for Performed Methods[a]

Determination (Method)	Matrix	Container	Preservation	Holding Time	Sample Vol.[d]
Methanol, Acetaldehyde, Methyl Ethyl Ketone, Propionaldehyde (NCASI – DI/MeOH 94.03 / NCASI – DI/HAPS 99.01)	Aqueous -Effluent	Glass w/Teflon Lined Lid	No Headspace; 4˚C±2˚C; HCl to pH 2-3 (Effluent only)	30 days	(1) 40mL Vial
Organic Vapors / NAPHTHAS (Diesel; etc.) (NIOSH 1550 / OSHA 7)	Air	Charcoal Tube; 3M 3500 or 3520 Badge; Silica Gel Tube w/ plastic caps	Sample Receipt-NA; Storage 4˚C±2˚C	14 days	Various
Methane, Ethane, Ethene, Propane, Propene (RSK 175)	Aqueous	Glass w/Teflon- Lined Lid	No Headspace; HCl to pH<2; 4˚C±2˚C	14 days when preserved	(3) 40mL Vials
Carbon Dioxide (RSK 175)	Aqueous	Glass w/Teflon Lined Lid	No Headspace; neutral pH (5-8); 4˚C±2˚C	N/A[e]	(3) 40mL Vials
Sulfur Compounds (In-House Method)	Aqueous	Glass w/Teflon- Lined Lid	No Headspace; pH>4; 4˚C±2˚C	Following pH adjustment – 24 hours	(2) 40mL Vials
Sulfur Compounds (ASTM D 5504; SCAQMD 307-91; Modified SCAQMD 307-91)	Air	Tedlar Bag, Fused Silica Lined Stainless Steel Canister	No direct sunlight	Bag – 24 hours; Canister[c] - 7 days	Bags – 500mL; Canisters – ≥1.0L
Methanol, Ethanol, Isopropyl alcohol, Freon, and Methylene Chloride (EPA TO-3 Modified)	Air	Tedlar Bag, Mylar Bag, Summa Canister	N/A	Bag – 72 hours; Canister[b] – 30 days[c]	Bags – 500mL; Canisters – ≥1.0L
Total Petroleum Hydrocarbons (TPHG) (EPA TO-3 Modified)	Air	Tedlar Bag, Mylar Bag, Summa Canister	N/A	Bag – 72 hours; Canister[b] – 30 days[c]	Bags – 500mL; Canisters – ≥1.0L
Pesticides and Polychlorinated Biphenyls (PCBs) (EPA TO-4A & TO-10A)	Air	Glass PUF Cartridge; TO-4A (High Volume); TO-10A (Low Volume)	Sample Receipt, <4˚C; Store sample and extract at <4˚C	7 days until extraction; extract – 40 days	2 m^3
Formaldehyde & Other Carbonyl Compounds (EPA TO-11A)	Air	DNPH-Coated Silica Gel Cartridge w/ Polypropylene Cap; SKC UME[x] and Bacharach GMD 570 Passive Monitors (formaldehyde only)	Sample Receipt, 4˚C±2˚C; Laboratory Preservation, 4˚C±2	14 days until extraction; 30 days for analysis	100 – 150L
Polycyclic Aromatic Hydrocarbons (PAHs) (EPA TO-13A)	Air	Polyurethane Foam (PUF) plugs, XAD Tube, PUF / XAD-2	Sample Receipt, <4˚C; Laboratory Preservation, <4˚C	7 days until extraction; 40 days after	130 – 400 m^3

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Table 6-1 (continued)
Sample Preservation and Holding Times for Performed Methods[a]

Determination (Method)	Matrix	Container	Preservation	Holding Time	Sample Vol.[d]
Volatile Organic Compounds (EPA TO-14A & TO-15)	Air	Tedlar Bag, Summa Canister (1L, 6L)	N/A	Bag – 72 hours; Canister – 30 days	Bags - 500mL; Canisters – 1.0L / 6.0L
Volatile Organic Compounds (EPA TO-17)	Air	Sorbent Tubes w/Swagelock Caps & PTFE Ferrules	<4˚C; organic solvent free environment; Laboratory Storage, 4˚C±2˚C	30 days	1-4L

Footnotes :

a.	See Section 16.0 for reference information.
b.	Some methods do not specify the utilization of canisters; therefore, there is no required hold time and this will be noted in the case narrative.
c.	Laboratory recommended hold time; therefore, samples analyzed outside this hold time will be noted in the case narrative accordingly.
d.	Sample volumes are the minimum, which should be received by the laboratory; however, canister volumes should match the canister size utilized.
e.	There is no holding time requirement available for CO_2 and laboratory studies are not available indicating the validity of data prior to or following a specified length of time. Therefore, no holding time notation or qualifier will be adhered to results for this compound.

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Figure 6-1
Air Chain of Custody Form

Air - Chain of Custody Record & Analytical Service Request

Page _____ of _____

2655 Park Center Drive, Suite A
Simi Valley, California 93065
Phone (805) 526-7161
Fax (805) 526-7270

Requested Turnaround Time in Business Days (Surcharges) please circle
1 Day (100%) 2 Day (75%) 3 Day (50%) 4 Day (35%) 5 Day (25%) 10 Day-Standard

ALS Project No.

ALS Contact:

Company Name & Address (Reporting Information)

Project Name

Project Number

Project Manager

P.O. # / Billing Information

Phone

Fax

Email Address for Result Reporting

Sampler (Print & Sign)

Analysis Method

Comments
e.g. Actual Preservative or specific instructions

Client Sample ID	Laboratory ID Number	Date Collected	Time Collected	Canister ID (Bar code # - AC, SC, etc.)	Flow Controller ID (Bar code #- FC #)	Canister Start Pressure "Hg	Canister End Pressure "Hg/psig	Sample Volume			

Report Tier Levels - please select
Tier I - Results (Default if not specified) _____ Tier III (Results + QC & Calibration Summaries) _____
Tier II (Results + QC Summaries) _____ Tier IV (Data Validation Package) 10% Surcharge _____

EDD required Yes / No
Type: _____ Units:_____

Chain of Custody Seal: (Circle)
INTACT BROKEN ABSENT

Project Requirements (MRLs, QAPP)

Relinquished by: (Signature)	Date:	Time:	Received by: (Signature)	Date:	Time:
Relinquished by: (Signature)	Date:	Time:	Received by: (Signature)	Date:	Time:

Cooler / Blank Temperature ____°C

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Figure 6-2
Soil / Water Chain of Custody Form

Soil / Water - Chain of Custody Record & Analytical Service Request

Page _____ of _____

2655 Park Center Drive, Suite A
Simi Valley, California 93065
Phone (805) 526-7161
Fax (805) 526-7270

Requested Turnaround Time in Business Days (Surcharges) please circle
1 Day (100%) 2 Day (75%) 3 Day (50%) 4 Day (35%) 5 Day (25%) 10 Day-Standard

ALS Project No.

ALS Contact:

Company Name & Address (Reporting Information)

Project Name

Project Number

P.O. # / Credit Card / Billing Information

Project Manager

Phone

Fax

Email Address for Result Reporting

Analysis

Comments

Client Sample ID	Laboratory ID Number	Date Collected	Time Collected	Water	Soil	Solid	Other				

Report Tier Levels - please select
Tier I - Results (Default if not specified) _____
Tier II (Results + QC Summaries) _____
Tier III (Results + QC & Calibration Summaries) _____
Tier IV (Data Validation Package) 10% Surcharge _____

EDD required Yes / No
Type: _____ Units: _____

Chain of Custody Seal: (Circle)
INTACT BROKEN ABSENT

Project Requirements (MRLs)

Reliquished by: (Signature)	Date:	Time:	Received by: (Signature)	Date:	Time:
Reliquished by: (Signature)	Date:	Time:	Received by: (Signature)	Date:	Time:
Reliquished by: (Signature)	Date:	Time:	Received by: (Signature)	Date:	Time:

Cooler / Blank / Ice / No Ice
Temperature _____°C

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Figure 6-3

Sample Acceptance Check Form

Client: _____ Work order: _____

Project: _____

Sample(s) received on: _____ Date opened: _____ by: _____

Note: This form is used forall samples received by CAS. The use of this form for custody seals is strictly meant to indicate presence/absence and not as an indication of compliance or nonconformity. Thermal preservation and pH will only be evaluated either at the request of the client and/or as required by the method/SOP.

		Yes	No	N/A
1	Were **sample containers** properly marked with client sample ID?	☐	☐	☐
2	Container(s) **supplied by CAS**?	☐	☐	☐
3	Did **sample containers** arrive in good condition?	☐	☐	☐
4	Were **chain-of-custody** papers used and filled out?	☐	☐	☐
5	Did **sample container labels** and/or tags agree with custody papers?	☐	☐	☐
6	Was **sample volume** received adequate for analysis?	☐	☐	☐
7	Are samples within specified holding times?	☐	☐	☐
8	Was proper **temperature** (thermal preservation) of cooler at receipt adhered to?	☐	☐	☐
9	Was a **trip blank** received?	☐	☐	☐
10	Were **custody seals** on outside of cooler/Box?	☐	☐	☐
	Location of seal(s)? _____ Sealing Lid?	☐	☐	☐
	Were signature and date included?	☐	☐	☐
	Were seals intact?	☐	☐	☐
	Were custody seals on outside of sample container?	☐	☐	☐
	Location of seal(s)? _____ Sealing Lid?	☐	☐	☐
	Were signature and date included?	☐	☐	☐
	Were seals intact?	☐	☐	☐
11	Do containers have appropriate **preservation**, according to method/SOP or Client specified information?	☐	☐	☐
	Is there a client indication that the submitted samples are **pH** preserved?	☐	☐	☐
	Were **VOA vials** checked for presence/absence of air bubbles?	☐	☐	☐
	Does the client/method/SOP require that the analyst check the sample pH and if necessary alter it?	☐	☐	☐
12	**Tubes:** Are the tubes capped and intact?	☐	☐	☐
	Do they contain moisture?	☐	☐	☐
13	**Badges:** Are the badges properly capped and intact?	☐	☐	☐
	Are dual bed badges separated and individually capped and intact?	☐	☐	☐

Lab Sample ID	Container Description	Required pH *	Received pH	Adjusted pH	VOA Headspace (Presence/Absence)	Receipt / Preservation Comments

Explain any discrepancies: (include lab sample ID numbers): _____

RSK - MEEPP, HCL (pH<2); RSK - CO2, (pH 5-8); Sulfur (pH>4)

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7) Analytical Procedures

ALS Environmental employs methods and analytical procedures from a variety of external sources. Reference documents include but are not limited to: ASTM, CARB, NCASI, NIOSH, OSHA, SCAQMD, USEPA SW-846, USEPA 600/4-79-020, 600/4-91-010, 600/R-93/100 (inorganic substances), 600/625/R-96/010b (air samples), EPA 40 CFR part 136, and associated Supplements; US EPA Methods Update Rule effective 4/11/07 and *Standard Methods for the Examination of Water and Wastewater* for water and wastewater samples. Complete citations for these references can be found in Section 16.0. Other published procedures, such as state-specific methods, program-specific methods, or in-house methods may be used. Several factors are involved with the selection of analytical methods to be used in the laboratory. These include the method detection limit, the concentration of the analyte being measured, method selectivity, accuracy and precision of the method, the type of sample being analyzed, and the regulatory compliance objectives. The implementation of methods by ALS Environmental is described in SOPs specific to each method. A list of NELAP-accredited methods is given in Appendix G. Further details are described below.

7.1 Standard Operating Procedures (SOPs) and Laboratory Notebooks

ALS Environmental maintains SOPs for use in both technical and administrative functions. SOPs are written following standardized format and content requirements as described in the *SOP for Preparation of Standard Operating Procedures* (ADM-SOP). Each SOP is reviewed and approved by a minimum of two managers (the Laboratory Director and/or Department Manager and the QA Manager). All SOPs undergo a documented annual review to make sure current practices are described. The QA Manager maintains a comprehensive list of current SOPs. The document control process ensures that only the most currently prepared version of an SOP is being used. The QA Manual, QAPPs, SOPs, standards preparation logbooks, maintenance logbooks, et al., are controlled documents, unless otherwise noted. The procedures for document control are described in the *SOP for Document Control* (CE-GEN005). In addition to SOPs, each laboratory department maintains a current file, accessible to all laboratory staff, of the current methodology used to perform analyses. Laboratory notebook entries are standardized following the guidelines in the *SOP for Making Entries Onto Analytical Records* (CE-QA007). Entries made into laboratory notebooks are reviewed and approved by the appropriate supervisor at a regular interval.

7.2 Deviation from Standard Operating Procedures

When a customer requests a modification to an SOP (such as a change in reporting limit, addition or deletion of target analyte(s), etc.), the Project Manager handling that project must discuss the proposed deviation with the department manager in charge of the analysis and obtain their approval to accept the project. The Project Manager is responsible for documenting the approved or allowed deviation from the SOP by placing a detailed description of the deviation attached to the quotation or in the project file and also providing an appropriate comment on the service request when the samples are received.

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For circumstances when a deviation or departure from company policies or procedures involving any non-technical function is found necessary, approval must be obtained from the appropriate supervisor, manager, the laboratory director, or other level of authority. Frequent departure from policy is not encouraged. However, if frequent departure from any policy is noted, the laboratory director will address the possible need for a change in policy.

7.3 Modified Procedures

ALS Environmental strives to perform published methods as described in the referenced documents. If there is a material deviation from the published method, the method is cited as a "Modified" method in the analytical report. Modifications to the published methods are listed in the standard operating procedure. Standard operating procedures are available to analysts and are also available to our clients for review, especially those for "Modified" methods. Client approval is obtained for the use of "Modified" methods prior to the performance of the analysis.

7.4 Analytical Batch

The basic unit for analytical quality control is the analytical batch. The definition that ALS Environmental-Simi Valley has adopted for the analytical batch is listed below. The overriding principle for describing an analytical batch is that all the samples in a batch, both field samples and quality control samples are to be handled exactly the same way, and all of the data from each analysis is to be manipulated in exactly the same manner. The minimum requirements of an analytical batch are:

1) The number of (field) samples in a batch is not to exceed 20.
2) All (field) samples in a batch are of the same matrix.
3) The QC samples to be processed with the (field) samples include:
   a) Method Blank (a.k.a. Laboratory Reagent Blank)

      Function: Determination of laboratory contamination

   b) Laboratory Control Sample

      Function: Assessment of method performance

   c) Matrix Spiked (field) Sample (a.k.a. Laboratory Fortified Sample Matrix)*

      Function: Assessment of matrix bias

   d) Duplicate Matrix Spiked (field) Sample or Duplicate (field) Sample (a.k.a. Laboratory Duplicate)*

      Function: Assessment of batch precision

   * A sample identified as a field blank, an equipment blank, or a trip blank is not to be matrix spiked or duplicated.

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4) A single lot of reagents is used to process the batch of samples.

5) Each operation within the analysis is performed by a single analyst, technician, chemist, or by a team of analysts/technicians/chemists.

6) Samples are analyzed in a continuous manner over a timeframe not to exceed 24-hours between the start of processing of the first and last sample of the batch.

7) (Field) samples are assigned to batches commencing at the time that sample processing begins. For example: for analysis of metals, sample processing begins when the samples are digested. For analysis of organic constituents, it begins when the samples are extracted.

8) The QC samples are to be analyzed in conjunction with the associated field samples prepared with them. However, for tests which have a separate sample preparation step that defines a batch (digestion, extraction, etc.), the QC samples in the batch do not require analysis each time a field sample within the preparation batch is analyzed (multiple instrument sequences to analyze all field samples in the batch need not include re-analyses of the QC samples).

9) The batch is to be assigned a unique identification number that can be used to correlate the QC samples with the field samples.

10) Batch QC refers to the QC samples that are analyzed in a batch of (field) samples.

11) Project-specific requirements may be exceptions. If project, program, or method requirements are more stringent than these laboratory minimum requirements, then the project, program, or method requirements will take precedence. However, if the project, program, or method requirements are less stringent than these laboratory minimum requirements, these laboratory minimum requirements will take precedence.

Note: Matrix spiked samples are often <u>not feasible</u> for air matrices. Therefore, the MS shall be used as required by the test method and as specified by the corresponding method SOP.

7.5 Specialized Procedures

ALS Environmental not only strives to provide results that are scientifically sound, legally defensible, and of known and documented quality; but also strives to provide the best solution to analytical challenges. Procedures using specialized instrumentation and methodology have been developed to improve sensitivity (provide lower detection limits), selectivity (minimize interferences while maintaining sensitivity), and overall data quality for low concentration applications. Examples are specialized GC/MS analyses, and low level organics analyses (including PAHs, pesticides and PCBs).

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8) Calibration Procedures

All equipment and instruments used at ALS Environmental are operated, maintained and calibrated according to the manufacturer's guidelines and recommendations, as well as to criteria set forth in the applicable analytical methodology. Operation and calibration are performed by personnel who have been properly trained in these procedures. Documentation of calibration information is maintained in appropriate reference files. Brief descriptions of the calibration procedures for our major laboratory equipment and instruments are described below. Calibration verification is performed according to the applicable analytical methodology. Calibration verification procedures and criteria are listed in laboratory Standard Operating Procedures. Documentation of calibration verification is maintained in appropriate reference files. Records are maintained to provide traceability of reference materials.

Traceability is defined as the property of a measurement result or value of a standard which can be related to stated references through an unbroken chain, each with stated uncertainties and is documented for all material used to perform calibrations. The documentation, a certificate of analysis containing, at a minimum, the manufacturer, address, accreditation number (where applicable), how traceability was achieved, the traceable values, their associated uncertainty, and the unique serial or laboratory identification number of the equipment or standard reference material (SRM) shall serve as initial point in the chain of traceability. The unique serial number or laboratory identification number is used throughout the laboratory to trace equipment and materials back to the original certificate of analysis.

Laboratory support equipment (thermometers, balances, and weights) are verified on an annual basis by a vendor accredited to ISO/IEC 17025:2005 International Standards. All analytical measurements generated at ALS Environmental are performed using materials and/or processes that are traceable to a reference material. Metrology equipment (analytical balances, thermometers, etc.) is calibrated using reference materials traceable to the National Institute of Standards and Technology (NIST). These primary reference materials are themselves recertified on an annual basis. Vendors used for metrology support are required to verify compliance to International Standards by supplying the laboratory with a copy of their scope of accreditation.

Equipment subjected to overloading or mishandling, or has been shown by verification to be defective, is taken out of service and labeled until repaired. That piece of equipment is placed back in service only after verifying, by calibration, that it performs satisfactorily.

8.1 Temperature Control and Measuring Devices

Temperatures are monitored and recorded for all critical measurement temperature-regulating devices including freezers, refrigerators and ovens. Each piece of equipment is labeled with a unique identifier, the required temperature or range of use according to the needs of the analysis or application. Temperature record books are kept which contain equipment identifier, daily-recorded temperatures (if in use, business days), acceptance criteria and the initials of the laboratory staff member who performed the checks for all temperature-regulating devices in daily use.

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A number of thermometers include a temperature range per certain project requirements (complies with Department of Defense Quality Systems Manual for Environmental Laboratories); this range is recorded to document consistent compliance with required temperatures for refrigerators and freezers, where applicable.

All thermometers are identified by a unique identifying number (i.e., serial number), and the calibration of these thermometers is checked annually against a National Institute of Standards and Technology (NIST) certified thermometer. All corresponding correction factors are noted on the device as well as in the thermometer calibration logbook. The NIST calibrated thermometer is recertified by an approved vendor accredited ISO/IEC 17025:2005 International Standard on an annual basis and certificates are retained on file for review. All temperature monitoring is conducted in accordance with the *SOP for Sample Receipt, Acceptance and Log-In* (SMO-SMPL_REC) and thermometer calibration requirements are performed in accordance with the *SOP for Calibration and Use of the Laboratory Support Equipment* (ADM-SupEQ).

8.2 Volumetric Dispensing Devices

The accuracy of pipettes used to make critical-volume measurements is verified on a quarterly basis. Typically, the indicated volume or range (where applicable) of the pipette is checked and both the accuracy and precision verification are performed using the above-mentioned procedure. The calibrations are evaluated against the intended use (volume or range) of the pipette and if the calibration is not approved for the specified volume(s) it is tagged accordingly (i.e. "Do Not Use Below 5uL"). The results for all calibration verifications are recorded and maintained.

Note: Glass microliter syringes including gas-tight syringes are considered in the same manner as Class A glassware and are not held to the calibration/verification requirements as are other volumetric dispensing devices.

8.3 Analytical Balances and Weights

Analytical balances and weights are calibrated / recertified and certificates issued annually by an approved vendor accredited to ISO/IEC 17025:2005 International Standard. The calibration of each balance is checked once each day of use in the expected range, utilizing the calibrated weights. Bound record books are kept which contain the identification of balance (serial number), recorded measurements and the initials of the analyst who performed the check. All certificates for the balances and weights are available for review.

8.4 Pressure/Vacuum Gauges

ALS Environmental-Simi Valley digital pressure/vacuum gauges are used in a number of critical measurements within the laboratory. The following is a list of the uses for this gauge type.

- Canister cleaning and conditioning
- Measure the vacuum on canisters before they are sent to the client for sampling.
- Measure the initial/final vacuum/pressure of canisters prior to analysis.
- Measure pressure during the preparation of selected standards.

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Digital pressure/vacuum gauges are calibrated and certificates issued once per year by an approved metrology organization. All calibrations are performed against standards traceable to the National Institute of Standards and Technology (NIST) or other recognized national metrology institutes. In addition, ALS Environmental-Simi Valley performs a calibration check for each gauge six months following the calibration date. The laboratory retains all corresponding calibration and verification documentation for review.

8.5 Water Purification Systems

Purified water is utilized for a number of laboratory functions including instrument and method blanks, trip blanks, washes and sample dilutions. The water purification system utilizes three mixed-ion beds, four filters, and resistively lights with constant water recirculation. It is designed to produce deionized water of ASTM Type II quality, with 16-18 megohm-cm resistance at 25˚C and is checked and recorded daily (prior to and if in use). Maintenance and repair on the system is conducted by an approved service supplier and all records including purification checks/verifications are maintained on file for review. For procedures on additional purification (i.e., boiling and/or purging) and purification checks/verifications, refer to the applicable method standard operating procedures.

8.6 Source and Preparation of Standards and Reference Materials

Consumable reference materials routinely purchased by the laboratories (e.g., analytical standards) are purchased from nationally recognized, reputable vendors. All vendors have fulfilled the requirements for ISO 9001 certification and/or are accredited by A_2LA. ALS Environmental-Simi Valley relies on a primary vendor for the majority of its analytical supplies. Consumable primary stock standards are obtained from certified commercial sources or from sources referenced in a specific method. Supelco, Ultra Scientific, AccuStandard, Chem Services, Inc., Aldrich Chemical Co., Baker, Spex, etc. are examples of the vendors used. Reference material information is recorded in the appropriate logbook(s) and materials are stored under conditions that provide maximum protection against deterioration and contamination. The logbook entry includes such information as an assigned logbook identification code, the source of the material (i.e. vendor identification), solvent (if applicable) and concentration of analyte(s), reference to the certificate of analysis and an assigned expiration date. The date that the standard is received in the laboratory is marked on the container. When the reference material is used for the first time, the date of usage and the initials of the analyst are also recorded on the container.

Stock solutions and calibration standard solutions are prepared fresh as often as necessary according to their stability. All standard solutions are properly labeled as to analyte concentration, solvent, date, preparer, and expiration date; these entries are also recorded in the appropriate notebook(s) following the *SOP for Making Entries Onto Analytical Records* (CE-QA007). Prior to sample analysis, all calibration reference materials are verified with a second, independent source of the material (see section 9.3.5).

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8.7 Instrument Calibration

The laboratory specifies the procedures and documentation for initial instrument calibration and continuing calibration verification in the applicable method standard operating procedures to ensure that data is of known quality and is appropriate for a specific regulation and/or client requirement. The procedural steps for calibration including, frequency, number of points, integration, calculations, acceptance criteria (appropriate to the calibration technique employed), corrective action, associated statistics, and data qualifications are included in applicable methods, method standard operating procedures and/or client project plans. The essential elements that define the procedures and required documentation for initial instrument calibrations are specified below.

- Sufficient raw data records are retained to permit reconstruction of all calibrations.
- If a reference or mandated method does not specify the number of calibration standards, the initial calibration range shall consist of a minimum of 5 contiguous calibration points for organics and a minimum of 3 contiguous calibration points for inorganics. The actual numbers of points utilized is specified in the corresponding method SOP.
- The concentrations should bracket the expected concentration range of samples.
- Initial instrument calibration procedures referenced in test methods (either directly or indirectly) are readily available to the analysts.
- All sample results are quantitated from the initial instrument calibration and are not quantitated from any continuing instrument calibration verification unless otherwise specified by regulation, method or program.
- The initial instrument calibration is verified with a standard obtained from a second manufacturer or lot and traceability to a national standard is maintained, where available.
- The acceptance criteria utilized is appropriate for the calibration technique employed.
- The lowest calibration standard in the initial calibration is at or below the lowest concentration for which quantitative data are to be reported and is referred to at this laboratory as the method reporting limit (MRL). Some programs and/or agencies refer to this limit as the practical quantitation limit (PQL) or Limit of Quantitation (LOQ).
- Any data reported below the MRL or above the highest calibration standard is considered to have an increased quantitative uncertainty and is appropriately qualified in the report.
- The lowest calibration standard is above the limit of detection or method detection limit (MDL).

8.8 Internal and External Calibrations

Internal standard calibration involves the comparison of instrument responses from the target compounds in the sample to the responses of specific standards added to the sample or sample extract prior to injection. The ratio of the peak area of the target compound in the sample or sample extract to the peak area of the internal standard in the sample or sample extract is compared to a similar

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ratio derived for each calibration standard. The ratio is termed the response factor (RF) or relative response factor (RRF) in some methods.

External standard calibration involves comparison of instrument responses from the sample to the responses from the target compounds in the calibration standards. Sample peak areas are compared to peak areas of the standards. The ratio of the detector responses to the amount (mass) of analyte in the calibration standard is defined as the calibration factor or in some cases it may be referred to as response factor.

8.9 Continuing Calibration Verification

The essential elements that define the procedures and required documentation for continuing instrument calibration verification are specified below.

- When an initial calibration is not performed on the day of analysis, continuing instrument calibration verification is analyzed with each batch.
- Calibration is verified for each reported compound, element or parameter; however, for multi-component analytes such as aroclors or total petroleum hydrocarbons a representative chemical related substance or mixture may be used. The allowance for this exception is dependent on applicable regulatory, method, or client project plans.
- Generally, the instrument calibration verification is performed at the beginning, end and every ten samples of each analytical batch (except, if an internal standard is used, only one verification needs to be performed at the beginning of the analytical batch); whenever it is suspected that the analytical system may be out of calibration; if the time period for calibration or most previous calibration verification has expired; or for analytical systems that contain a specific calibration verification requirement. Specific requirements for the frequency of continuing calibration verification, for a particular method, is specified in the corresponding method standard operating procedure.

9) Quality Control

A primary focus of ALS Environmental's QA Program is to ensure the accuracy, precision and comparability of all analytical results. Prior to using a procedure for the analysis on field samples, acceptable method performance is established by performing demonstration of capability analyses. Performance characteristics are established by performing method detection limit studies and assessing accuracy and precision according to the reference method. ALS Environmental has established Quality Control (QC) objectives for precision and accuracy that are used to determine the acceptability of the data that is generated. These QC limits are either specified in the test methodology or are statistically derived based on the laboratory's historical data. Quality Control objectives are defined below.

9.1 Quality Control Objectives

9.1.1 Demonstration of Capability - A demonstration of capability (DOC) is made prior to using any new test method or when a technician is new to the method. This demonstration is made following regulatory, accreditation, or method specified procedures. In general, this demonstration does not test the performance of the method in real world

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samples, but in the applicable clean matrix free of target analytes and interferences.

A quality control sample material may be obtained from an outside source or may be prepared in the laboratory. The analyte(s) is (are) diluted in a volume of clean matrix (for analytes which do not lend themselves to spiking, e.g., TSS, the demonstration of capability may be performed using quality control samples). Where specified, the method-required concentration levels are used. Four aliquots are prepared and analyzed according to the test procedure. The mean recovery and standard deviations are calculated and compared to the corresponding acceptance criteria for precision and accuracy in the test method or laboratory-generated acceptance criteria (if there are not established mandatory criteria). All parameters must meet the acceptance criteria. Where spike levels are not specified, actual Laboratory Control Sample results may be used to meet this requirement, provided acceptance criteria are met.

9.1.2 Accuracy - Accuracy is a measure of the closeness of an individual measurement (or an average of multiple measurements) to the true or expected value. Accuracy is determined by calculating the mean value of results from ongoing analyses of laboratory-fortified blanks, standard reference materials, and standard solutions. In addition, laboratory-fortified (i.e. matrix-spiked) samples are also measured; this indicates the accuracy or bias in the actual sample matrix. Accuracy is expressed as percent recovery (% REC.) of the measured value, relative to the true or expected value. If a measurement process produces results whose mean is not the true or expected value, the process is said to be biased. Bias is the systematic error either inherent in a method of analysis (e.g., extraction efficiencies) or caused by an artifact of the measurement system (e.g., contamination).

ALS Environmental utilizes several quality control measures to eliminate analytical bias, including systematic analysis of method blanks, laboratory control samples and independent calibration verification standards. Because bias can be positive or negative, and because several types of bias can occur simultaneously, only the net, or total, bias can be evaluated in a measurement.

9.1.3 Precision - Precision is the ability of an analytical method or instrument to reproduce its own measurement. It is a measure of the variability, or random error, in sampling, sample handling and in laboratory analysis. The American Society of Testing and Materials (ASTM) recognizes two levels of precision: repeatability - the random error associated with measurements made by a single test operator on identical aliquots of test material in a given laboratory, with the same apparatus, under constant operating conditions, and reproducibility - the random error associated with measurements made by different test operators, in different laboratories, using the same method but different equipment to analyze identical samples of test material.

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"Within-batch" precision is measured using replicate sample or QC analyses and is expressed as the relative percent difference (RPD) between the measurements. The "batch-to-batch" precision is determined from the variance observed in the analysis of standard solutions or laboratory control samples from multiple analytical batches.

9.1.4 Control Limits - The control limits for accuracy and precision originate from two different sources. For analyses having enough QC data, control limits are calculated at the 99% confidence limits. For analyses not having enough QC data, or where the method is prescriptive, control limits are taken from the method on which the procedure is based. If the method does not have stated control limits, then control limits are assigned method-default or reasonable values. Control limits are updated periodically when new statistical limits are generated for the appropriate surrogate, laboratory control sample, and matrix spike compounds (typically once a year) or when method prescribed limits change. The updated limits are reviewed by the QA Manager. The new control limits replace the previous limits and data is assessed using the new values. Current acceptance limits for accuracy and precision are available from the laboratory. For inorganics, the precision limit values listed are for laboratory duplicates. For organics, the precision limit values listed are for duplicate laboratory control samples or duplicate matrix spike analyses.

9.1.5 Representativeness - Representativeness is the degree to which the field sample, being properly preserved, free of contamination, and analyzed within holding time, represents the overall sample site or material. This can be extended to the sample itself, in that representativeness is the degree to which the subsample that is analyzed represents the entire field sample submitted for analysis. ALS Environmental has sample handling procedures to ensure that the sample used for analysis is representative of the entire sample. Further, analytical SOPs specify appropriate sample handling and sample sizes to further ensure the sample aliquot that is analyzed is representative in entire sample. Air samples received by the laboratory in canisters and bags are considered to be homogenous and therefore, no special sample preparation procedures are necessary.

9.1.6 Comparability – Comparability expresses the confidence with which one data set can be compared to another and is directly affected by data quality (accuracy and precision) and sample handling (sampling, preservation, etc). Only data of known quality can be compared. The objective is to generate data of known quality with the highest level of comparability, completeness, and usability. This is achieved by employing the quality controls listed below and standard operating procedures for the handling and analysis of all samples. Data is reported in units specified by the client and using ALS Environmental or project-specified data qualifiers.

9.2 Method Detection Limits and Method Reporting Limits & Limits of Detection / Quantitation

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Method Detection Limits (MDL) for methods performed at ALS Environmental-Simi Valley are determined during initial method set up and if any significant changes are made. If an MDL study is not performed annually, the established MDL is verified by performing a limit of detection (LOD) verification on every instrument used in the analysis. The MDLs are determined by following the *SOP for Performing Method Detection Limits Studies and Establishing Limits of Detection and Quantitation* (ADM-MDL), which is based on the procedure in 40 CFR Part 136, Appendix B. As required by NELAP and DoD protocols, the validity of MDLs is verified using LOD verification samples.

The Method Reporting Limit (MRL) is the lowest amount of an analyte in a sample that can be quantitatively determined with stated, acceptable precision and accuracy under stated analytical conditions (i.e. limit of quantitation- LOQ). LOQ are analyzed on an annual basis and cannot be lower than the lowest calibration standard. Current MDLs and MRLs are available from the laboratory.

9.3 Quality Control Procedures

The specific types, frequencies, and processes for quality control sample analysis are described in detail in method-specific standard operating procedures and listed below. These sample types and frequencies have been adopted for each method and a definition of each type of QC sample is provided below.

9.3.1 Method Blank (a.k.a. Laboratory Reagent Blank)

The method blank is an analyte-free matrix (air, water, soil, etc.) subjected to the entire analytical process. When analyte-free soil is not available, anhydrous sodium sulfate, organic-free sand, or an acceptable substitute is used. The method blank is analyzed to demonstrate that the analytical system itself does not introduce contamination. The method blank results should be below the Method Reporting Limit (MRL) or, if required for DoD projects, < ½ MRL for the analyte(s) being tested. Otherwise, corrective action must be taken. A method blank is included with the analysis of every sample preparation batch, every 20 samples, or as stated in the method, whichever is more frequent.

9.3.2 Calibration Blanks

For some methods, calibration blanks are prepared along with calibration standards in order to create a calibration curve. Calibration blanks are free of the analyte of interest and, where applicable, provide the zero point of the calibration curve. Additional project-specific requirements may also apply to calibration blanks.

9.3.3 Continuing Calibration Blanks

Continuing calibration blanks (CCBs) are solutions of analyte-free water, reagent, or solvent that are analyzed in order to verify the system is contamination-free when CCV standards are analyzed. The frequency of CCB analysis is once every ten samples or as indicated in the method, whichever is greater. Additional project-specific requirements may also apply to continuing calibration blanks.

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9.3.4 Calibration Standards

Calibration standards are vapors, liquids or solutions of known concentration prepared from primary standard or stock standard materials. Calibration standards are used to calibrate the instrument response with respect to analyte concentration. Standards are analyzed in accordance with the requirements stated in the particular method being used.

9.3.5 Initial (or Independent) Calibration Verification Standards

Initial (or independent) calibration verification standards (ICVs) are standards that are analyzed *after* calibration but *prior to* sample analysis, in order to verify the validity and accuracy of the standards used for calibration. Once it is determined that there is no defect or error in the calibration standard(s), standards are considered valid and may be used for subsequent calibrations and quantitative determinations (as expiration dates and methods allow). The ICV standards are prepared from materials obtained from a source independent of that used for preparing the calibration standards ("second-source"). ICVs are also analyzed in accordance with method-specific requirements.

9.3.6 Continuing Calibration Verification Standards

Continuing calibration verification standards (CCVs) are midrange standards that are analyzed in order to verify that the calibration of the analytical system is still acceptable. The frequency of CCV analysis is either once every ten samples, or as indicated in the method.

9.3.7 Internal Standards

Internal standards are known amounts of specific compounds that are added to each sample prior to instrument analysis. Internal standards are generally used for GC/MS procedures to correct sample results that have been affected by changes in instrument conditions or changes caused by matrix effects. The requirements for evaluation of internal standards are specified in each method and SOP.

9.3.8 Surrogates

Surrogates are organic compounds which are similar in chemical composition and chromatographic behavior to the analytes of interest, but which are not normally found in environmental samples. Depending on the analytical method, one or more of these compounds is added to method blanks, calibration and check standards, and samples (including duplicates, matrix spike samples, duplicate matrix spike samples and laboratory control samples) prior to extraction and analysis in order to monitor the method performance on each sample. The percent recovery is calculated for each surrogate, and the recovery is a measurement of the overall method performance.

$$\text{Recovery (\%)} = (M/T) \times 100$$

Where: M = The measured concentration of analyte,
T = The theoretical concentration of analyte added.

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9.3.9 Laboratory Control Samples

The laboratory control sample (LCS) is an aliquot of analyte-free liquid, solid or air matrix to which known amounts of the method analyte(s) is (are) added. A reference material of known matrix type, containing certified amounts of target analytes, may also be used as an LCS. An LCS is prepared and analyzed at a minimum frequency of one LCS per 20 samples, with every analytical batch or as stated in the method, whichever is more frequent. The LCS sample is prepared and analyzed in exactly the same manner as the field samples.

The percent recovery of the target analytes in the LCS is compared to established control limits and assists in determining whether the methodology is in control and whether the laboratory is capable of making accurate and precise measurements at the required reporting limit. Comparison of batch-to-batch LCS analyses enables the laboratory to evaluate batch-to-batch precision and accuracy.

Recovery (%) = (M/T) x 100

Where: M = The measured concentration of analyte,
T = The theoretical concentration of analyte added.

9.3.10 Laboratory Fortified Blanks - LFB

A laboratory blank fortified at the MRL used to verify the minimum reporting limit. The LFB is carried through the entire extraction and analytical procedure. A LFB is required with every batch of drinking water samples.

9.3.11 Matrix Spikes (a.k.a. Laboratory Fortified Sample Matrix)

Matrix spiked samples are aliquots of samples to which a known amount of the target analyte (or analytes) is (are) added. The samples are then prepared and analyzed in the same analytical batch, and in exactly the same manner as are routine samples. For the appropriate methods, matrix spiked samples are prepared and analyzed and at a minimum frequency of one spiked sample (and one duplicate spiked sample, if appropriate) per twenty samples. The spike recovery measures the effects of interferences caused by the sample matrix and reflects the accuracy of the method for the particular matrix in question. Spike recoveries are calculated as follows:

Recovery (%) = (S - A) x 100 ÷ T

Where: S = The observed concentration of analyte in the spiked sample,
A = The analyte concentration in the original sample, and
T = The theoretical concentration of analyte added to the spiked sample.

Note: Matrix spiked samples are often not feasible for air matrices. Therefore, the MS shall be used as required by the test method and as specified by the corresponding method SOP.

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9.3.12 Laboratory Duplicates and Duplicate Matrix Spikes

Duplicates are additional replicates of samples that are subjected to the same preparation and analytical scheme as the original sample. Depending on the method of analysis, either a duplicate analysis (and/or a matrix spiked sample) or a matrix spiked sample and duplicate matrix spiked sample (MS/DMS) are analyzed. The relative percent difference between duplicate analyses or between an MS and DMS is a measure of the precision for a given method and analytical batch. The relative percent difference (RPD) for these analyses is calculated as follows:

$$\textit{Relative Percent Difference (RPD)} = (S1 - S2) \times 100 \div S_{ave}$$

Where S1 and S2 = The observed concentrations of analyte in the sample and its duplicate, or in the matrix spike and its duplicate matrix spike, and

S_{ave} = The average of observed analyte concentrations in the sample and its duplicate, or in the matrix spike and its duplicate matrix spike.

Depending on the method of analysis, either duplicates (and/or matrix spikes) or MS/DMS analyses are performed at a minimum frequency of one set per 20 samples. If an insufficient quantity of sample is available to perform a laboratory duplicate or duplicate matrix spikes, duplicate LCSs will be prepared and analyzed.

9.3.13 Control Charting

The generation of control charts is routinely performed at ALS Environmental. Surrogate, Matrix Spike and LCS recoveries are all monitored and charted. In addition, the laboratory also monitors the Relative Percent Difference (RPD) measurement of precision. Control charts are available to each individual laboratory unit to monitor the data generated in its facility using control charts that have been programmed to identify various trends in the analytical results. If trends in the data are perceived, various means of corrective action may then be employed in order to prevent future problems with the analytical system(s). Finally, data quality reports using control charts are generated for specific clients and projects pursuant to contract requirements. The control charting procedure is described in the SOP for *Control Limits* (CE-QA009).

9.3.14 Glassware Washing

Glassware washing and maintenance play a crucial role in the daily operation of a laboratory. The glassware used at ALS Environmental undergoes a rigorous cleansing procedure prior to every usage. The *SOP for Glassware Cleaning* (ADM-GLASS) has been generated and outlines the various procedures used at ALS Environmental-Simi Valley; each procedure is specific to the end-use of the equipment as well as to the overall analytical requirements of the project. In addition, other equipment that may be routinely used at the laboratory is also cleaned following instructions in the appropriate SOP.

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9.3.15 Collection Efficiency

In the case of sampling trains (consisting of one or more multi-section sorbent tubes), which are received intact by the laboratory, the "front" and "back" sections shall be separated if required by the client. Each section shall be processed and analyzed separately and the analytical results reported accordingly.

9.3.16 Desorption Efficiency and Method Reporting Limits (Industrial Hygiene)

Desorption efficiency (DE) is the ability of an analytical method to recover the analyte from the collection media. Desorption efficiencies are determined initially and for each analyte to be reported. In addition, a DE study is performed each time there is a change in the test method, or with each new lot of media. Desorption efficiency shall be determined using sorbent media from the same lot number used for the field samples, if possible, and of the identical size and type. The DE values are used to correct the sample results (for all samples except passive samplers) before reporting.

Minimum reporting limits for each reportable analyte are determined initially by the analysis of spiked media, prepared at the desired reporting limit and carried through the entire analytical process. The reporting limit is verified or re-established annually (or if there is a change in methodology or instrumentation) and instrument performance is checked with each analytical batch through the analysis of an analytical standard prepared at the reporting limit.

9.3.17 Field and Trip Blanks

Field and trip blanks are analyzed when they are submitted to the laboratory for analysis. The actual field samples are flagged (when analytes are found in the blank) if and only if the laboratory is able to analyze the samples in the same analytical sequence as the corresponding field or trip blank. If this is not possible due to client submission restrictions then the results for the samples and blanks shall be reported independently with no flag. However, an explanation of this is included in the final report. This laboratory does not feel that Summa canisters are suitable for use as trip blanks. It is for this reason that the results for these types of containers are reported as separate samples and flagging is not considered appropriate.

10) Data Processing, Validation, and Reporting

ALS Environmental reports the analytical data produced in its laboratories to the client via the certified analytical report. This report includes a transmittal letter, a case narrative, client project information, specific test results, quality control data, chain of custody information, and any other project-specific support documentation. The following procedures describe our data reduction, validation and reporting procedures.

10.1 Data Reduction and Review

Results are generated by the analyst who performs the analysis and works up the data. All data is initially reviewed and processed by analysts using appropriate methods (e.g., chromatographic software, instrument printouts, hand calculation,

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etc.). Equations used for calculation of results are found in the applicable analytical SOPs. The resulting data set is either manually entered into an electronic report form or is electronically transferred into the report from the software used to process the original data set (e.g., chromatographic software). The hardcopy version of the data is then reviewed by the analyst for accuracy. Once the primary analyst has checked the data for accuracy and acceptability, the hardcopy is forwarded to the supervisor or second qualified analyst, who reviews the data for errors. Where calculations are not performed using a validated software system, the reviewer rechecks a minimum of 10% of the calculations. When the entire data set has been found to be acceptable it is turned into the reporting department where final reports are generated and then validated by a Data Validation Coordinator. The hardcopy or electronic final report is physically or electronically signed by the project manager and the final report may be stored electronically or in hardcopy format. Test analysis data shall be kept in the appropriate service request folder. Data review and reporting procedures are described in the *SOP for Data Review and Reporting* (ADM-DATA_REV).

Policies and procedures for manual editing of data are established. The analyst making the change must initial and date the edited data entry, without obliteration of the original entry. The policies and procedures are described in the *SOP for Making Entries Onto Analytical Records* (ADM-DATANTRY).

Policies and procedures for electronic manual integration of chromatographic data are established. The analyst performing the integration must document the integration change by printing both the "before" and "after" integrations and including them in the raw data records. The policies and procedures are described in the *SOP for Manual Integration Policy* (CE-QA002).

10.2 Confirmation Analysis

10.2.1 Gas Chromatographic and Liquid Chromatographic Analyses

For gas chromatographic (GC) and liquid chromatographic (LC) analyses, all positive results are confirmed as required by the method, typically by a second column, a second detector, a second wavelength (HPLC/UV), or by GC/MS analysis, unless exempted by one of the following situations:

- The analyte of interest produces a chromatogram containing multiple peaks exhibiting a characteristic pattern, which matches appropriate standards. This is limited to petroleum hydrocarbon analyses (e.g., gasoline and diesel) and does not include polychlorinated biphenyls.
- The sample meets all of the following requirements:
    1. All samples (liquid or solid) come from the same source (e.g., groundwater samples from the same well) for continuous monitoring. Samples of the same matrix from the same site, but from different sources (e.g., different sampling locations) are not exempt.
    2. All analytes have been previously analyzed in sample(s) from the same source, identified and confirmed by a second column or by GC/MS. The chromatogram is largely unchanged from the one

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for which confirmation was carried out. The documents indicating previous confirmation must be available for review.

10.2.2 Confirmation Data

Confirmation data will be provided as specified in the method. Identification criteria for GC, LC or GC/MS methods are summarized below:

- GC and LC Methods
  1. The analyte must fall within plus or minus three times the standard deviation (established for the analyte/column) of the retention time of the daily midpoint standard in order to be qualitatively identified. The retention-time windows will be established and documented, as specified in the appropriate Standard Operating Procedure (SOP).
  2. When sample results are confirmed by two dissimilar columns or detectors, the agreement between quantitative results must be evaluated. The relative percent difference between the two results is calculated and evaluated against SOP and/or method criteria.
- GC/MS Methods - Two criteria are used to verify identification:
  1. Elution of the analyte in the sample will occur at the same relative retention time (RRT) as that of the analyte in the standard.
  2. The mass spectrum of the analyte in the sample must, in the opinion of a qualified analyst or the department manager, correspond to the spectrum of the analyte in the standard or the current GC/MS reference library.

10.3 Data Review and Validation of Results

The integrity of the data generated is assessed through the evaluation of the sample results, calibrations, and QC samples (method blanks, laboratory control samples, sample duplicates, matrix spikes, trip blanks, etc.). A brief description of the evaluation of these analyses is described below, with details listed in applicable SOPs. The criteria for evaluation of QC samples are listed within each method-specific SOP. Other data evaluation measures may include (as necessary) a check of the accuracy check of the QC standards and a check of the system sensitivity. Data transcriptions and calculations are also reviewed.

Note: Within the scope of this document, all possible data assessment requirements for various project protocols cannot be included in the listing below. This listing gives a general description of data evaluation practices used in the laboratory in compliance with NELAP Quality Systems requirements. Additional requirements exist for certain programs, such as projects under the DoD QSM protocols, and project-specific QAPPs.

- Method Calibration – Following the analysis of calibration blanks and standards according to the applicable SOP the calibration correlation

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coefficient, average response factor, etc. is calculated and compared to specified criteria. If the calibration meets criteria analysis may continue. If the calibration fails, any problems are isolated and corrected and the calibration standards reanalyzed. Following calibration and analysis of the independent calibration verification standard(s) the percent difference for the ICV is calculated. If the percent difference is within the specified limits the calibration is complete. If not, the problem associated with the calibration and/or ICV are isolated and corrected and verification and/or calibration is repeated.

- Continuing Calibration Verification (CCV) – Following the analysis of the CCV standard the percent difference is calculated and compared to specified criteria. If the CCV meets the criteria analysis may continue. If the CCV fails, routine corrective action is performed and documented and a 2nd CCV is analyzed. If this CCV meets criteria, analysis may continue, including any reanalysis of samples that were associated with a failing CCV. If the routine corrective action failed to produce an immediate CCV within criteria, then either acceptable performance is demonstrated (after additional corrective action) with two consecutive calibration verifications or a new initial calibration is performed.
- Method Blank – Results for the method blank are calculated as performed for samples. If results are less than the MRL (<½ MRL for DoD projects), the blank may be reported. If not, associated sample results are evaluated to determine the impact of the blank result. If possible, the source of the contamination is determined. If the contamination has affected sample results the blank and samples are reanalyzed. If positive blank results are reported, the blank (and sample) results are flagged with an appropriate flag, qualifier, or footnote.
- Sample Results (Inorganic) – Following sample analysis and calculations (including any dilutions made due to the sample matrix) the result is verified to fall within the calibration range. If not, the sample is diluted and analyzed to bring the result into calibration range. When sample and sample duplicates are analyzed for precision, the calculated RPD is compared to the specified limits.

  The sample and duplicate are reanalyzed if the criteria are exceeded. The samples may require re-preparation and reanalysis. Results are reported when within the calibration range, or as estimates when outside the calibration range. When dilutions are performed the MRL is elevated accordingly.
- Sample Results (Organic) – For GC/MS analyses, it is verified that the analysis was within the prescribed tune window. If not, the sample is reanalyzed. Following sample analysis and calculations (including any dilutions made due to the sample matrix) peak integrations, retention times, and spectra are evaluated to confirm qualitative identification. Internal standard responses and surrogate recoveries are evaluated against specified criteria. If internal standard response does not meet criteria, the sample is diluted and reanalyzed. Results outside of the calibration range

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are diluted to within the calibration range. When dilutions are performed the MRL is elevated accordingly.

- Surrogate Results (Organic) – The percent recovery of each surrogate is compared to specified control limits. If recoveries are acceptable, the results are reported. If recoveries do not fall within control limits, the sample matrix is evaluated. When matrix interferences are present or documented, the results are reported with a qualifier that matrix interferences are present. If no matrix interferences are present and there is no cause for the outlier, the sample is reanalyzed. However, if the recovery is above the upper control limit with non-detected target analytes, the sample may be reported. All surrogate recovery outliers are appropriately qualified on the report.
- Duplicate Sample and/or Duplicate Matrix Spike Results – The RPD is calculated and compared to the specified control limits. If the RPD is within the control limits the result is reported. If not, an evaluation of the sample is made to verify that a homogenous sample was used and the results are compared to the MRL. The samples and duplicates are reanalyzed and if re-analysis also produces out-of-control results, the results are reported with an appropriate qualifier.
- Laboratory Control Sample Results – Following analysis of the LCS the percent recovery is calculated and compared to specified control limits. If the recovery is within control limits, the analysis is in control and results may be reported. If not, this indicates that the analysis is not in control. Samples associated with the 'out of control' LCS, shall be considered suspect and the samples reanalyzed or the data reported with the appropriate qualifiers.
- Matrix Spike Results – Following analysis of the MS the percent recovery is calculated and compared to specified control limits. If the recovery is within control limits the results may be reported. If not, and the LCS is within control limits, this indicates that the matrix potentially biases analyte recovery. It is verified that the spike level is at least five times the background level. If not, the results are reported with a qualifier that the background level is too high for accurate recovery determination. If matrix interferences are present or results indicate a potential problem with sample preparation, steps may be taken to improve results; such as dilution and reanalysis, or re-preparation and reanalysis. Results that do not meet acceptance limits are reported with an appropriate qualifier.

10.4 Data Reporting

When an analyst determines that a data package has met the data quality objectives (and/or any client-specific data quality objectives) of the method and has qualified any anomalies in a clear, acceptable fashion, the data package will undergo a peer review by a trained chemist. Prior to release of the report to the client, the Project Manager reviews and approves the entire report for completeness and to ensure that any and all client-specified objectives were successfully achieved. The original raw test data, along with a copy of the final report, is retained by service request number for archival purposes. ALS

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Environmental maintains control of analytical results by adhering to standard operating procedures and by observing sample custody requirements. All data is calculated and reported in units consistent with project specifications, to enable easy comparison of data from report to report.

To the extent possible, samples shall be reported only if all QC measures are acceptable. If a QC measure is found to be out of control, and the data is to be reported, all samples associated with the failed quality control measure shall be reported with the appropriate data qualifier(s). The *SOP for Data Review and Reporting* (ADM-DATA_REV) addresses the flagging and qualification of data. The ALS Environmental-defined data qualifiers, state-specific data qualifiers, or project-defined data qualifiers are used depending on project requirements. A case narrative may be written by the analyst or project manager to explain problems with a specific analysis or sample, etc.

For subcontracted analyses, the Project Manager verifies that the report received from the subcontractor is complete. This includes checking that the correct analyses were performed, the analyses were performed for each sample as requested, a report is provided for each analysis, and the report is signed. The Project Manager accepts the report if all verification items are complete. Acceptance is demonstrated by forwarding the report to the ALS Environmental client.

10.5 Documentation

ALS Environmental maintains a records system which ensures that all laboratory records of analysis data are retained and available. Analysis data is retained for 5 years from the report date unless contractual terms or regulations specify a longer retention time. Archival procedures are described in the *SOP for Data and Record Archiving* (ADM-ARC).

10.5.1 Documentation and Archiving of Sample Analysis Data

The archiving system includes, but is not limited to, the following items (where applicable) for each set of analyses performed:

- Benchsheets describing sample preparation (if appropriate) and analysis;
- Instrument parameters (or reference to the data acquisition method);
- Sample analysis sequence;
- Instrument printouts, including chromatograms and peak integration reports for all samples, standards, blanks, spikes, duplicates and reruns;
- Applicable standard identification numbers;
- Chain of custody, service request and sample acceptance check forms;
- Initial calibration and data review checklist(s);
- Copies of report sheets submitted to the work request file; and
- Copies of Nonconformity and Corrective Action Reports, if necessary.

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Individual sets of analyses are identified by analysis date and service request number. Since many analyses are performed with computer-based data systems, the final sample concentrations can be automatically calculated. If additional calculations are needed, they are written on the integration report or securely stapled to the chromatogram, if done on a separate sheet.

For organics analysis, data applicable to all analyses within the batch, such as GCMS tunes, CCVs, batch QC, and analysis sequences; are kept using a separate documentation system. This system is used to archive data on a batch-specific basis and is segregated according to the date of analysis. This system also includes results for the most recent calibration curves, as well as method validation results.

10.6 Deliverables

In order to meet individual project needs, ALS Environmental provides several levels of analytical reports. Standard specifications for each level of deliverable are described in Table 10-1. Variations may be provided based on client or project specifications. This includes (but is not limited to) the following specialized deliverables:

- ACOE/HTRW – Army Corps of Engineers specified data package and reporting requirements (HTRW, CERP, FUDS, etc.)
- AFCEE – Air Force Center for Environmental Excellence project-specific reporting

When requested by the client or relevant to the validity of reported results, the estimation of measurement uncertainty will be provided to a client or regulatory agency. How the uncertainty will be reported may be dictated by the client's reporting specifications. Procedures for determining and reporting uncertainty are given in the *SOP for Estimation of Uncertainty of Measurements* (ADM-UNCERT).

When requested, ALS Environmental provides Electronic Data Deliverables (EDDs) in the format specified by client need or project specification. ALS Environmental is capable of generating EDDs with many different formats and specifications. The EDD is prepared by report production staff using the electronic version of the laboratory report to minimize transcription errors. User guides and EDD specification outlines are used in preparing the EDD. The EDD is reviewed and compared to the final report for accuracy.

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Table 10-1
Descriptions of ALS Environmental Standard Data Deliverables

Tier I. Routine Certified Analytical Report includes the following:

1. Transmittal letter
2. Chain of custody documents and sample/cooler receipt documentation
3. Sample analytical results
4. Method blank results
5. Surrogate recovery results and acceptance criteria for applicable organic methods
6. Dates of sample preparation and analysis for all tests
7. Case narrative - optional

Tier II. In addition to the Tier I Deliverables, this includes the following:

1. Matrix spike result(s) with calculated recovery and including associated acceptance criteria
2. Duplicate or duplicate matrix spike result(s) (as appropriate to method), with calculated relative percent difference
3. Laboratory Control Sample result(s) with calculated recovery and including associated acceptance criteria
4. Case narrative - optional

Tier III. Data Validation Package. In addition to the Tier II Deliverables, this includes the following:

1. Case narrative - required
2. Summary forms for all associated QC and Calibration parameters, with associated control criteria/acceptance limits

Note: Other summary forms specified in QAPPs or project/program protocols, or those related to specialized analyses such as HRGC/MS will be included.

Tier IV. Full Data Validation Package:

1. All raw data associated with the sample analysis, including but not limited to:
   a. Preparation and analysis bench sheets and instrument printouts,
   b. For organics analyses, all applicable chromatograms, spectral, confirmation, and manual integration raw data. For GC/MS this includes tuning results, mass spectra of all positive hits, and the results and spectra of TIC compounds when requested.
   c. QC data,
   d. Calibration data (initial, verification, continuing, etc),
   e. Calibration blanks or instrument blanks (as appropriate to method).
2. If a project QAPP or program protocol applies, the report will be presented as required by the QAPP.

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11) Performance and System Audits

Quality audits are an essential part of ALS Environmental-Simi Valley's quality assurance program. There are two types of audits used at the facility: System Audits are conducted to qualitatively evaluate the operational details of the QA program, while Performance Audits are conducted by analyzing proficiency testing samples in order to quantitatively evaluate the outputs of the various measurement systems.

11.1 System Audits

The system audit examines the presence and appropriateness of laboratory systems. External system audits of ALS Environmental-Simi Valley are conducted regularly by various regulatory agencies and clients. Appendix G lists the certification and accreditation programs in which ALS Environmental-Simi Valley participates. Programs and certifications are added as required. Additionally, internal system audits of ALS Environmental-Simi Valley are conducted regularly under the direction of the QA Manager. The internal audit procedures are described in the *SOP for Internal Audits* (CE-QA001). The internal audits are performed as follows:

- Comprehensive lab-wide system audit – performed annually. This audit is conducted such that all elements of the ALS Quality System are assessed.
- Technical/method audits
- Hardcopy report audits

All audit findings, and corrective actions are documented. The results of each audit are reported to the Laboratory Director and Department Managers for review. Any deficiencies identified are summarized in the audit report. Managers must respond with corrective actions correcting the deficiency within a defined timeframe. Should problems impacting data quality be found during an internal audit, any client whose data is adversely impacted will be given written notification within the corrective action period (if not already provided).

Electronic data audits may be performed in conjunction with hardcopy data audits. The electronic audits focus on organic chromatographic data and include an examination of audit trails, peak integrations, calibration practices, GCMS tuning data, peak response data, use of appropriate files, and other components of the analysis. The audit also verifies that the electronic data supports the hardcopy reported data.

Additional internal audits or data evaluations may be performed as needed to address any potential data integrity issues that may arise.

11.2 Performance Audits

ALS Environmental-Simi Valley also participates in the analysis of interlaboratory proficiency testing (PT) samples. Participation in PT studies is performed on a regular basis and is designed to evaluate all analytical areas of the laboratory. General procedures for these analyses are described in the *SOP for Proficiency Sample Testing Analysis* (CE-QA006). ALS Environmental-Simi Valley routinely participates in the following studies:

- Water Pollution (WP) PT studies, 2 per year.
- Water Supply (WS) PT studies, 2 per year.

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- Hazardous Waste/Soil PT studies, 2 per year.
- American Industrial Hygiene Association (AIHA) PT Program, 4 per year
- Air and Emissions PT studies, 2 per year
- Other studies as required for specific certifications, accreditations, or validations.

PT samples are processed by entering them into the LIMS system as samples (assigned Service Request, due date, testing requirements, etc.) and are processed the same as field samples. The laboratory sections handle samples the same as field samples, performing the analyses following method requirements and performing data review. The laboratory sections submit results to the QA Manager for subsequent reporting to the appropriate agencies or study provider. Results of the performance evaluation samples and audits are reviewed by the QA Manager, Laboratory Director, the laboratory staff, and the Corporate Quality Assurance Manager. For any results outside acceptance criteria, the analysis data is reviewed to identify a root cause for the deficiency, and corrective action is taken and documented through nonconformance (NCAR) procedures.

12) Preventive Maintenance

Preventive maintenance is a crucial element of the Quality Assurance program. Instruments at ALS Environmental (e.g., GC/MS systems, gas and liquid chromatographs, analytical balances, gas and liquid chromatographs, etc.) are maintained under commercial service contracts or by qualified, in-house personnel. All instruments are operated and maintained according to the instrument operating manuals. All routine and special maintenance activities pertaining to the instruments are recorded in instrument maintenance logbooks. The maintenance logbooks used at ALS Environmental contain extensive information about the instruments used at the laboratory.

An initial demonstration of analytical control is required on every instrument used at ALS Environmental before it maybe used for sample analysis. Each instrument must be recalibrated following any instrument maintenance which may change or effect the sensitivity or linearity of the instrument or if the continuing calibration verification acceptance criteria have not been met as specified in the standard operating procedure. If an instrument is modified or repaired, a return to analytical control is required before subsequent sample analyses can occur. When an instrument is acquired at the laboratory, the following information is noted in a bound maintenance notebook specifically associated with the new equipment:

- The equipment's serial number;
- Date the equipment was received;
- Date the equipment was placed into service;
- Condition of equipment when received (new, used, reconditioned, etc.); and
- Prior history of damage, malfunction, modification or repair (if known).

Preventive maintenance procedures, frequencies, etc. are available for each instrument used at ALS Environmental. They may be found in the various SOPs for routine methods performed on an instrument and may also be found in the operating or maintenance manuals provided with the equipment at the time of purchase.

Responsibility for ensuring that routine maintenance is performed lies with the department supervisor or laboratory director. The supervisor may perform the maintenance or assign

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the maintenance task to a qualified bench level analyst who routinely operates the equipment. In the case of non-routine repair of capital equipment, the department supervisor is responsible for providing the repair, either by performing the repair themselves with manufacturer guidance or by acquiring on-site manufacturer repair. The laboratory maintains an adequate supply of expendable maintenance items (expected lifetime of part of less than 1 year.) These parts include items needed to perform the preventive maintenance procedures listed in Appendix E.

When performing maintenance on an instrument (whether preventive or corrective), additional information about the problem, attempted repairs, etc. is also recorded in the notebook. Typical logbook entries include the following information:

- Details and symptoms of the problem;
- Repairs and/or maintenance performed;
- Description and/or part number of replaced parts;
- Source(s) of the replaced parts;
- Analyst's signature and date; and
- Demonstration of return to analytical control.

See the table in Appendix E for a list of preventive maintenance activities and frequency for each instrument.

For further information regarding Instrumentation see the *SOP for Analytical Instrument Acquisition, Reassignment, Maintenance and Documentation* (ADM-INSTRUM).

13) Corrective and Preventive Action

The laboratory takes all appropriate steps necessary to ensure all sample results are reported with acceptable quality control results. When sample results do not conform to established quality control procedures, responsible management will evaluate the significance of the nonconforming work and take corrective action to address the nonconformance.

Nonconforming events such as errors, deficiencies, deviations from SOP, proficiency (PT) failure or results that fall outside of established QC limits are documented using a *Nonconformity and Corrective Action Report* form. The procedure and responsibilities for addressing nonconforming work is defined in the *SOP for* Nonconformance and *Corrective Action* (CE-QA008). Nonconformances are reported to the client using various means (voice, email, narrative, etc). When a nonconformance occurs that casts doubt on the validity of the test results or additional client instructions are needed, the Project Manager notifies the client the same business day that the nonconformance is confirmed and reported. The QA Manager reviews each problem, ensuring that appropriate corrective action has been taken by the appropriate personnel. The Nonconformity and Corrective Action Report (NCAR) is filed in the associated service request file and a copy is kept by the QA Manager. The QA Manager periodically reviews all NCARs looking for chronic, systematic problems that need more in-depth investigation and alternative corrective action consideration. In addition, the appropriate Project Manager is promptly notified of any problems in order to inform the client and proceed with any action the client may want to initiate.

If a quality control measure is found to be out of control, and the data is to be reported, all samples associated with the failed quality control measure shall be reported with the

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appropriate data qualifier(s). Failure to meet established analytical controls, such as the quality control objectives, prompts corrective action. Corrective action may take several forms and may involve a review of the calculations, a check of the instrument maintenance and operation, a review of analytical technique and methodology, and reanalysis of quality control and field samples. If a potential problem develops that cannot be solved directly by the responsible analyst, the supervisor, team leader, the department manager, and/or the QA Manager may examine and pursue alternative solutions. In addition, the appropriate Project Manager is notified in order to ascertain if the client needs to be notified.

Part of the corrective action process involves determining the root cause. Identifying the root cause of a nonconformance can be difficult, but important for implementing effective corrective action. Root cause principles are used to determine assignable causes, which leads to corrective action taken to prevent recurrence. Various preventive action processes are used for eliminating a potential problem or averting a problem before it occurs. This is explained in the *SOP for Preventive Action* (CE-GEN004).

In addition to internal communication of data issues, the laboratory also maintains a system for dealing with customer complaints. The person who initially receives the feedback (typically the Project Manager) is responsible for documenting the complaint. If the Project Manager is unable to satisfy the customer, the complaint is brought to the attention of the Client Services Manager, Laboratory Director, or QA Manager for final resolution. The complaint and resolution are documented. The procedure is described in the *SOP for Handling Customer Feedback* (ADM-FDBK).

14) Quality Assurance Reports and Management Review

Quality assurance requires an active, ongoing commitment by ALS Environmental personnel at all levels of the organization. Communication and feedback mechanisms are designed so that analysts, supervisors and managers are aware of QA issues in the laboratory. Analysts performing routine testing are responsible for generating a data quality narrative or data review document with every analytical batch processed. This report also allows the analyst to provide appropriate notes and/or a narrative if problems were encountered with the analyses. A Non-Conformity and Corrective Action Report (NCAR) (see Section 13.0) may also be attached to the data prior to review. Supervisors or qualified analysts review all of the completed analytical batches to ensure that all QC criteria have been examined and any deficiencies noted and addressed.

It is the responsibility of each laboratory unit to provide the reporting department with reviewed data accompanied by signature approval. The data validation coordinators provide the Project Manager with a final report of the data. Footnotes and/or narrative notes must accompany any data package if problems were encountered that require further explanation to the client. Each data package is submitted to the appropriate Project Manager, who in turn reviews the entire collection of analytical data for completeness and to ensure that any and all client-specified objectives were successfully achieved. A case narrative is written (or approved) by the Project Manager to explain any unusual problems with a specific analysis or sample, etc.

The QA Manager provides overview support to the Project Managers as required (e.g., contractually specified, etc.). The QA Manager is also responsible for the oversight of all internal and external audits, for all proficiency testing sample and analysis programs, and for all laboratory certification/accreditation responsibilities. The QA Manager regularly

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communicates with the Laboratory Director to review the various QA/QC activities, priorities, and status of program implementation; including such topics as the following:

- Status, schedule, and results of internal and external audits;
- Status, schedule, and results of internal and external proficiency testing studies;
- Status of certifications, accreditations, and approvals;
- Status of QA Manual and SOP review and revision;
- Status of MDLs studies;
- Discussion of QC problems in the laboratory;
- Discussion of corrective action program issues;
- Status of staff training and qualification; and
- Other topics as appropriate.

An annual management review of the quality and testing systems is performed as described in the *SOP for Laboratory Management Review* (CE-QA005). This is done to identify any necessary changes or improvements to the quality system or quality assurance policies. This review is documented in a Managerial Review of the Laboratory's Quality Systems and Testing Activities and sent to senior management.

15) Personnel Training

Technical position descriptions are available for all employees, regardless of position or level of seniority. These documents are maintained by the Human Resources personnel and are available for review. In order to assess the technical capabilities and qualifications of a potential employee, all candidates for employment at ALS Environmental are evaluated, in part, against the appropriate technical description.

Training begins the first day of employment at ALS Environmental when the company policies are presented and discussed. Safety and QA/QC requirements are integral parts of all technical SOPs and, consequently, are integral parts of all training processes at ALS Environmental. Safety training begins with reading the *Environmental Health and Safety Manual.* Employees are also required to participate in periodic safety training performed by the Environmental, Health and Safety Coordinator.

Employees are responsible for complying with the requirements of the QA Manual and QA/QC requirements associated with their function(s). Quality Systems training begins with Quality Assurance orientation for new employees and reading the Quality Assurance Manual. During the employees first year, the employee attends Core Ethics training and learns about ALS Environmental quality systems. Each employee participates in annual Ethics Refresher training, which is part of the ALS Environmental Improper Practices Prevention Program.

ALS Environmental also encourages its personnel to continue to learn and develop new skills that will enhance their performance and value to the Company. Ongoing training occurs for all employees through a variety of mechanisms. The corporate, company-wide training and development program, external and internal technical seminars and training courses, and laboratory-specific training exercises are all used to provide employees with professional growth opportunities.

All technical training is documented and records are maintained by the QA department. Training requirements and its documentation are described in the *SOP for Training Policy* (CE-QA003). A training plan is developed whenever an employee starts a new

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procedure or new position. The training plan includes a description of the step-by-step process for training an employee and for initial demonstration of capability. Where the analyst performs the entire procedure, a generic training plan may be used.

15.1 Initial Demonstration of Capability (IDOC)

Training in analytical procedures typically begins with the reading of the Standard Operating Procedure (SOP) for the method. Hands-on training begins with the observation of an experienced analyst performing the method, followed by the trainee performing the method under close supervision, and culminating with independent performance of the method on quality control samples. Successful completion of the applicable Demonstration of Capability analysis qualifies the analyst to perform the method independently. Demonstration of Capability is performed by one of the following:

- Successful completion of an Initial Precision and Recovery (IPR) study (required where mandated by the method).
- Analysis of 4 consecutive Laboratory Control Samples, with acceptable accuracy and precision.
- Where spiking is not possible but QC standards are used ("non-spiked" Laboratory Control Samples), analysis of 4 consecutive Laboratory Control Samples with acceptable accuracy and precision.
- Where one of the three above is not possible training is performed and supervisor approval is documented.

A flowchart identifying the Demonstration of Proficiency requirements is given in Figure 15-1. The flowchart identifies allowed approaches to assessing Demonstration of Capability when a 4-replicate study is not mandated by the method, when spiking is not an option, or when QC samples are not readily available.

15.2 Continuing Demonstration of Proficiency

A periodic demonstration of proficiency is required to maintain continuing qualification. Continuing Demonstration of Proficiency is required each year, and may be performed one of the following ways:

- Successful performance on external (independent) single-blind sample analyses using the test method, or a similar test method using the same technology. I.e. PT sample or QC sample blind to the analyst.
- Performing Initial Demonstration of Capability as described above, with acceptable levels of precision and accuracy.
- Analysis of at least 4 consecutive LCSs with acceptable levels of accuracy and precision from in-control analytical batches.
- If the above cannot be performed, analysis of authentic samples with results statistically indistinguishable from those obtained by another trained analyst.
- For methods for which PT samples are not available and a spiked analysis (LFB, MDL, etc.) is not possible, analysis of field samples that have been analyzed by another analyst with statistically indistinguishable results.

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15.3 Documentation of Training

Records are maintained to indicate the employee has the necessary training, education, and experience to perform their functions. Information of previously acquired skills and abilities for a new employee is maintained in Human Resources personnel files and ALS Environmental resumes. QA maintains a database to record the various technical skills and training acquired while employed by ALS Environmental. Information includes the employee's name, a description of the skill including the appropriate method and SOP reference, the mechanism used to document proficiency, and the date the training was completed. General procedures for documenting technical training are described in the *SOP for Training Policy* (CE-QA003).

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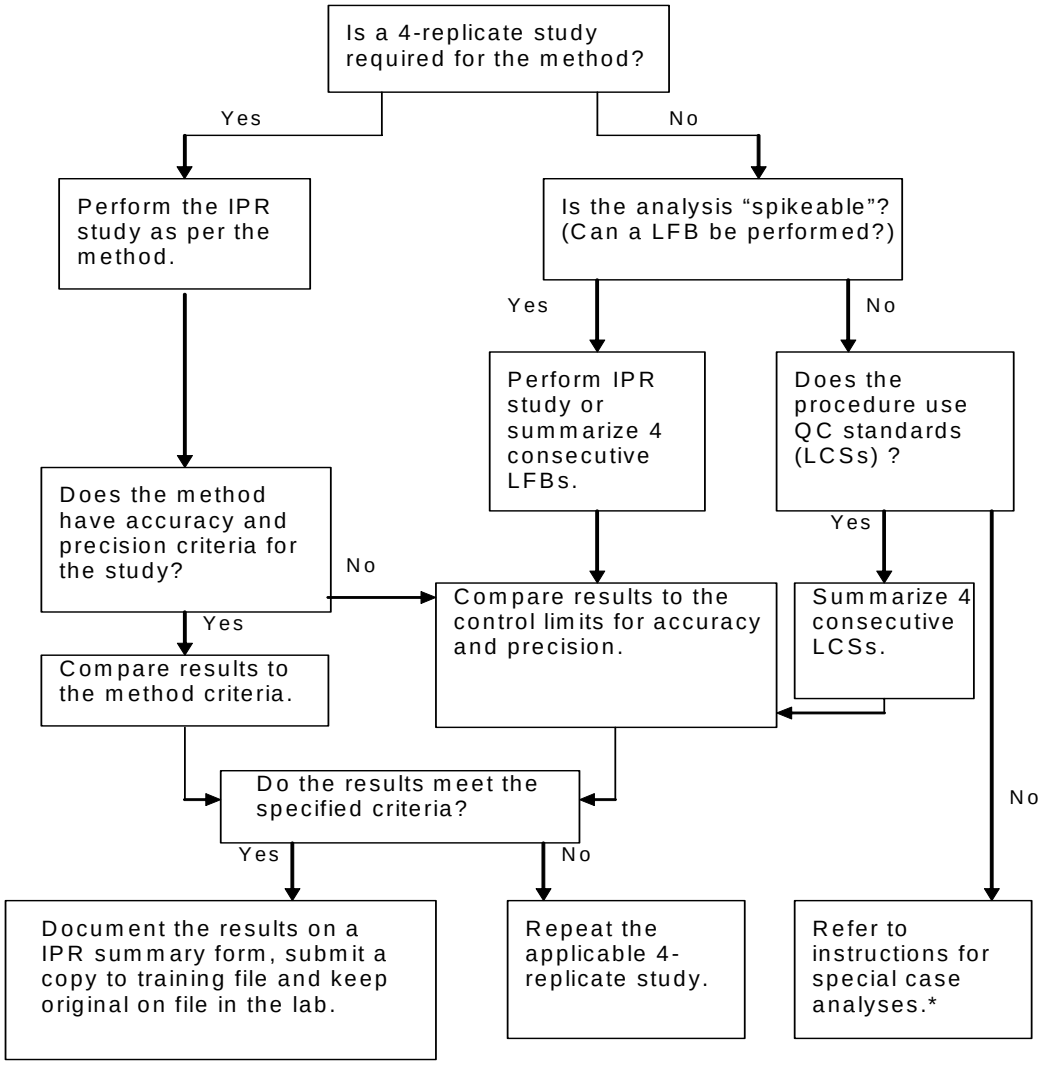
Figure 15-1
Initial Demonstration of Capability Requirements[a]



[a] For IDOC IPR or LFB studies, "second-source" reference materials are used, as per TNI/NELAP requirements
* Refer to the SOP for Training Policy for details. References for Quality Systems, External Documents, Manuals, Standards, and Analytical Procedures

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16) References for Quality Systems, External Documents, Manuals, Standards, and Analytical Procedures

The analytical methods used at ALS Environmental generally depend upon the end-use of the data. Since most of our work involves the analysis of environmental samples for regulatory purposes, specified federal and/or state testing methodologies are used and followed closely. Typical methods used at ALS Environmental are taken from the following references:

- National Environmental Laboratory Accreditation Program (NELAP), 2003 Quality Standards
- 2009 TNI Standards.
- American National Standard *General requirements for the competence of testing and calibration laboratories,* ANSI/ISO/IEC 17025:2005(E)
- *DoD Quality Systems Manual for Environmental Laboratories,* Version 4.2, 10/25/2010
- American Industrial Hygiene Association-LAP, LLC Policy Document Modules (2A Revision 11, July 15, 2011; 2B Revision 9, July 15, 2011; 6 Revision 0, July 15, 2011), Appendix G (Revision 1, July 15, 2011), and Appendix H (Revision 0, February 17, 2010).
- 3M Organic Vapor Monitor Sampling and Analysis Guide, *Organic Vapor Monitors 3500/3510 and Organic Vapor Monitors 3520/3530*, Technical Bulletin 1028, January 1, 2004.
- 40 CFR Part 60, Test Methods for Standards of Performance for New Stationary Sources, Appendix A.
- 40 CFR Part 63, Methods for Organic Chemical Analysis of Municipal and Industrial Wastewater, Appendix A.
- 40 CFR Part 63, National Emission Standards for Hazardous Air Pollutants for Source Categories, Subchapter C.
- 40 CFR Part 136, Definition and Procedure for the Determination of the Method Detection Limit, Appendix B
- American Society for Testing and Materials (ASTM), Gaseous Fuel, Coal and Coke, Volume 05.06, September 2006.
- American Society for Testing and Materials (ASTM), Annual Book of ASTM Standards, Philadelphia, PA.
- Arizona Administrative Code, *Department of Health Services – Laboratories*, Title 9, Ch. 14, Article 6. *Licensing of Environmental Laboratories*, R9-14-601 through R9-14-621, December 31, 2006 (Supp. 06-4)
- California Environmental Protection Agency Air Resources Board, *Methods for Determining Emissions of Toxic Air Contaminants from Stationary Sources*, Volume 3, July 28, 1997.

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- California Code of Regulations (CCR), Title 22, Chapter 11 *Identification and Listing of Hazardous Waste*, 7/20/05.
- Minnesota Administrative Rules, *Department of Health*, Chapter 4740, Laboratories; Accreditation Requirements.
- *Good Automated Laboratory Practices, Principles and Guidance to Regulations For Ensuring Data Integrity In Automated Laboratory Operations*, EPA 2185 (August 1995).
- Environmental Protection Agency, Methods Update Rule (MUR), Guidelines for Establishing Test Procedures for the Analysis of Pollutants Under the Clean Water Act; Analysis and Sampling Procedures; 40 CFR Parts 122, 136, 143, 430, 455 & 465; Final Rule 3/12/07, Effective April 11, 2007.
- Environmental Protection Agency, *Test Methods for Evaluating Solid Waste, Physical/Chemical Methods,* SW-846, Third Edition, 1986 and Updates I (7/92), II (9/94), III (12/96), IIIA (4/98), IIIB (11/04), IVA & IVB. See Chapters 1, 2, 3, 4, 5, 6, and 8.
- Environmental Protection Agency, *Methods for Chemical Analysis of Water and Wastes*, EPA-600/4-79-020, 1983.
- Environmental Protection Agency, *Methods for the Determination of Inorganic Substances in Environmental Samples,* EPA 600/R-93-100, August 1993.
- Environmental Protection Agency, *EPA Compendium of Methods for the Determination of Toxic Organic Compounds in Ambient Air*, Second Edition, EPA/625/R-96-010b, January 1999.
- Environmental Protection Agency, *EPA Compendium of Methods for the Determination of Toxic Organic Compounds in Ambient Air*, Second Edition Addendum, October 4, 2000.
- HQ Air Force Center for Environmental Excellence, Technical Services Quality Assurance Program, Guidance for Contract Deliverables, Appendix C: Quality Assurance Project Plan (QAPP), Final Version 4.0.02, May 2006.
- National Institute for Occupational Safety and Health (NIOSH) *Manual of Analytical Methods*, Third Edition (August 1987); Fourth Edition (August 1994); 1st Supplement Publication 96-135, 2nd Supplement Publication 98-119, 3rd Supplement 2003-154
- National Council for Air and Stream Improvement, Inc. (NCASI). 2007. *Appendix E - Technical Bulletin Cross Reference Guide for NCASI Methods.* Methods Manual (05).
- *SKC 575 Series Passive Sampler Rate/Selection Guide,* Form #37021, Rev 0012.
- *Standard Methods for the Examination of Water and Wastewater*, 20th Edition (1998).
- South Coast Air Quality Management District, *Laboratory Methods of Analysis for Enforcement Samples.*
- U.S. Department of Labor, Occupational Safety and Health Administration *OSHA Analytical Methods Manual.*

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APPENDIX A - Approved Signatories, QA Program Documents, and Corporate SOP List

Name / Title	Signature	Initials	Years of Experience
Kelly Horiuchi, B.A.* *Laboratory Director / Project Manager*		KMH	12
Ku-Jih Chen, B.S. *Principle Chemist*		KJC	37
Chaney Humphrey, B.S.* *Quality Assurance Manager*		CH	8
Wade Henton, B.S.* *Team Leader (VOA GC - Air)*		WH	26
Chris Parnell, B.S.* *Operations Manager / Technical Director (VOA GC/MS – Air)*		CP	26
Madeleine Dangazyan, B.S.* *Team Leader (Semi-Volatiles/ Industrial Hygiene) / Environmental Health & Safety Coordinator*		MD	17
Wida Ang, B.S., M.S.* *Team Leader (Volatiles GC/MS – Air)*	On File	On File	27
Sue Anderson, B.S.* *Project Manager / Technical Director (General Chemistry)*			22
Samantha Henningsen, B.S. * *Project Manager*		SH	3
Kathleen Aguilera, B.A.* *Client Services Manager / Project Manager*	On File	On File	23
Robin Gill *Data Validation Coordinator / Team Leader (Reporting)*		RG	32
Rusty Bravo, B.S. *Data Validation Coordinator*			22
Robert De La O *Systems Analyst / IT*			22
Shreejana Singh, B.S. *Systems Analyst*		SSM	7

* Approved Signatory

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Name / Title	Signature	Initials	Years of Experience
Manny Zamora *Team Leader (Sample Management)*	(b)(4)		
Karen Ryan, B.S. *Team Leader (Canister Cleaning & Shipping) / Alternate Sample Management Custodian*			
Sasha Sommer, B.S. *GC/MS Analyst*			
Llesenia Cercado, A.S. *GC/MS Analyst*			
Simon Cao, B.S. *GC/MS Analyst*			
Lusine Hakobyan, B.S. *GC/MS Analyst*			
Elsa Moctezuma, B.S. *GC/MS Analyst*			
Chris Cornett, B.S., M.S. *GC/MS Analyst*			
John Rice, B.S. *GC/MS Analyst*			
Michael Conejo, B.A. *GC Analyst*			
Jennifer Young, B.S., M.S. *GC Analyst*			
Zheng Wang, B.S., M.S. *SVOA Analyst*			
Evelyn Ibarra, B.S. *SVOA Analyst*			
Gilbert Gutierrez *SVOA Technician*			

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QA Program Files	
Item	**Location / Name**
Software Quality Assurance Plan	Q:\SOPs (PDF)\CORP\SQAP Rev 8.pdf
Simi Valley Certification Status	Q:\Certifications\Cert Status.xls
Control Limit\Chart Status	Q:\Control Charts\CntrlChrt(status1).xls
MDL,LOD,LOQ Status	Q:\MDL Status\MDL Status Table (EACH DEPT).xls
Technical Training Status	Q:\Training\TRAINING STATUS\TRAINING STATUS.xls
Simi Valley Data Quality Objectives	Q:\MDL_MRL\DQO Spreadsheet.xls
Master List of Controlled Documents (Logbooks, SOPs, etc.)	Q:\Master List of Controlled Documents\Master List of Controlled Documents.xls
Personnel Resumes, Transcripts	HR and QA Departments
Job Descriptions	HR Department
Approved Signatories List	QA Manual Appendix A

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Corporate SOP Titles	SOP ID
Laboratory Ethics and Data Integrity	CE-GEN001
Records Management Policy	CE-GEN003
Preventive Action	CE-GEN004
Document Control	CE-GEN005
Data Recall	CE-GEN006
Procurement and Control of Laboratory Services and Supplies	CE-GEN007
Method Development	CE-GEN008
Establishing Standard Operating Procedures	CE-GEN009
Internal Audits	CE-QA001
Manual Integration Policy	CE-QA002
Training Policy	CE-QA003
Qualification of Subcontract Laboratories	CE-QA004
Laboratory Management Review	CE-QA005
Proficiency Testing Sample Analysis	CE-QA006
Making Entries Onto Analytical Records	CE-QA007
Nonconformance and Corrective Action	CE-QA008
Control Limits	CE-QA009
Checking New Lots of Chemicals for Contamination	ADM-CTMN
Handling Customer Feedback	ADM-FDBK
Performing Method Detection Limit Studies and Establishing Limits of Detection and Quantitation	ADM-MDL
Significant Figures	ADM-SIGFIG
Tape Backup and Tape Archiving	ADM-TapeBU
Estimation of Uncertainty of Measurements	ADM-UNCERT

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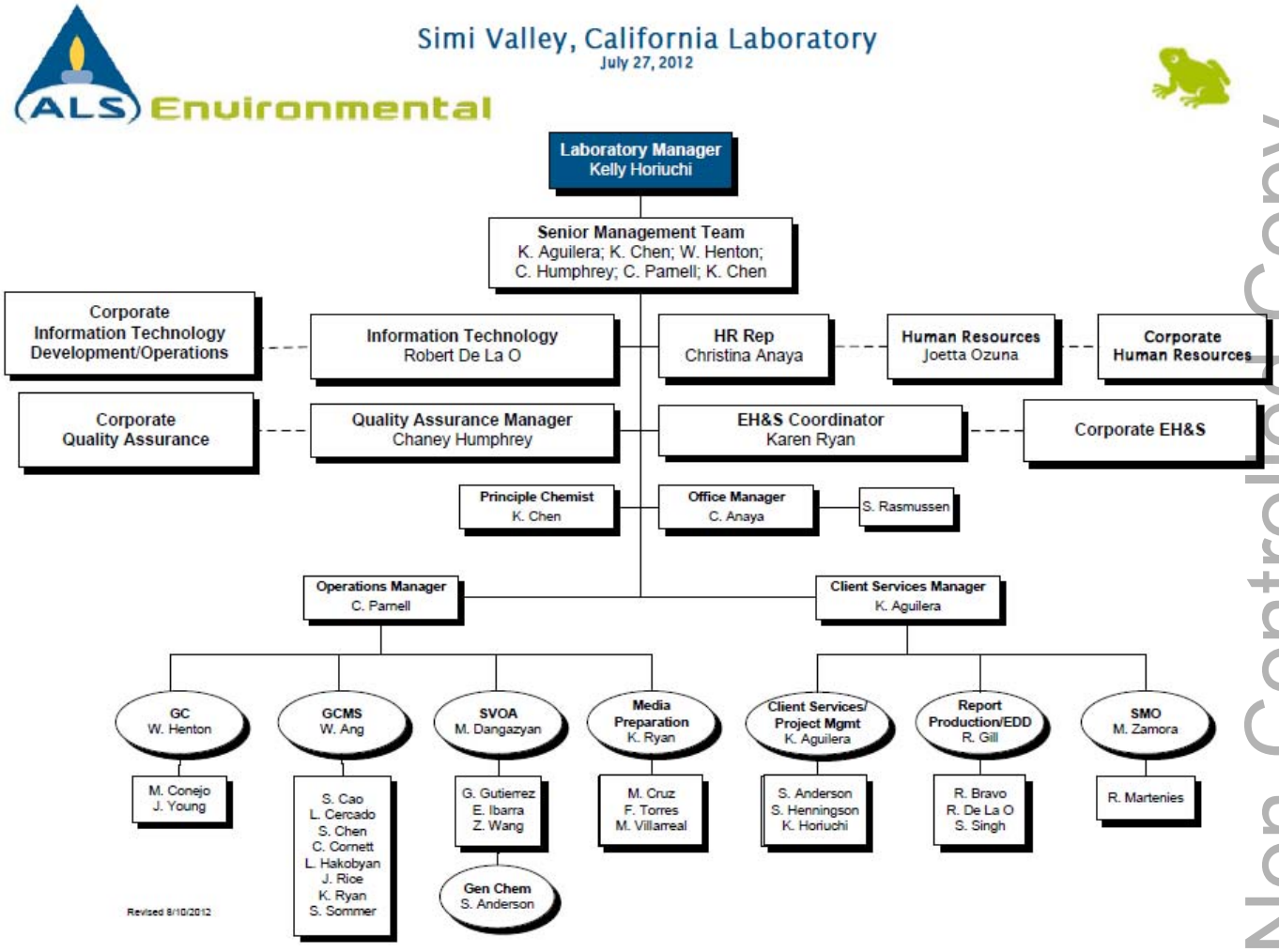
APPENDIX B - Organizational Chart and Resumes of Key Personnel



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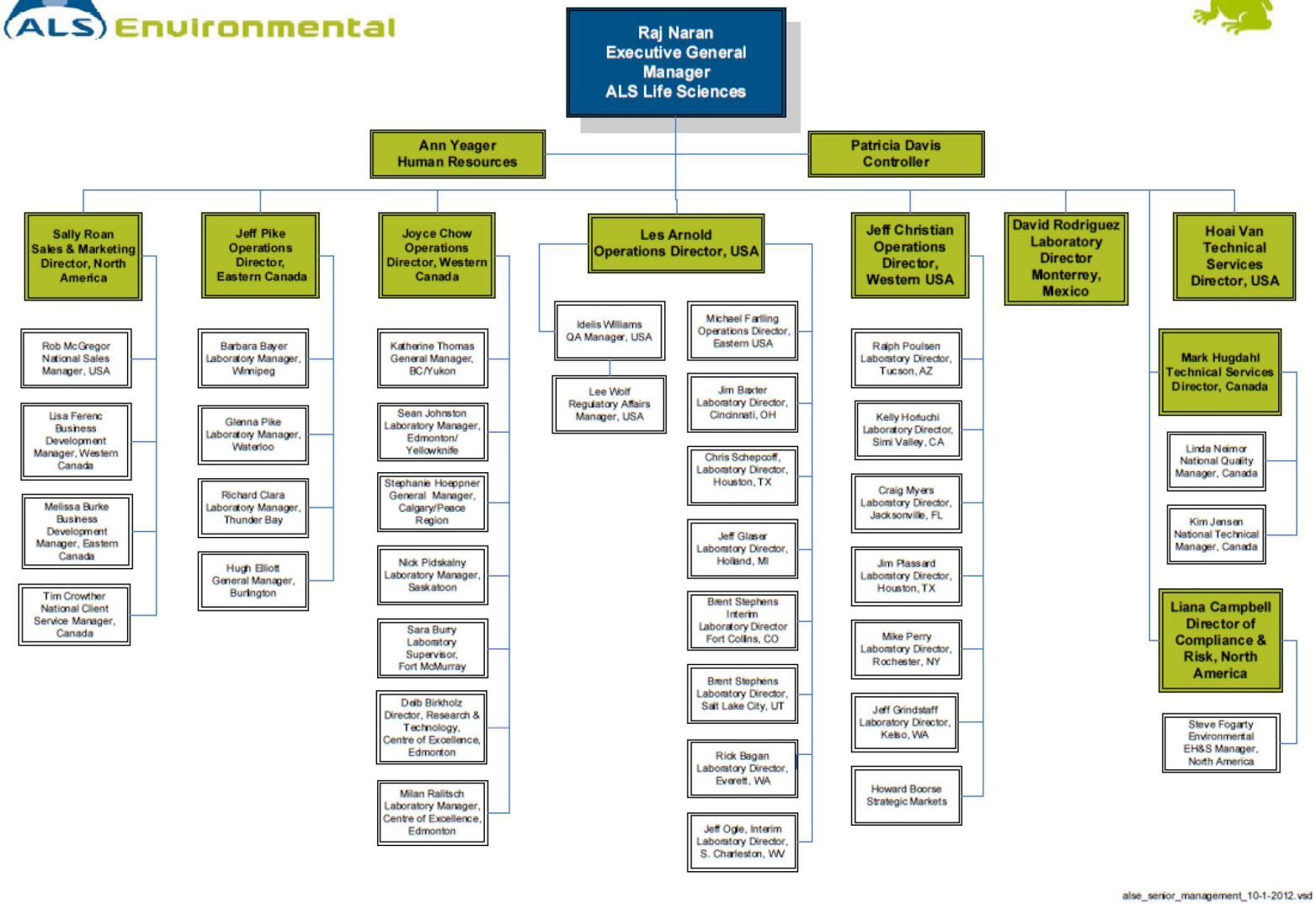

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APPENDIX C - Major Analytical Equipment

Equipment Description - Gas Chromatography	Purchased / Acquired	Location
Screen 02: Hewlett-Packard 5890 with FID/ECD Detectors	-	VOA GC/MS
Screen 03: Hewlett-Packard 5890 with FID Detector	-	VOA GC/MS
GC01: Hewlett-Packard 5890 with FID/TCD Detectors *Fixed Gas Analyzer/Total Combustion Analyzer (TCA)*	1995	VOA GC
GC03: Hewlett-Packard 5890 with ECD/FID Detectors *Hewlett-Packard 7673 Autosampler*	1995	SVOA
GC05: Hewlett-Packard 5890 Series II Combined with Sievers 355 (SCD 1)	1996	VOA GC
GC06: Hewlett-Packard 6890 with ECD/ECD Detectors *Hewlett-Packard 6890 Autosampler*	1995	SVOA
GC07: Hewlett-Packard 6890 with FID/FID Detectors	1995	VOA GC
GC08: Hewlett-Packard 5890 Series II with TCD/FID Detectors	1998	VOA GC
GC09: Hewlett-Packard 5890 Series II with FID/ECD Detectors	1999	GC/MS Screen
GC10: Hewlett-Packard 5890A with FID/TCD Detectors	1999	VOA GC
GC11: Hewlett-Packard 5890 Series II+ with FID Detector (Combined with MS01)	1999	SVOA
GC12: Hewlett-Packard 5890 Series II+ with FID Detector (Combined with MS02)	2004	SVOA
GC13: Agilent 6890A Combined with Sievers 355 (SCD 2)	2001	VOA GC
GC14: Agilent 6890N with NPD/FID Detectors *Agilent 7683B Autosampler*	2005	SVOA
GC15: Agilent 6890N with NPD/FID Detectors *Agilent 7683 Autosampler*	2005	SVOA
GC16: Agilent 6890N with PFPD Detector and *OI Detector Controller* *Agilent 7683 Autosampler*	2005	SVOA
GC19: Hewlett-Packard 5890 with FID Detector	2007	VOA GC
GC20: Agilent 7890A with FID/TCD Detectors	2008	VOA GC
GC21:Hewlett-Packard 5890 Series II with ECD/FID Detectors	2009	SVOA
GC22: Agilent 7890A Combined with Agilent 355 (SCD 3)	2009	VOA GC
GC23: Hewlett-Packard 6890+ with ECD Detector (Combined with MS14)	2007	SVOA
GC24: Hewlett-Packard 5890 Series II (Combined with MS04)	2011	VOA GC R&D
GC25: Hewlett-Packard 5890 Series II (Combined with MS12)	2006	SVOA
GC26: Agilent 7890A (Combined with MS19)	2011	VOA GC/MS
GC27: Agilent 7890A (Combined with MS20)	2011	VOA GC/MS

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Equipment Description - GC/MS Systems	Purchased / Acquired	Location
MS01: HP 5890 Series II+ with FID Detector (GC11) & HP 5971A MSD *Hewlett-Packard 7673 Autosampler*	1991	SVOA
MS02: HP 5890 Series II+ with FID Detector (GC12) & HP 5972 MSD *Hewlett-Packard 7673 Autosampler*	1994	SVOA
MS03: HP 6890A/5973 MSD *Tekmar AUTOCAN Autosampler*	1997	VOA GC/MS
MS04: HP 5890 Series II (GC24) & HP 5970 MSD	2004	VOA GC R&D
MS05: Agilent 6890+/5973N MSD *Perkin Elmer TurboMatrix ATD-50 Thermal Desorber*	1999	VOA GC/MS
MS07: HP 6890A/ Agilent 5973N MSD *Tekmar AUTOCAN Autosampler*	2001	VOA GC/MS
MS08: Agilent 6890N/5973inert MSD Tekmar AUTOCAN Autosampler	2004	VOA GC/MS
MS09: Agilent 6890N/5973inert MSD Tekmar AUTOCAN Autosampler	2005	VOA GC/MS
MS10: HP 6890A/5973 MSD Tekmar AUTOCAN Autosampler	2006	VOA GC/MS
MS11: HP 5890 Series II/5972 MSD Tekmar AUTOCAN Autosampler	2006	VOA GC/MS
MS12: HP 5890 Series II/5971 MSD HP 7673 Autosampler	2006	SVOA
MS13: Agilent 6890N/5975B inert MSD Tekmar AUTOCAN Autosampler	2006	VOA GC/MS
MS14: HP 6890+ with ECD Detector (GC23) & HP 5973 MSD HP 6890 Injector	2007	SVOA
MS15: HP 5890 Series II/5972 MSD HP 7673 Autosampler	2007	SVOA
MS16: Agilent 6890N/5975C inert MSD Tekmar AUTOCAN Autosampler	2007	VOA GC/MS
MS17: Shimadzu GCMS QP-2010 Plus	2008	SVOA
MS18: Agilent 7890A /5975C inert XL MSD Markes Series 2 Unity Thermal Desorber Markes Series 2 Ultra TD Autosampler	2010	VOA GC/MS
MS19: Agilent 7890A (GC26) & 5975C inert XL MSD Tekmar AUTOCAN Autosampler	2011	VOA GC/MS
MS20: Agilent 7890A (GC27) & /5975C inert XL MSD Markes Series 2 Unity Thermal Desorber Markes Series 2 Ultra TD Autosampler	2011	VOA GC/MS

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Liquid Chromatography	Purchased / Acquired	Location
LC03: Agilent Infinity LC 1220 (Combined with LCMS01)	2011	SVOA
LCMS01: Agilent 6120 Quadrupole MS (Combined with LC03)	2011	SVOA
Ion Chromatography	**Purchased / Acquired**	**Location**
IC02: Metrohm with Lambda 1010 830 IC Interface, 830 IC Liquid Handling Units 818 IC Pump, 820 IC Separator Center	2004	GENCHEM
IC03: Dionex ICS 2000 with Self-regenerating suppressor AS40 Autosampler	2008	GENCHEM
Spectrophotometer	**Purchased / Acquired**	**Location**
SPM01: Spectronic Instrument 20+ from SC	2001	GENCHEM
pH and Specific Ion Meters	**Purchased / Acquired**	**Location**
pH01: Thermo Orion 920 Selective Ion Meter	2001	GENCHEM
pH02: Orion 720A	1992	GENCHEM
Miscellaneous Equipment	**Purchased / Acquired**	**Location**
US Filter Water Purification System	2006	Main Lab
US Filter Water Purification System	2008	Extraction facility

Note: Purchase / Acquired year may represent when instrument was first maintained by ALS Environmental-Simi Valley or other in-network ALS Laboratory and does not reflect age of instrument.

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Air sampling containers / Flow Controllers / Critical Orifices

Six-liter Summa passivated stainless steel canisters

- 994 Ambient
- 996 Source
- 184 Standard

Six-liter Silonite passivated stainless steel canisters

- 183 Ambient
- 101 Source

Three-liter Silco passivated stainless steel canisters (78)

One-liter Summa passivated stainless steel canisters (939)

400-millilter mini passivated stainless steel canisters (19)

Low volume flow controllers for time integrated sampling

- 559 Ambient
- 97 Source

Low-flow flow controllers for multi-day sampling (59)

Mini-canister flow controllers for time integrated sampling (16)

Critical orifices (1856)

Automated Summa Canister Conditioning Units

- Twelve-position, microprocessor controlled conditioners with heater controller, vacuum gauge, humidified nitrogen fill capability and large-capacity vacuum pump (2)
- Ten-position, microprocessor controlled conditioners with heater controller, vacuum gauge, humidified nitrogen fill capability and large-capacity vacuum pump (2)
- Fourteen-position, microprocessor controlled conditioners with heater controller, vacuum gauge, humidified nitrogen fill capability and large-capacity vacuum pump (1)
- Sixteen-position, microprocessor controlled conditioner with heater controller, vacuum gauge, humidified nitrogen fill capability and large-capacity vacuum pump (2)
- Six-position, microprocessor controlled conditioner with heater controller, vacuum gauge, humidified nitrogen fill capability and large-capacity vacuum pump (1)

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APPENDIX D - Data Qualifiers and Acronyms

CODE	CATEGORY	DESCRIPTION
BC	AIHA	Reported results are not blank corrected.
BH	AIHA	Results indicate breakthrough; back section of tube greater than front section.
BT	AIHA	Results indicated possible breakthrough; back section ≥10% front section.
DE	AIHA	Reported results are corrected for desorption efficiency.
RA	AIHA	Result not available.
G	GENERAL	Improper container.
G1	GENERAL	Unpreserved or improperly preserved sample.
X	GENERAL	See case narrative.
H1	HOLD TIME	Sample analysis performed past holding time. See case narrative.
H2	HOLD TIME	Initial analysis within holding time. Reanalysis for the required dilution was past holding time.
H3	HOLD TIME	Sample was received and analyzed past holding time.
H4	HOLD TIME	Sample was extracted past required extraction holding time, but analyzed within analysis holding time. See case narrative.
i	MATRIX	The MDL/MRL has been elevated due to matrix interference.
M	MATRIX	Matrix interference; results may be biased (high/low).
M1	MATRIX	Matrix interference due to coelution with a non-target compound. (TO-15 only)
Q	PETROLEUM	The chromatographic fingerprint of the sample resembles a petroleum product, but the elution pattern indicates the presence of a greater amount of lighter/heavier molecular weight constituents than the calibration standard.
Y	PETROLEUM	The chromatogram resembles a petroleum product but does not match the calibration standard.
Z	PETROLEUM	The chromatogram does not resemble a petroleum product.
#	QC	The control limit criterion is not applicable. See case narrative.
*	QC	The result is an outlier. See case narrative.
B	QC	Analyte detected in both the sample and associated method blank.
I	QC	Internal standard not within the specified limits. See case narrative.
L	QC	Laboratory control sample recovery outside the specified limits; results may be biased (high/low).
N	QC	The matrix spike sample recovery is not within control limits. See case narrative.
R	QC	Duplicate precision not met.

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CODE	CATEGORY	DESCRIPTION
R1	QC	Duplicate precision not within the specified limits; however, the results are below the MRL and considered estimated.
S	QC	Surrogate recovery not within specified limits.
V	QC	The continuing calibration verification standard was outside (biased high/low) the specified limits for this compound.
C	RESULT	Result identification confirmed.
CE	RESULT	Co-elution.
D	RESULT	The reported result is from a dilution.
E	RESULT	Estimated; concentration exceeded calibration range.
J	RESULT	The result is an estimated concentration that is less than the MRL but greater than or equal to the MDL.
J1	RESULT	The analyte was positively identified below the method reporting limit prior to utilizing the dilution factor; the associated numerical value is considered estimated.
K	RESULT	Analyte was detected above the method reporting limit prior to normalization.
ND	RESULT	Compound was analyzed for, but not detected above the laboratory reporting/detection limit.
P	RESULT	The confirmation criterion was exceeded. The relative percent difference was greater than 40/25% between the two analytical results.
U	RESULT	Compound was analyzed for, but not detected (ND) at or above the MRL/MDL.
W	RESULT	Result quantified, but the corresponding peak was detected outside the generated retention time window.
UJ	RESULT	The analyte was not detected; however, the result is estimated due to discrepancies in meeting certain analyte-specific quality control criteria.
Ui	RESULT	The compound was analyzed for, but was not detected ("Non-detect") at or above the MRL/MDL; however, the MRL/MDL has been elevated due to a matrix interference.
T	TIC	Analyte is a tentatively identified compound, result is estimated.

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APPENDIX E - Preventive Maintenance Procedures

Instrument	Applicable Activity	Frequency	Performed
Gas Chromatographs	Replace septum	As required	In-House and Outside Vendor
	Check system for gas leaks, loose/fray wires and insulation	With cylinder change/Open system	
	Replace injection port liner	As required	
	ECD wipe test	Every 6 months	
	Thermally Clean ECD	As needed	
	Clean FID	As required	
	Change TCD assembly	As required	
	SCD – Change reaction tube	As required	
	Catalyst check	Annually	
Gas Chromatography / Mass Spectrometers	Tune MSD	As needed	In-House and Outside Vendor
	Change Semi-VOA capillary column	As needed	
	Change Semi-VOA injection port septum	As required	
	Change Semi-VOA injection port liner	As required	
	Replace trap (VOA)	As required	
	Clean ion source	As required	
	Change filament	As required	
	Change electron multiplier	As required	
	Vacuum System: • Mechanical pumps: change oil, change trap pellets (HP only) • Diffusion pump: check oil, check cooling fan, change oil • Turbo pump	• Check every 6 months, check level monthly, change at least annually or sooner is necessary • As required • Replace as required	In-House
	Air Preconcentrators / Autosampler: • Change traps • Inspect Rotors • Calibrate Mass Flow Controllers	• As required • As required • Every 6 months	In-House

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Instrument	Applicable Activity	Frequency	Performed
HPLC	Replace/clean check valve filter	As required	In-House
	Replace lamp UV/vis detector	As required	
	Replace flow cell	As required	
	Check flow	Quarterly	
Analytical Balances	Clean pan and compartment	Prior to and after use	In-House and Outside Vendor
	Check with NIST traceable weights	Prior to use	
	Field service	Annually	
Refrigerators and Freezers	Monitor Temperature	Daily	In-House
	Adjust Temperature	As required	
	Clean, Defrost	As required	
Ovens	Clean	As needed or if temperature is outside limit	In-House
pH probes	Condition probe	When fluctuations occur	In-House
	Change Filling Solution	Weekly	
Fluoride ISE	Store in storage solution	Between uses	In-House
Ammonia ISE	Store in storage solution	Between uses	In-House
UV-visible Spectrophotometer	Wavelength check	Annually	In-House
Ion Chromatographs	Change column bed supports	Monthly or as needed	In-House
	Clean column	Monthly or as needed	
	Change column	Every six months or as needed	
	Change valve port face and hex nut	Every six months or as needed	
	Clean valve slider	Every six months or as needed	
	Change tubing	Annually or as needed	
	Eluent pump	Annually	
Restek Thermal Gas Purifier	Check getter tube	Monthly, change as required	In-House

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APPENDIX F - Laboratory SOP List

Local ADM SOP Titles	SOP Code
Data and Record Archiving	ADM-ARC
Batches and Sequences	ADM-BATCH_SEQ
Handling Consumable Materials	ADM-CONSUM
Data Review and Reporting	ADM-DATA_REV
Glassware Cleaning	ADM-GLASS
Analytical Instrument Acquisition, Reassignment, Maintenance and Documentation	ADM-INSTRUM
Laboratory Storage, Analysis and Tracking	ADM-LabSAT
Media Request Fulfillment	ADM-Media_Req
Project Management	ADM-PMgmt
Software and Data Quality Assurance	ADM-SftwreQA
Calibration and Use of Laboratory Support Equipment	ADM-SupEQ
Training Documentation	ADM-TRAIN
Waste Disposal	DSP-WASTE
Cleaning and Certification of Summa Canisters and Other Specially Prepared Canisters	SMO-Can_Cert
Evaluation and Pressurization of Specially Prepared Stainless Steel Canisters	SMO-Can_Press
Flow Controllers and Critical Orifices	SMO-Flow_Cntrl
Sample Receiving, Acceptance and Log-In	SMO-SMPL_REC

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Semi-Volatile SOP Titles	SOP Code
Determination of Formaldehyde and Other Carbonyl Compounds in Ambient Air Using Adsorbent Cartridge Followed by High Performance Liquid Chromatography (HPLC) EPA Compendium Method T0-11A	SVO-11A
Determination of Polycyclic Aromatic Hydrocarbons (PAHs) in Ambient Air Using Gas Chromatography/Mass Spectrometry (GC/MS)	SVO-13A
Determination of Volatile Amines in Ambient Air Using GC/NPD	SVO-Amines
Determination of Carboxylic Acids in Ambient Air Using GC/MS	SVO-CACIDS
Analysis of Halogenated Volatile Organic Compounds in Emissions from Stationary Sources using GC/ECD in Accordance with a Modification of CARB Method 422	SVO-CARB422
NCASI Method RSC-02.02 Reduced Sulfur Compounds by Direct Injection GC/PFPD	SVO-NCASI_RSC
Determination of Organic Vapors Using GC/FID in Accordance with OSHA Method 07	SVO-OSHA_07
Preparation and Analysis of Orthorhombic Cyclooctasulfur by Gas Chromatography/Electron Capture Detector (GC/ECD)	SVO-S8_ECD
Preparation and Analysis of Orthorhombic Cyclooctasulfur by Gas Chromatography/Mass Spectrometry (GC/MS)	SVO-S8_MS
Analysis of Sulfur Hexafluoride in Accordance with a Modification of NIOSH 6602	SVO-SF6
Determination of Pesticides and Polychlorinated Biphenyls (PCBs) in Ambient Air by GC/ECD per EPA Compendium Methods TO-4 and TO-10A	SVO-TO4A
Sample and Media Preparation per EPA Compendium Method TO-13A	SVP-TO13A
Sample Extraction and Preparation of Pesticide and PCB Samples According to EPA Compendium Methods TO-4A and TO-10A	SVP-TO4A

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Volatile SOP Titles	SOP Code
Analysis of Argon Using Gas Chromatography with Thermal Conductivity Detection (TCD)	VOA-Argon
Calculating Heat Value, Compressibility Factor, and Relative Density of Gaseous Fuels in Accordance with ASTM D 3588	VOA-BTU
Dissolved Gas Analysis in Aqueous Samples Using a GC Headspace Equilibration Technique	VOA-DISGAS
Sample Preparation of Drywall for Sulfur Analysis and the Determination of Copper Corrosion	VOC-Drywall
Determination of Total Gaseous Nonmethane Organic (TGNMO) Emissions as Carbon in Landfill Gases in Accordance with EPA Method 25C	VOA-EPA25C
Determination of Methane, Carbon Monoxide, Carbon Dioxide, and Total Gaseous Nonmethane Organic (TGNMO) Emissions as Carbon in Landfill Gases According to Modified EPA Method 25C	VOA-EPA25CM
Determination of Hydrogen, Carbon Monoxide, Carbon Dioxide, Nitrogen, Methane, and Oxygen using Gas Chromatography with Thermal Conductivity Detection (TCD) in Accordance or Modification of EPA 3C or ASTM D 1946	VOA-EPA3C
Analysis of Hydrogen and Helium using Gas Chromatography with Thermal Conductivity Detection (TCD)	VOA-HHe
Analysis of Sulfur Compounds in a Gaseous Matrix by Gas Chromatography with Sulfur Chemiluminescence Detection per ASTM D 5504 and Modified SCAQMD Method 307	VOA-S307M_SCD
Analysis of Sulfur Compounds in Liquid Samples by Gas Chromatography with Sulfur Chemiluminescence Detection	VOA-SH_2O_SCD
Analysis of C1-C6+ using Gas Chromatography with Flame Ionization Detection (FID) in Accordance with a Modification of EPA Compendium Method TO-3	VOA-TO3C1C6
Analysis of Various Compounds using GC with Flame Ionization Detection (FID) in Accordance with a Modification of EPA Compendium Method TO-3	VOA-TO3MeOH
Analysis of Total Petroleum Hydrocarbons as Gasoline in Air by Gas Chromatography with Flame Ionization Detection	VOA-TPHG_TO3
Determination of Air-Phase Petroleum Hydrocarbons by Gas Chromatography/Mass Spectrometry (GC/MS)	VOA-MAPH
Determination of Volatile Organic Compounds in Air Samples Collected in Specially Prepared Canisters and Gas Collection Bags and Analyzed by Gas Chromatography/Mass Spectrometry (GC/MS)	VOA-TO15; CASS TO-15/GC-MS
Determination of Volatile Organic Compounds in Ambient Air Using Active or Passive Sampling Onto Sorbent Tubes	VOA-TO17; CASS TO-17/GC-MS

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General Chemistry (WET) SOP Titles	SOP Code
Determination of Inorganic Anions by Ion Chromatography	WET-Anions_IC
Hexavalent Chromium: Colorimetric, Liquids	WET-Cr6L
Colorimetric Determination of Hexavalent Chromium in Solids	WET-Cr6S
Hydrogen Sulfide (H_2S) in Air, Colorimetric	WET-H_2S Air
Analysis of Hexavalent Chromium by Ion Chromatography	WET-HexCr_IC
Analysis of Hydrofluoric Acid (HF) in Air by Ion Selective Electrode	WET-HFAir
Ammonia in Air by Ion Selective Electrode	WET-NH_3Air
Colorimetric Determination of Nitrogen Dioxide (NO_2) in Air	WET-NO_2Air
Ozone (O_3) in Air, Colorimetric	WET-O_3Air
pH Electrometric Measurement for Liquids by Ion Selective Electrodes	WET-pHL
pH Electrometric Measurement for Solids by Ion Selective Electrodes	WET-pHS

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APPENDIX G - Certifications, Accreditations, and Primary NELAP Accredited Methods

American Industrial Hygiene Association (AIHA)

Industrial Hygiene Laboratory Accreditation Program Laboratory
Laboratory # 101661
Approved Method(s):

- NIOSH 1450
- NIOSH 1457
- NIOSH 1500
- NIOSH 1501
- NIOSH 1550
- OSHA 07

State of Arizona, Department of Health Services

License No. AZ0694
Approved Method(s):

- EPA TO-15

State of California, Department of Health Services, (NELAP-Primary)

Certification No. 02115CA
Approved Method(s):

- EPA 7196A
- EPA 7199
- EPA 9040B
- EPA 9045C
- EPA 9056

Department of Defense, Environmental Laboratory Accreditation Program (DoD-ELAP)

Perry Johnson Laboratory Accreditation, Inc. Certificate No. L11 – 203
Approved Method(s):

- EPA TO-15
- RSK 175
- EPA 3C
- ASTM D 1946-90
- CAS SOP VOA-EPA3C (EPA 3C Modified)
- CAS SOP VOA-TPHG_TO3 (JP-4 and TPHG by Modified EPA TO-3)
- CAS SOP VOA-TO3C1C6 (Hydrocarbons and ranges by Modified EPA TO-3)
- CAS SOP VOA-TO15 (EPA TO-15 Modified)

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State of Florida, Department of Health (NELAP-Primary)
Laboratory ID No.: E871020
Approved Method(s):
- EPA TO-13A
- EPA TO-15
- EPA TO-17

State of Maine, Department of Health and Human Services
Laboratory ID: CA01527
Certificate No.: 2012039
Approved Methods
- EPA TO-15
- MADEP APH

State of Minnesota, Department of Health, Environmental Laboratory Certification Program (NELAP-Secondary)
Laboratory ID: 006-999-438
Approved Method(s):
- EPA TO-15

State of New York, Department of Health (NELAP –Secondary)
Environmental Analyses/Air and Emissions
Laboratory ID No. 11221
Approved Method(s):
- EPA TO-13A
- EPA TO-15
- EPA TO-17

State of New Jersey, Department of Environmental Protection (NELAP-Secondary)
Laboratory ID: CA009
Approved Method(s):
- EPA TO-15

State of Oregon, Environmental Laboratory Accreditation Program (NELAP-Secondary)
Laboratory ID: CA200007
Approved Method(s):
- EPA TO-15

Commonwealth of Pennsylvania, Department of Environmental Protection Bureau of Laboratories
Registration Number: 68-03307

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State of Texas, Texas Commission on Environmental Quality (NELAP-Secondary)
Certificate # T104704413-12-3
Approved Method(s):
- EPA TO-15

State of Utah, Department of Health, Environmental Laboratory Certification Program (NELAP-Secondary)
Certificate # CA015272012-2
Approved Method(s):
- EPA TO-15

State of Washington, Department of Ecology
Laboratory ID: C946
Approved Method(s):
- EPA TO-15
- EPA RSK-175

Note 1: This Quality Assurance Manual is revised annually with AIHA, DoD and NELAP-Primary Certificates, and the Scope of Accreditations/Parameters are revised annually (where necessary). During this interim period Certificates may expire and the Scope of Accreditations/Parameters may change; therefore, these may not be updated until the next revision.

Note 2: Current Certificates and Scope of Accreditations/Parameters are on file and displayed in the front hallway. Updated or Specific Certificates and Scope of Accreditations/Parameters are available upon request.

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AIHA Laboratory Accreditation Programs, LLC

acknowledges that

Columbia Analytical Services, Inc.
2655 Park Center Drive, Suite A, Simi Valley, CA 93065-6200
Laboratory ID: 101661

along with all premises from which key activities are performed, as listed above, has fulfilled the requirements of the AIHA Laboratory Accreditation Programs (AIHA-LAP), LLC accreditation to the ISO/IEC 17025:2005 international standard, *General Requirements for the Competence of Testing and Calibration Laboratories in* the following:

LABORATORY ACCREDITATION PROGRAMS

✓	**INDUSTRIAL HYGIENE**	Accreditation Expires: 11/01/2014
☐	**ENVIRONMENTAL LEAD**	Accreditation Expires:
☐	**ENVIRONMENTAL MICROBIOLOGY**	Accreditation Expires:
☐	**FOOD**	Accreditation Expires:

Specific Field(s) of Testing (FoT)/Method(s) within each Accreditation Program for which the above named laboratory maintains accreditation is outlined on the attached **Scope of Accreditation**. Continued accreditation is contingent upon successful on-going compliance with ISO/IEC 17025:2005 and AIHA-LAP, LLC requirements. This certificate is not valid without the attached **Scope of Accreditation**. Please review the AIHA-LAP, LLC website (www.aihaaccreditedlabs.org) for the most current Scope.

S. D. Allen Iske, PhD, CIH, CSP
Chairperson, Analytical Accreditation Board

Cheryl O. Morton
Managing Director, AIHA Laboratory Accreditation Programs, LLC

Revision 12: 03/29/2012

Date Issued: 08/31/2012

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AIHA Laboratory Accreditation Programs, LLC

SCOPE OF ACCREDITATION

Columbia Analytical Services, Inc.
2655 Park Center Drive, Suite A, Simi Valley, CA 93065-6200

Laboratory ID: **101661**
Issue Date: 08/31/2012

The laboratory is approved for those specific field(s) of testing/methods listed in the table below. Clients are urged to verify the laboratory's current accreditation status for the particular field(s) of testing/Methods, since these can change due to proficiency status, suspension and/or revocation. A complete listing of currently accredited Industrial Hygiene laboratories is available on the AIHA-LAP, LLC website at: http://www.aihaaccreditedlabs.org

Industrial Hygiene Laboratory Accreditation Program (IHLAP)

Initial Accreditation Date: 09/01/1994

IHLAP Scope Category	Field of Testing (FoT)	Technology sub-type/ Detector	Published Reference Method/Title of In-house Method	Method Description or Analyte *(for internal methods only)*
Chromatography Core	Gas Chromatography	GC/FID	NIOSH 1450	
			NIOSH 1457	
			NIOSH 1500	
			NIOSH 1501	
			NIOSH 1550	
			OSHA 07	
	Gas Chromatography (Diffusive Samplers)		OSHA 07	

The laboratory participates in the following AIHA-LAP, LLC-approved proficiency testing programs:

- ❑ AIHA-PAT Programs, LLC IHPAT Metals
- ✓ AIHA-PAT Programs, LLC IHPAT Organic Solvents
- ❑ AIHA-PAT Programs, LLC IHPAT Silica
- ✓ AIHA-PAT Programs, LLC IHPAT Diffusive Sampler (3M)
- ❑ AIHA-PAT Programs, LLC IHPAT Diffusive Sampler (SKC)
- ❑ AIHA-PAT Programs, LLC IHPAT Diffusive Sampler (AT)
- ❑ AIHA-PAT Programs, LLC IHPAT Asbestos
- ❑ AIHA-PAT Programs, LLC Bulk Asbestos (BAPAT)
- ❑ AIHA-PAT Programs, LLC Beryllium (BePAT)
- ❑ HSE Workplace Analytical Scheme for Proficiency (WASP) (Formaldehyde)
- ❑ HSE Workplace Analytical Scheme for Proficiency (WASP) (Thermal Desorption Tubes)
- ❑ Pharmaceutical Round Robin
- ❑ Compressed/Breathing Air Round Robin
- ❑ National Voluntary Laboratory Accreditation Program (NVLAP - determined at the time of site assessment)
- ❑ New York State Department of Health (NYS DOH – PCM and TEM)
- ❑ ERA Air and Emissions standards for indoor air quality
- ❑ Institut für Arbeitsschutz der Deutschen Gesetzlichen Unfallversicherung (IFA, formerly BGIA)
- ❑ Institut de Recherche Robert-Sauvé en Santé et en Sécurité du Travail (IRSST)

Effective: 09/28/2011
Scope_IHLAP_R6
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PJLA

PERRY JOHNSON LABORATORY ACCREDITATION, INC.

Certificate of Accreditation

Perry Johnson Laboratory Accreditation, Inc. has assessed the Laboratory of:

Columbia Analytical Services, Inc.
2655 Park Center Drive, Suite A, Simi Valley, CA 93065

(Hereinafter called the Organization) and hereby declares that Organization has met the requirements of ISO/IEC 17025:2005 "General Requirements for the competence of Testing and Calibration Laboratories" and the DoD Quality Systems Manual for Environmental Laboratories Version 4.2 11/15/2010 and is accredited is accordance with the:

United States Department of Defense
Environmental Laboratory Accreditation Program
(DoD-ELAP)

This accreditation demonstrates technical competence for the defined scope:
Environmental Testing
(As detailed in the supplement)

Accreditation claims for such testing and/or calibration services shall only be made from addresses referenced within this certificate. This Accreditation is granted subject to the system rules governing the Accreditation referred to above, and the Organization hereby covenants with the Accreditation body's duty to observe and comply with the said rules.

For PJLA:

Initial Accreditation Date:	*Issue Date:*	*Accreditation No.:*	*Certificate No.:*
January 11, 2010	December 26, 2011	65818	L11-203

Tracy Szerszen
President/Operations Manager

Perry Johnson Laboratory
Accreditation, Inc. (PJLA)
755 W. Big Beaver, Suite 1325
Troy, Michigan 48084

The validity of this certificate is maintained through ongoing assessments based on a continuous accreditation cycle. The validity of this certificate should be confirmed through the PJLA website: www.pjlabs.com

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Certificate of Accreditation: Supplement

ISO/IEC 17025:2005 and DoD-ELAP

PJLA

Columbia Analytical Services, Inc.
2655 Park Center Drive, Suite A, Simi Valley, CA 93065
Chaney Humphrey Phone: 805-526-7161

Accreditation is granted to the facility to perform the following testing:

Matrix	Standard Method	Technology	Analyte
Aqueous	RSK 175	GC/FID	Methane
Aqueous	RSK 175	GC/FID	Ethane
Aqueous	RSK 175	GC/FID	Ethene
Air	(CAS SOP) VOA-EPA3C	GC/TCD	Hydrogen
Air	(CAS SOP) VOA-EPA3C	GC/TCD	Oxygen
Air	(CAS SOP) VOA-EPA3C	GC/TCD	Nitrogen
Air	(CAS SOP) VOA-EPA3C	GC/TCD	Methane
Air	(CAS SOP) VOA-EPA3C	GC/TCD	Carbon Dioxide
Air	(CAS SOP) VOA-EPA3C	GC/TCD	Carbon Monoxide
Air	(CAS SOP) VOA-TPHG_TO3	GC/FID	Total Petroleum Hydrocarbons Gasoline (TPHG)
Air	(CAS SOP) VOA-TPHG_TO3	GC/FID	JP-4
Air	(CAS SOP) VOA-TO3C1C6	GC/FID	C1 - C6+
Air	(CAS SOP) VOA-TO3C1C6	GC/FID	Ethane
Air	(CAS SOP) VOA-TO3C1C6	GC/FID	Ethene
Air	(CAS SOP) VOA-TO3C1C6	GC/FID	Methane
Air	(CAS SOP) VOA-TO3C1C6	GC/FID	n-Butane
Air	(CAS SOP) VOA-TO3C1C6	GC/FID	n-Hexane
Air	(CAS SOP) VOA-TO3C1C6	GC/FID	n-Pentane
Air	(CAS SOP) VOA-TO3C1C6	GC/FID	Propane
Air	(CAS SOP) VOA-TO3C1C6	GC/FID	Propene
Air	(CAS SOP) VOA-TO3C1C6	GC/FID	Total Volatile Petroleum Hydrocarbons (TVPH) as Hexane
Air	EPA TO-15	GC/MS	1,1,1-Trichloroethane
Air	EPA TO-15	GC/MS	1,1,2,2-Tetrachloroethane
Air	EPA TO-15	GC/MS	1,1,2-Trichloroethane
Air	EPA TO-15	GC/MS	1,1-Dichloroethane
Air	EPA TO-15	GC/MS	1,1-Dichloroethene
Air	EPA TO-15	GC/MS	1,2,3-Trimethylbenzene
Air	EPA TO-15	GC/MS	1,2,4-Trichlorobenzene

Issue: 12/11 *This supplement is in conjunction with certificate #L11-203* *Page 2 of 8*

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Certificate of Accreditation: Supplement

ISO/IEC 17025:2005 and DoD-ELAP

PJLA

Columbia Analytical Services, Inc.
2655 Park Center Drive, Suite A, Simi Valley, CA 93065
Chaney Humphrey Phone: 805-526-7161

Accreditation is granted to the facility to perform the following testing:

Matrix	Standard Method	Technology	Analyte
Air	EPA TO-15	GC/MS	1,2,4-Trimethylbenzene
Air	EPA TO-15	GC/MS	1,2,Dibromo-3-Chloropropane
Air	EPA TO-15	GC/MS	1,2-Dibromoethane
Air	EPA TO-15	GC/MS	1,2-Dichloro-1,1,2,2-tetrafluoroethane (Freon 114)
Air	EPA TO-15	GC/MS	1,2-Dichlorobenzene
Air	EPA TO-15	GC/MS	1,2-Dichloroethane
Air	EPA TO-15	GC/MS	1,2-Dichloropropane
Air	EPA TO-15	GC/MS	1,3,5-Trimethylbenzene
Air	EPA TO-15	GC/MS	1,3-Butadiene
Air	EPA TO-15	GC/MS	1,3-Dichlorobenzene
Air	EPA TO-15	GC/MS	1,4-Dichlorobenzene
Air	EPA TO-15	GC/MS	1,4-Dioxane
Air	EPA TO-15	GC/MS	1-Butanol
Air	EPA TO-15	GC/MS	2-Butanone (MEK)
Air	EPA TO-15	GC/MS	2-Ethyltoluene
Air	EPA TO-15	GC/MS	2-Hexanone
Air	EPA TO-15	GC/MS	3-Ethyltoluene
Air	EPA TO-15	GC/MS	4-Ethyltoluene
Air	EPA TO-15	GC/MS	4-Methyl-2-Pentanone
Air	EPA TO-15	GC/MS	Acetone
Air	EPA TO-15	GC/MS	Acetonitrile
Air	EPA TO-15	GC/MS	Acrolein
Air	EPA TO-15	GC/MS	Acrylonitrile
Air	EPA TO-15	GC/MS	Allyl Chloride
Air	EPA TO-15	GC/MS	alpha-Methylstyrene
Air	EPA TO-15	GC/MS	alpha-Pinene
Air	EPA TO-15	GC/MS	Benzene
Air	EPA TO-15	GC/MS	Benzyl Chloride
Air	EPA TO-15	GC/MS	Bromodichloromethane
Air	EPA TO-15	GC/MS	Bromoform
Air	EPA TO-15	GC/MS	Bromomethane
Air	EPA TO-15	GC/MS	Carbon Disulfide
Air	EPA TO-15	GC/MS	Carbon Tetrachloride
Air	EPA TO-15	GC/MS	Chlorobenzene
Air	EPA TO-15	GC/MS	Chloroethane

Issue: 12/11 *This supplement is in conjunction with certificate #L11-203* *Page 3 of 8*

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Certificate of Accreditation: Supplement

ISO/IEC 17025:2005 and DoD-ELAP

PJLA

Columbia Analytical Services, Inc.
2655 Park Center Drive, Suite A, Simi Valley, CA 93065
Chaney Humphrey Phone: 805-526-7161

Accreditation is granted to the facility to perform the following testing:

Matrix	Standard Method	Technology	Analyte
Air	EPA TO-15	GC/MS	Chloroform
Air	EPA TO-15	GC/MS	Chloromethane
Air	EPA TO-15	GC/MS	cis-1,2-Dichloroethene
Air	EPA TO-15	GC/MS	cis-1,3-Dichloropropene
Air	EPA TO-15	GC/MS	Cumene
Air	EPA TO-15	GC/MS	Cyclohexane
Air	EPA TO-15	GC/MS	Cyclohexanone
Air	EPA TO-15	GC/MS	Dibromochloromethane
Air	EPA TO-15	GC/MS	Dichlorodifluoromethane (CFC 12)
Air	EPA TO-15	GC/MS	Diisopropyl Ether
Air	EPA TO-15	GC/MS	d-Limonene
Air	EPA TO-15	GC/MS	Ethanol
Air	EPA TO-15	GC/MS	Ethyl Acetate
Air	EPA TO-15	GC/MS	Ethyl tert-Butyl Ether
Air	EPA TO-15	GC/MS	Ethylbenzene
Air	EPA TO-15	GC/MS	Hexachlorobutadiene
Air	EPA TO-15	GC/MS	Isooctane
Air	EPA TO-15	GC/MS	Isopropyl acetate
Air	EPA TO-15	GC/MS	Isopropyl Alcohol
Air	EPA TO-15	GC/MS	m-&, p-Xylenes
Air	EPA TO-15	GC/MS	Methyl Methacrylate
Air	EPA TO-15	GC/MS	Methyl tert-Butyl Ether
Air	EPA TO-15	GC/MS	Methylene Chloride
Air	EPA TO-15	GC/MS	Naphthalene
Air	EPA TO-15	GC/MS	n-Butyl Acetate
Air	EPA TO-15	GC/MS	n-Butylbenzene
Air	EPA TO-15	GC/MS	n-Decane
Air	EPA TO-15	GC/MS	n-Dodecane
Air	EPA TO-15	GC/MS	n-Heptane
Air	EPA TO-15	GC/MS	n-Hexane
Air	EPA TO-15	GC/MS	n-Nonane

Issue: 12/11 *This supplement is in conjunction with certificate #L11-203* *Page 4 of 8*

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Certificate of Accreditation: Supplement

ISO/IEC 17025:2005 and DoD-ELAP

PJLA

Columbia Analytical Services, Inc.
2655 Park Center Drive, Suite A, Simi Valley, CA 93065
Chaney Humphrey Phone: 805-526-7161

Accreditation is granted to the facility to perform the following testing:

Matrix	Standard Method	Technology	Analyte
Air	EPA TO-15	GC/MS	n-Octane
Air	EPA TO-15	GC/MS	n-Propylbenzene
Air	EPA TO-15	GC/MS	n-Undecane
Air	EPA TO-15	GC/MS	o-Xylene
Air	EPA TO-15	GC/MS	p-Isopropyltoluene
Air	EPA TO-15	GC/MS	Propene
Air	EPA TO-15	GC/MS	sec-Butylbenzene
Air	EPA TO-15	GC/MS	Styrene
Air	EPA TO-15	GC/MS	tert-Amyl Methyl Ether
Air	EPA TO-15	GC/MS	tert-Butanol
Air	EPA TO-15	GC/MS	tert-Butylbenzene
Air	EPA TO-15	GC/MS	Tetrachloroethene
Air	EPA TO-15	GC/MS	Tetrahydrofuran
Air	EPA TO-15	GC/MS	Toluene
Air	EPA TO-15	GC/MS	trans-1,2-Dichloroethene
Air	EPA TO-15	GC/MS	trans-1,3-Dichloropropene
Air	EPA TO-15	GC/MS	Trichloroethene
Air	EPA TO-15	GC/MS	Trichlorofluoromethane
Air	EPA TO-15	GC/MS	Trichlorotrifluoroethane
Air	EPA TO-15	GC/MS	Vinyl Acetate
Air	EPA TO-15	GC/MS	Vinyl Chloride
Air	ASTM D 1946-90	GC/TCD	Hydrogen
Air	ASTM D 1946-90	GC/TCD	Oxygen
Air	ASTM D 1946-90	GC/TCD	Nitrogen
Air	ASTM D 1946-90	GC/TCD	Methane
Air	ASTM D 1946-90	GC/TCD	Carbon Dioxide
Air	ASTM D 1946-90	GC/TCD	Carbon Monoxide
Air	EPA 3C	GC/TCD	Oxygen
Air	EPA 3C	GC/TCD	Nitrogen
Air	EPA 3C	GC/TCD	Methane
Air	EPA 3C	GC/TCD	Carbon Dioxide
Air	(CAS SOP) VOA-TO15	GC/MS	1,1,1-Trichloroethane
Air	(CAS SOP) VOA-TO15	GC/MS	1,1,2,2-Tetrachloroethane
Air	(CAS SOP) VOA-TO15	GC/MS	1,1,2-Trichloroethane
Air	(CAS SOP) VOA-TO15	GC/MS	1,1-Dichloroethane

Issue: 12/11 *This supplement is in conjunction with certificate #L11-203* *Page 5 of 8*

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Certificate of Accreditation: Supplement

ISO/IEC 17025:2005 and DoD-ELAP

PJLA

Columbia Analytical Services, Inc.
2655 Park Center Drive, Suite A, Simi Valley, CA 93065
Chaney Humphrey Phone: 805-526-7161

Accreditation is granted to the facility to perform the following testing:

Matrix	Standard Method	Technology	Analyte
Air	(CAS SOP) VOA-TO15	GC/MS	1,1-Dichloroethene
Air	(CAS SOP) VOA-TO15	GC/MS	1,2,3-Trimethylbenzene
Air	(CAS SOP) VOA-TO15	GC/MS	1,2,4-Trichlorobenzene
Air	(CAS SOP) VOA-TO15	GC/MS	1,2,4-Trimethylbenzene
Air	(CAS SOP) VOA-TO15	GC/MS	1,2-Dibromo-3-Chloropropane
Air	(CAS SOP) VOA-TO15	GC/MS	1,2-Dibromoethane
Air	(CAS SOP) VOA-TO15	GC/MS	1,2-Dichloro-1,1,2,2-tetrafluoroethane (Freon 114)
Air	(CAS SOP) VOA-TO15	GC/MS	1,2-Dichlorobenzene
Air	(CAS SOP) VOA-TO15	GC/MS	1,2-Dichloroethane
Air	(CAS SOP) VOA-TO15	GC/MS	1,2-Dichloropropane
Air	(CAS SOP) VOA-TO15	GC/MS	1,3,5-Trimethylbenzene
Air	(CAS SOP) VOA-TO15	GC/MS	1,3-Butadiene
Air	(CAS SOP) VOA-TO15	GC/MS	1,3-Dichlorobenzene
Air	(CAS SOP) VOA-TO15	GC/MS	1,4-Dichlorobenzene
Air	(CAS SOP) VOA-TO15	GC/MS	1,4-Dioxane
Air	(CAS SOP) VOA-TO15	GC/MS	1-Butanol
Air	(CAS SOP) VOA-TO15	GC/MS	2-Butanone (MEK)
Air	(CAS SOP) VOA-TO15	GC/MS	2-Ethyltoluene
Air	(CAS SOP) VOA-TO15	GC/MS	2-Hexanone
Air	(CAS SOP) VOA-TO15	GC/MS	3-Ethyltoluene
Air	(CAS SOP) VOA-TO15	GC/MS	4-Ethyltoluene
Air	(CAS SOP) VOA-TO15	GC/MS	4-Methyl-2-Pentanone
Air	(CAS SOP) VOA-TO15	GC/MS	Acetone
Air	(CAS SOP) VOA-TO15	GC/MS	Acetonitrile
Air	(CAS SOP) VOA-TO15	GC/MS	Acrolein
Air	(CAS SOP) VOA-TO15	GC/MS	Acrylonitrile
Air	(CAS SOP) VOA-TO15	GC/MS	Allyl Chloride
Air	(CAS SOP) VOA-TO15	GC/MS	alpha-Methylstyrene
Air	(CAS SOP) VOA-TO15	GC/MS	alpha-Pinene
Air	(CAS SOP) VOA-TO15	GC/MS	Benzene
Air	(CAS SOP) VOA-TO15	GC/MS	Benzyl Chloride
Air	(CAS SOP) VOA-TO15	GC/MS	Bromodichloromethane
Air	(CAS SOP) VOA-TO15	GC/MS	Bromoform
Air	(CAS SOP) VOA-TO15	GC/MS	Bromomethane

Issue: 12/11 *This supplement is in conjunction with certificate #L11-203* *Page 6 of 8*

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Certificate of Accreditation: Supplement

ISO/IEC 17025:2005 and DoD-ELAP

Columbia Analytical Services, Inc.
2655 Park Center Drive, Suite A, Simi Valley, CA 93065
Chaney Humphrey Phone: 805-526-7161

Accreditation is granted to the facility to perform the following testing:

Matrix	Standard Method	Technology	Analyte
Air	(CAS SOP) VOA-TO15	GC/MS	Carbon Disulfide
Air	(CAS SOP) VOA-TO15	GC/MS	Carbon Tetrachloride
Air	(CAS SOP) VOA-TO15	GC/MS	Chlorobenzene
Air	(CAS SOP) VOA-TO15	GC/MS	Chloroethane
Air	(CAS SOP) VOA-TO15	GC/MS	Chloroform
Air	(CAS SOP) VOA-TO15	GC/MS	Chloromethane
Air	(CAS SOP) VOA-TO15	GC/MS	cis-1,2-Dichloroethene
Air	(CAS SOP) VOA-TO15	GC/MS	cis-1,3-Dichloropropene
Air	(CAS SOP) VOA-TO15	GC/MS	Cumene
Air	(CAS SOP) VOA-TO15	GC/MS	Cyclohexane
Air	(CAS SOP) VOA-TO15	GC/MS	Cyclohexanone
Air	(CAS SOP) VOA-TO15	GC/MS	Dibromochloromethane
Air	(CAS SOP) VOA-TO15	GC/MS	Dichlorodifluoromethane (CFC 12)
Air	(CAS SOP) VOA-TO15	GC/MS	Diisopropyl Ether
Air	(CAS SOP) VOA-TO15	GC/MS	d-Limonene
Air	(CAS SOP) VOA-TO15	GC/MS	Ethanol
Air	(CAS SOP) VOA-TO15	GC/MS	Ethyl Acetate
Air	(CAS SOP) VOA-TO15	GC/MS	Ethyl tert-Butyl Ether
Air	(CAS SOP) VOA-TO15	GC/MS	Ethylbenzene
Air	(CAS SOP) VOA-TO15	GC/MS	Hexachlorobutadiene
Air	(CAS SOP) VOA-TO15	GC/MS	Isooctane
Air	(CAS SOP) VOA-TO15	GC/MS	Isopropyl acetate
Air	(CAS SOP) VOA-TO15	GC/MS	Isopropyl Alcohol
Air	(CAS SOP) VOA-TO15	GC/MS	m-&,p-Xylenes
Air	(CAS SOP) VOA-TO15	GC/MS	Methyl Methacrylate
Air	(CAS SOP) VOA-TO15	GC/MS	Methyl tert-Butyl Ether
Air	(CAS SOP) VOA-TO15	GC/MS	Methylene Chloride
Air	(CAS SOP) VOA-TO15	GC/MS	Naphthalene
Air	(CAS SOP) VOA-TO15	GC/MS	n-Butyl Acetate
Air	(CAS SOP) VOA-TO15	GC/MS	n-Butylbenzene
Air	(CAS SOP) VOA-TO15	GC/MS	n-Decane
Air	(CAS SOP) VOA-TO15	GC/MS	n-Dodecane
Air	(CAS SOP) VOA-TO15	GC/MS	n-Heptane
Air	(CAS SOP) VOA-TO15	GC/MS	n-Hexane
Air	(CAS SOP) VOA-TO15	GC/MS	n-Nonane

Issue: 12/11 *This supplement is in conjunction with certificate #L11-203* *Page 7 of 8*

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Certificate of Accreditation: Supplement

ISO/IEC 17025:2005 and DoD-ELAP

PJLA

Columbia Analytical Services, Inc.
2655 Park Center Drive, Suite A, Simi Valley, CA 93065
Chaney Humphrey Phone: 805-526-7161

Accreditation is granted to the facility to perform the following testing:

Matrix	Standard Method	Technology	Analyte
Air	(CAS SOP) VOA-TO15	GC/MS	n-Octane
Air	(CAS SOP) VOA-TO15	GC/MS	n-Propylbenzene
Air	(CAS SOP) VOA-TO15	GC/MS	n-Undecane
Air	(CAS SOP) VOA-TO15	GC/MS	o-Xylene
Air	(CAS SOP) VOA-TO15	GC/MS	p-Isopropyltoluene
Air	(CAS SOP) VOA-TO15	GC/MS	Propene
Air	(CAS SOP) VOA-TO15	GC/MS	sec-Butylbenzene
Air	(CAS SOP) VOA-TO15	GC/MS	Styrene
Air	(CAS SOP) VOA-TO15	GC/MS	tert-Amyl Methyl Ether
Air	(CAS SOP) VOA-TO15	GC/MS	t-Butanol
Air	(CAS SOP) VOA-TO15	GC/MS	tert-Butylbenzene
Air	(CAS SOP) VOA-TO15	GC/MS	Tetrachloroethene
Air	(CAS SOP) VOA-TO15	GC/MS	Tetrahydrofuran
Air	(CAS SOP) VOA-TO15	GC/MS	Toluene
Air	(CAS SOP) VOA-TO15	GC/MS	trans-1,2-Dichloroethene
Air	(CAS SOP) VOA-TO15	GC/MS	trans-1,3-Dichloropropene
Air	(CAS SOP) VOA-TO15	GC/MS	Trichloroethene
Air	(CAS SOP) VOA-TO15	GC/MS	Trichlorofluoromethane
Air	(CAS SOP) VOA-TO15	GC/MS	Trichlorotrifluoroethane
Air	(CAS SOP) VOA-TO15	GC/MS	Vinyl Acetate
Air	(CAS SOP) VOA-TO15	GC/MS	Vinyl Chloride

Issue: 12/11 *This supplement is in conjunction with certificate #L11-203* *Page 8 of 8*

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FLORIDA DEPARTMENT OF HEALTH

State of Florida
Department of Health, Bureau of Laboratories
This is to certify that
E871020

COLUMBIA ANALYTICAL SERVICES, INC. - SIMI VALLEY
2655 PARK CENTER DRIVE, SUITE A
SIMI VALLEY, CA 93065

has complied with Florida Administrative Code 64E-1,
for the examination of Environmental samples in the following categories

AIR AND EMISSIONS - EXTRACTABLE ORGANICS, AIR AND EMISSIONS - VOLATILE ORGANICS

Continued certification is contingent upon successful on-going compliance with the NELAC Standards and FAC Rule 64E-1 regulations. Specific methods and analytes certified are cited on the Laboratory Scope of Accreditation for this laboratory and are on file at the Bureau of Laboratories, P. O. Box 210, Jacksonville, Florida 32231. Clients and customers are urged to verify with this agency the laboratory's certification status in Florida for particular methods and analytes.

Date Issued: July 01, 2012 Expiration Date: June 30, 2013

Max Salfinger, M.D.
Chief, Bureau of Laboratories
Florida Department of Health
DH Form 1697, 7/04
NON-TRANSFERABLE E871020-09-07/01/2012
Supersedes all previously issued certificates

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Rick Scott
Governor

FLORIDA DEPARTMENT OF HEALTH

John H. Armstrong, MD
State Surgeon General

Page 1 of 5

Laboratory Scope of Accreditation

Attachment to Certificate #: E871020-09, expiration date June 30, 2013. This listing of accredited analytes should be used only when associated with a valid certificate.

State Laboratory ID: **E871020** EPA Lab Code: **CA01527** **(805) 526-7161**

E871020
Columbia Analytical Services, Inc. - Simi Valley
2655 Park Center Drive, Suite A
Simi Valley, CA 93065

Matrix: **Air and Emissions**

Analyte	Method/Tech	Category	Certification Type	Effective Date
1,1,1-Trichloroethane	EPA TO-15	Volatile Organics	NELAP	12/27/2007
1,1,1-Trichloroethane	EPA TO-17	Volatile Organics	NELAP	7/9/2010
1,1,2,2-Tetrachloroethane	EPA TO-15	Volatile Organics	NELAP	12/27/2007
1,1,2,2-Tetrachloroethane	EPA TO-17	Volatile Organics	NELAP	7/9/2010
1,1,2-Trichloro-1,2,2-trifluoroethane (Freon 113)	EPA TO-15	Volatile Organics	NELAP	12/27/2007
1,1,2-Trichloro-1,2,2-trifluoroethane (Freon 113)	EPA TO-17	Volatile Organics	NELAP	7/9/2010
1,1,2-Trichloroethane	EPA TO-15	Volatile Organics	NELAP	12/27/2007
1,1,2-Trichloroethane	EPA TO-17	Volatile Organics	NELAP	7/9/2010
1,1-Dichloroethane	EPA TO-15	Volatile Organics	NELAP	12/27/2007
1,1-Dichloroethane	EPA TO-17	Volatile Organics	NELAP	7/9/2010
1,1-Dichloroethylene	EPA TO-15	Volatile Organics	NELAP	12/27/2007
1,1-Dichloroethylene	EPA TO-17	Volatile Organics	NELAP	7/9/2010
1,2,3-Trimethylbenzene	CASS TO-15/GC-MS	Volatile Organics	NELAP	7/9/2010
1,2,4-Trichlorobenzene	EPA TO-15	Volatile Organics	NELAP	12/27/2007
1,2,4-Trichlorobenzene	EPA TO-17	Volatile Organics	NELAP	7/9/2010
1,2,4-Trimethylbenzene	EPA TO-15	Volatile Organics	NELAP	12/27/2007
1,2,4-Trimethylbenzene	EPA TO-17	Volatile Organics	NELAP	7/9/2010
1,2-Dibromo-3-chloropropane (DBCP)	EPA TO-15	Volatile Organics	NELAP	12/27/2007
1,2-Dibromo-3-chloropropane (DBCP)	EPA TO-17	Volatile Organics	NELAP	7/9/2010
1,2-Dibromoethane (EDB, Ethylene dibromide)	EPA TO-15	Volatile Organics	NELAP	12/27/2007
1,2-Dibromoethane (EDB, Ethylene dibromide)	EPA TO-17	Volatile Organics	NELAP	7/9/2010
1,2-Dichloro-1,1,2,2-tetrafluoroethane	EPA TO-15	Volatile Organics	NELAP	12/27/2007
1,2-Dichloro-1,1,2,2-tetrafluoroethane	EPA TO-17	Volatile Organics	NELAP	7/9/2010
1,2-Dichlorobenzene	EPA TO-15	Volatile Organics	NELAP	12/27/2007
1,2-Dichlorobenzene	EPA TO-17	Volatile Organics	NELAP	7/9/2010
1,2-Dichloroethane	EPA TO-15	Volatile Organics	NELAP	12/27/2007
1,2-Dichloroethane	EPA TO-17	Volatile Organics	NELAP	7/9/2010
1,2-Dichloropropane	EPA TO-15	Volatile Organics	NELAP	12/27/2007
1,2-Dichloropropane	EPA TO-17	Volatile Organics	NELAP	7/9/2010
1,3,5-Trimethylbenzene	EPA TO-15	Volatile Organics	NELAP	12/27/2007
1,3,5-Trimethylbenzene	EPA TO-17	Volatile Organics	NELAP	7/9/2010
1,3-Butadiene	EPA TO-15	Volatile Organics	NELAP	12/27/2007
1,3-Butadiene	EPA TO-17	Volatile Organics	NELAP	7/9/2010
1,3-Dichlorobenzene	EPA TO-15	Volatile Organics	NELAP	12/27/2007
1,3-Dichlorobenzene	EPA TO-17	Volatile Organics	NELAP	7/9/2010
1,4-Dichlorobenzene	EPA TO-15	Volatile Organics	NELAP	12/27/2007

Clients and Customers are urged to verify the laboratory's current certification status with the Environmental Laboratory Certification Program. **Issue Date: 7/1/2012** **Expiration Date: 6/30/2013**

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Rick Scott
Governor

FLORIDA DEPARTMENT OF HEALTH

John H. Armstrong, MD
State Surgeon General

Laboratory Scope of Accreditation

Page 2 of 5

Attachment to Certificate #: E871020-09, expiration date June 30, 2013. This listing of accredited analytes should be used only when associated with a valid certificate.

State Laboratory ID: **E871020** EPA Lab Code: **CA01527** **(805) 526-7161**

E871020
Columbia Analytical Services, Inc. - Simi Valley
2655 Park Center Drive, Suite A
Simi Valley, CA 93065

Matrix: **Air and Emissions**

Analyte	Method/Tech	Category	Certification Type	Effective Date
1,4-Dichlorobenzene	EPA TO-17	Volatile Organics	NELAP	7/9/2010
1,4-Dioxane (1,4-Diethyleneoxide)	EPA TO-15	Volatile Organics	NELAP	12/27/2007
1,4-Dioxane (1,4-Diethyleneoxide)	EPA TO-17	Volatile Organics	NELAP	7/9/2010
2,2,4-Trimethylpentane	EPA TO-15	Volatile Organics	NELAP	7/9/2010
2,2,4-Trimethylpentane	EPA TO-17	Volatile Organics	NELAP	7/9/2010
2-Butanone (Methyl ethyl ketone, MEK)	EPA TO-15	Volatile Organics	NELAP	12/27/2007
2-Butanone (Methyl ethyl ketone, MEK)	EPA TO-17	Volatile Organics	NELAP	7/9/2010
2-Hexanone	CASS TO-15/GC-MS	Volatile Organics	NELAP	12/27/2007
2-Hexanone	EPA TO-17	Volatile Organics	NELAP	7/9/2010
4-Ethyltoluene	CASS TO-15/GC-MS	Volatile Organics	NELAP	12/27/2007
4-Methyl-2-pentanone (MIBK)	EPA TO-15	Volatile Organics	NELAP	12/27/2007
4-Methyl-2-pentanone (MIBK)	EPA TO-17	Volatile Organics	NELAP	7/9/2010
Acenaphthene	EPA TO-13A	Extractable Organics	NELAP	12/27/2007
Acenaphthylene	EPA TO-13A	Extractable Organics	NELAP	12/27/2007
Acetone	CASS TO-15/GC-MS	Volatile Organics	NELAP	12/27/2007
Acetone	EPA TO-17	Volatile Organics	NELAP	7/9/2010
Acetonitrile	EPA TO-15	Volatile Organics	NELAP	12/27/2007
Acetonitrile	EPA TO-17	Volatile Organics	NELAP	7/9/2010
Acrolein (Propenal)	EPA TO-15	Volatile Organics	NELAP	12/27/2007
Acrylonitrile	EPA TO-15	Volatile Organics	NELAP	12/27/2007
Allyl chloride (3-Chloropropene)	EPA TO-15	Volatile Organics	NELAP	12/27/2007
alpha-Methylstyrene	CASS TO-15/GC-MS	Volatile Organics	NELAP	7/9/2010
Anthracene	EPA TO-13A	Extractable Organics	NELAP	12/27/2007
a-Pinene	CASS TO-15/GC-MS	Volatile Organics	NELAP	12/27/2007
Benzene	EPA TO-15	Volatile Organics	NELAP	12/27/2007
Benzene	EPA TO-17	Volatile Organics	NELAP	7/9/2010
Benzo(a)anthracene	EPA TO-13A	Extractable Organics	NELAP	12/27/2007
Benzo(a)pyrene	EPA TO-13A	Extractable Organics	NELAP	12/27/2007
Benzo(b)fluoranthene	EPA TO-13A	Extractable Organics	NELAP	12/27/2007
Benzo(g,h,i)perylene	EPA TO-13A	Extractable Organics	NELAP	12/27/2007
Benzo(k)fluoranthene	EPA TO-13A	Extractable Organics	NELAP	12/27/2007
Benzyl chloride	EPA TO-15	Volatile Organics	NELAP	12/27/2007
Bromodichloromethane	CASS TO-15/GC-MS	Volatile Organics	NELAP	12/27/2007
Bromodichloromethane	EPA TO-17	Volatile Organics	NELAP	7/9/2010
Bromoform	EPA TO-15	Volatile Organics	NELAP	12/27/2007
Bromoform	EPA TO-17	Volatile Organics	NELAP	7/9/2010

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Laboratory Scope of Accreditation

Attachment to Certificate #: E871020-09, expiration date June 30, 2013. This listing of accredited analytes should be used only when associated with a valid certificate.

State Laboratory ID: **E871020** EPA Lab Code: **CA01527** **(805) 526-7161**

E871020
Columbia Analytical Services, Inc. - Simi Valley
2655 Park Center Drive, Suite A
Simi Valley, CA 93065

Matrix: **Air and Emissions**

Analyte	Method/Tech	Category	Certification Type	Effective Date
Carbon disulfide	EPA TO-15	Volatile Organics	NELAP	12/27/2007
Carbon disulfide	EPA TO-17	Volatile Organics	NELAP	7/9/2010
Carbon tetrachloride	EPA TO-15	Volatile Organics	NELAP	12/27/2007
Carbon tetrachloride	EPA TO-17	Volatile Organics	NELAP	7/9/2010
Chlorobenzene	EPA TO-15	Volatile Organics	NELAP	12/27/2007
Chlorobenzene	EPA TO-17	Volatile Organics	NELAP	7/9/2010
Chloroethane	EPA TO-15	Volatile Organics	NELAP	12/27/2007
Chloroethane	EPA TO-17	Volatile Organics	NELAP	7/9/2010
Chloroform	EPA TO-15	Volatile Organics	NELAP	12/27/2007
Chloroform	EPA TO-17	Volatile Organics	NELAP	7/9/2010
Chrysene	EPA TO-13A	Extractable Organics	NELAP	12/27/2007
cis-1,2-Dichloroethylene	EPA TO-15	Volatile Organics	NELAP	12/27/2007
cis-1,2-Dichloroethylene	EPA TO-17	Volatile Organics	NELAP	7/9/2010
cis-1,3-Dichloropropene	EPA TO-15	Volatile Organics	NELAP	12/27/2007
cis-1,3-Dichloropropene	EPA TO-17	Volatile Organics	NELAP	7/9/2010
Cyclohexane	CASS TO-15/GC-MS	Volatile Organics	NELAP	7/9/2010
Cyclohexane	EPA TO-17	Volatile Organics	NELAP	7/9/2010
Cyclohexanone	CASS TO-15/GC-MS	Volatile Organics	NELAP	7/9/2010
Dibenz(a,h)anthracene	EPA TO-13A	Extractable Organics	NELAP	12/27/2007
Dibromochloromethane	CASS TO-15/GC-MS	Volatile Organics	NELAP	12/27/2007
Dibromochloromethane	EPA TO-17	Volatile Organics	NELAP	7/9/2010
Dichlorodifluoromethane	EPA TO-15	Volatile Organics	NELAP	12/27/2007
Dichlorodifluoromethane	EPA TO-17	Volatile Organics	NELAP	7/9/2010
Di-isopropylether (DIPE)	CASS TO-15/GC-MS	Volatile Organics	NELAP	7/9/2010
D-Limonene	CASS TO-15/GC-MS	Volatile Organics	NELAP	12/27/2007
Ethanol	CASS TO-15/GC-MS	Volatile Organics	NELAP	12/27/2007
Ethanol	EPA TO-17	Volatile Organics	NELAP	7/9/2010
Ethyl acetate	CASS TO-15/GC-MS	Volatile Organics	NELAP	7/9/2010
Ethylbenzene	EPA TO-15	Volatile Organics	NELAP	12/27/2007
Ethylbenzene	EPA TO-17	Volatile Organics	NELAP	7/9/2010
Ethyl-t-butylether (ETBE)	CASS TO-15/GC-MS	Volatile Organics	NELAP	7/9/2010
Fluoranthene	EPA TO-13A	Extractable Organics	NELAP	12/27/2007
Fluorene	EPA TO-13A	Extractable Organics	NELAP	12/27/2007
Hexachlorobutadiene	EPA TO-15	Volatile Organics	NELAP	12/27/2007
Hexachlorobutadiene	EPA TO-17	Volatile Organics	NELAP	7/9/2010
Indeno(1,2,3-cd)pyrene	EPA TO-13A	Extractable Organics	NELAP	12/27/2007

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Laboratory Scope of Accreditation

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State Laboratory ID: **E871020** EPA Lab Code: **CA01527** **(805) 526-7161**

E871020
Columbia Analytical Services, Inc. - Simi Valley
2655 Park Center Drive, Suite A
Simi Valley, CA 93065

Matrix: **Air and Emissions**

Analyte	Method/Tech	Category	Certification Type	Effective Date
Isopropanol	CASS TO-15/GC-MS	Volatile Organics	NELAP	12/27/2007
Isopropyl acetate	CASS TO-15/GC-MS	Volatile Organics	NELAP	7/9/2010
Isopropyl alcohol (2-Propanol)	EPA TO-17	Volatile Organics	NELAP	7/9/2010
Isopropylbenzene	EPA TO-15	Volatile Organics	NELAP	12/27/2007
Isopropylbenzene	EPA TO-17	Volatile Organics	NELAP	7/9/2010
Methyl bromide (Bromomethane)	EPA TO-15	Volatile Organics	NELAP	12/27/2007
Methyl chloride (Chloromethane)	EPA TO-15	Volatile Organics	NELAP	12/27/2007
Methyl chloride (Chloromethane)	EPA TO-17	Volatile Organics	NELAP	7/9/2010
Methyl methacrylate	EPA TO-15	Volatile Organics	NELAP	12/27/2007
Methyl tert-butyl ether (MTBE)	EPA TO-15	Volatile Organics	NELAP	12/27/2007
Methyl tert-butyl ether (MTBE)	EPA TO-17	Volatile Organics	NELAP	7/9/2010
Methylene chloride	EPA TO-15	Volatile Organics	NELAP	12/27/2007
Methylene chloride	EPA TO-17	Volatile Organics	NELAP	7/9/2010
m-Ethyltoluene	CASS TO-15/GC-MS	Volatile Organics	NELAP	7/9/2010
Naphthalene	CASS TO-15/GC-MS	Volatile Organics	NELAP	12/27/2007
Naphthalene	EPA TO-13A	Extractable Organics	NELAP	12/27/2007
Naphthalene	EPA TO-17	Volatile Organics	NELAP	7/9/2010
n-Butyl acetate	CASS TO-15/GC-MS	Volatile Organics	NELAP	12/27/2007
n-Butyl alcohol	CASS TO-15/GC-MS	Volatile Organics	NELAP	7/9/2010
n-Butylbenzene	CASS TO-15/GC-MS	Volatile Organics	NELAP	7/9/2010
n-Decane	CASS TO-15/GC-MS	Volatile Organics	NELAP	7/9/2010
n-Dodecane	CASS TO-15/GC-MS	Volatile Organics	NELAP	7/9/2010
n-Heptane	EPA TO-15	Volatile Organics	NELAP	7/9/2010
n-Heptane	EPA TO-17	Volatile Organics	NELAP	7/9/2010
n-Hexane	EPA TO-15	Volatile Organics	NELAP	12/27/2007
n-Hexane	EPA TO-17	Volatile Organics	NELAP	7/9/2010
n-Nonane	CASS TO-15/GC-MS	Volatile Organics	NELAP	12/27/2007
n-Octane	CASS TO-15/GC-MS	Volatile Organics	NELAP	7/9/2010
n-Octane	EPA TO-17	Volatile Organics	NELAP	7/9/2010
n-Propylbenzene	CASS TO-15/GC-MS	Volatile Organics	NELAP	7/9/2010
n-Undecane	CASS TO-15/GC-MS	Volatile Organics	NELAP	7/9/2010
o-Ethyltoluene	CASS TO-15/GC-MS	Volatile Organics	NELAP	7/9/2010
Phenanthrene	EPA TO-13A	Extractable Organics	NELAP	12/27/2007
p-Isopropyltoluene	CASS TO-15/GC-MS	Volatile Organics	NELAP	7/9/2010
Propylene	CASS TO-15/GC-MS	Volatile Organics	NELAP	7/9/2010
Pyrene	EPA TO-13A	Extractable Organics	NELAP	12/27/2007

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State Laboratory ID: **E871020** EPA Lab Code: **CA01527** **(805) 526-7161**

E871020
Columbia Analytical Services, Inc. - Simi Valley
2655 Park Center Drive, Suite A
Simi Valley, CA 93065

Matrix: **Air and Emissions**

Analyte	Method/Tech	Category	Certification Type	Effective Date
sec-Butylbenzene	CASS TO-15/GC-MS	Volatile Organics	NELAP	7/9/2010
Styrene	EPA TO-15	Volatile Organics	NELAP	12/27/2007
Styrene	EPA TO-17	Volatile Organics	NELAP	7/9/2010
T-amylmethylether (TAME)	CASS TO-15/GC-MS	Volatile Organics	NELAP	7/9/2010
tert-Butyl alcohol	CASS TO-15/GC-MS	Volatile Organics	NELAP	7/9/2010
tert-Butylbenzene	CASS TO-15/GC-MS	Volatile Organics	NELAP	7/9/2010
Tetrachloroethylene (Perchloroethylene)	EPA TO-15	Volatile Organics	NELAP	12/27/2007
Tetrachloroethylene (Perchloroethylene)	EPA TO-17	Volatile Organics	NELAP	7/9/2010
Tetrahydrofuran (THF)	CASS TO-15/GC-MS	Volatile Organics	NELAP	7/9/2010
Tetrahydrofuran (THF)	EPA TO-17	Volatile Organics	NELAP	7/9/2010
Toluene	EPA TO-15	Volatile Organics	NELAP	12/27/2007
Toluene	EPA TO-17	Volatile Organics	NELAP	7/9/2010
trans-1,2-Dichloroethylene	EPA TO-15	Volatile Organics	NELAP	12/27/2007
trans-1,2-Dichloroethylene	EPA TO-17	Volatile Organics	NELAP	7/9/2010
trans-1,3-Dichloropropene	EPA TO-15	Volatile Organics	NELAP	12/27/2007
trans-1,3-Dichloropropene	EPA TO-17	Volatile Organics	NELAP	7/9/2010
Trichloroethene (Trichloroethylene)	EPA TO-15	Volatile Organics	NELAP	12/27/2007
Trichloroethene (Trichloroethylene)	EPA TO-17	Volatile Organics	NELAP	7/9/2010
Trichlorofluoromethane	EPA TO-15	Volatile Organics	NELAP	12/27/2007
Trichlorofluoromethane	EPA TO-17	Volatile Organics	NELAP	7/9/2010
Vinyl acetate	EPA TO-15	Volatile Organics	NELAP	12/27/2007
Vinyl chloride	EPA TO-15	Volatile Organics	NELAP	12/27/2007
Vinyl chloride	EPA TO-17	Volatile Organics	NELAP	7/9/2010
Xylene (total)	EPA TO-15	Volatile Organics	NELAP	12/27/2007
Xylene (total)	EPA TO-17	Volatile Organics	NELAP	7/9/2010

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APPENDIX C

EXAMPLE FIELD DATA RECORDS

AIR SAMPLING FIELD DATA RECORD

AIR SAMPLING FIELD DATA RECORD

Project Name: CTS of Asheville, Inc. **Project Numer:** _____

Sampling Personnel: _____ **Sample ID:** _____

Sample Address: _____ **Sample Location*:** _____

Canister ID: _____ **Flow Controller ID:** _____

	Start	Stop
Sample Date:	_____	_____
Sample Time:	_____	_____
Canister Pressure:**	_____	_____
Outdoor Temperature:**	_____	_____
Interior Temperature:**	_____	_____
PID Reading (ppm):	_____	_____
Wind Direction:	_____	_____

Antecedent weather conditions:

_____

Weather conditions during sample period:

_____

Sketch of sampling area:

* Indicate crawlspace or ambient and approximate height of air intake.

** Indicate unit of measurement.

PHOTOGRAPH RECORD

PHOTOGRAPH RECORD

Project Name: CTS of Asheville, Inc. Superfund Site **Project Number:** _____

Date/Time: _____ **Taken By:** _____

File Name	Direction of Photo	Description

APPENDIX D

ALS ENVIRONMENTAL
STANDARD OPERATING PROCEDURE FOR CLEANING AND CERTIFICATION OF SUMMA CANISTERS AND OTHER SPECIALLY-PREPARED CANISTERS

DOCUMENT TITLE:	*CLEANING AND CERTIFICATION OF SUMMA CANISTERS AND OTHER SPECIALLY PREPARED CANISTERS*
REFERENCED METHOD:	*N/A*
SOP ID:	*SMO-CAN_CERT*
REV. NUMBER:	*14.0*
EFFECTIVE DATE:	*01/12/2013*

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CLEANING AND CERTIFICATION OF SUMMA CANISTERS AND OTHER SPECIALLY PREPARED CANISTERS

SOPID:	SMO-Can_Cert	Rev. Number:	14.0	Effective Date:	01/12/2013

Approved By: _____ Date: 12/21/12
Team Leader (Canister Conditioning and Shipping) - Karen Ryan

Approved By: _____ Date: 12/20/12
Operations Manager - Chris Parnell

Approved By: _____ Date: 12/20/12
QA Manager - Chaney Humphrey

Approved By: _____ Date: 12/31/12
Laboratory Director - Kelly Horiuchi

Archival Date: _____ Doc Control ID#: Non-Controlled Editor: _____

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CLEANING AND CERTIFICATION OF SUMMA CANISTERS AND OTHER SPECIALLY PREPARED CANISTERS

1) Scope and Applicability

1.1 This document describes the procedures required to prepare all passivated stainless steel canisters for sample collection and use within the laboratory. It outlines the steps necessary to internally clean the canisters (all volumes) and document their acceptability for re-use. Also included are procedures for leak checking, evaluation of the condition of canisters, canister repair, and maintenance of the cleaning apparatus.

Canister cleaning and certification is needed to ensure that each sampling container is free of contaminants and leaks that could jeopardize the integrity of the analytical data. The process must uniformly and consistently render each canister clean enough to meet the criteria for all of the relevant test methods.

2) Summary of Procedure

2.1 After all analyses are complete on the canister sample in question, the analyst records the identification and concentration level of the most prominent compounds found in the sample on the sample identification tag. This includes not only target compounds from the analytical methods, but tentatively identified compounds and any anomalous observations. Only after the corresponding data is reviewed and approved including verification that all reporting and QA/QC requirements are met are the canisters released for cleaning and certification.

First, the canisters are pre-purged to remove the bulk of the sample from each canister. Then they are placed on a cleaning manifold (the manifold may be located inside an oven), canisters not placed in the oven shall be fitted with heating bands at 120˚C to 140ºC. All canisters connected to the cleaning manifolds shall be evacuated to under 100 millitorr. The canisters are then serially filled and evacuated with humidified nitrogen. Following the cleaning process, a minimum of one canister per batch (10 to 16 canisters) is selected as the quality control (QC) canister. The canister(s) is analyzed by EPA Method TO-15 in accordance with the *SOP for Determination of Volatile Organic Compounds in Air Samples Collected in Specially Prepared Canisters and Gas Collection Bags by Gas Chromatography/Mass Spectrometry (GC/MS).*

Following the cleaning procedure, the canisters are pressurized to approximately 15 psig and held for a minimum of 24 hours to determine if any leaks exist. The pressure of each canister is evaluated using a calibrated pressure/vacuum gauge and the results recorded. All canisters that pass both the batch analyte concentration QC and individual leak check are certified and ready for use.

3) Definitions

3.1 Summa Canister A stainless steel air sampling canister whose inner surface has been electropolished by the proprietary "Summa" process to provide chemical inertness.

3.2 psig pounds per square inch gauge

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3.3 GC/MS Gas Chromatography/Mass Spectrometry

3.4 ppbv parts per billion, volume

3.5 ppmv parts per million, volume

3.6 Pressurized Air Zero grade with <0.1ppmv of total hydrocarbons

3.7 QC Canister This is a quality control canister selected and analyzed in order for the cleaning batch to be certified as clean. There may be more than one canister selected within a cleaning batch to serve as a QC canister and this is dependant on a number of factors each of which are included in this standard operating procedure.

3.8 Standard Canister This is a canister labeled for containing standards; they are segregated from source and ambient canisters and are not to be sent to clients. Standard canisters may only be cleaned with other standard canisters on the manifolds. The most contaminated canister is analyzed in order to determine if the cleaning batch is to be certified as clean. There may be more than one canister selected within a cleaning batch to serve as a QC canister and this is dependant on a number of factors each of which are included in this standard operating procedure.

4) Responsibilities

4.1 It is the responsibility of all canister cleaning department technicians to accurately perform the procedures of this SOP and to complete applicable training documentation. Documented training plans must be completed by the trainee and trainer before submitting to the Quality Assurance department for final approval. This paperwork will be maintained by the QA department in an employee training file.

5) Interferences

5.1 Not Applicable

6) Safety

6.1 Burns

Caution must be exercised when working around the canister cleaning system because of the potential for burns from the canister heating bands and the manifold tubing heating coil as these temperatures are often near 100˚C. The oil from the vacuum pumps is also hot enough to cause burns when operating at normal system temperatures. The operator should wait for the oil to cool prior to attempting to change the pump oil.

6.2 Contaminated Pump Oil

Besides the burn hazard, contact with used oil from the vacuum pumps should be avoided since it becomes contaminated during normal use with toxic organic residues from the summa canisters. Proper attire must be worn when working with used pump

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oil that must be disposed of as hazardous waste in accordance with the *SOP for Waste Disposal* and applicable regulations.

6.3 Electrical Shock

All electrical wiring should be periodically checked for insulation cuts and chafing as electrical shock can occur from exposed wires. If an exposed metal wire is discovered, it must be repaired immediately.

6.4 Fires

All flammable materials, especially solvents, shall be kept clear of the cleaning manifold and pumps since the canister heaters generate considerable heat. Also, all plugs must be securely seated in their receptacles.

6.5 Hydrochloric acid (HCl)

When Hydrochloric acid is used to prepare acidified water for manual cleaning, special care must be taken. Always wear protective eyewear and gloves when handling. Prepare mixture in a hood and keep tightly capped when transporting. Hydrochloric acid is a severe health and contact hazard.

7) Sample Collection, Containers, Preservation, and Storage

7.1 There are no requirements for sample collection in this document. The following are a list of canisters that may be cleaned, certified and maintained by this procedure. This list is not intended to be all-inclusive as it contains only the most requested and current canister types.

- Six-liter Summa passivated stainless steel canisters
- Six-liter Silco passivated stainless steel canisters
- Three-liter Silco passivated stainless steel canisters
- One-liter Summa passivated stainless steel canisters
- 400-millilter mini passivated stainless steel canisters

All canisters must be stored on the clearly designated portable shelves with the caps securely placed on the valves (unless the canister is being leak checked).

8) Apparatus and Equipment

8.1 The number and capacity of the pre-purge and cleaning manifolds may be increased or expanded depending on necessity as long as they are clearly identified. As long as the minimum requirements for identification, cleaning, certification and maintenance are met, additional manifolds may be added at any point during the effective dates of this document. Additionally, the equipment listed in this section may be modified as long as the performance of the equipment selected is at least equivalent and this may include the size of heating bands, pump horsepower, etc.

8.2 Pre-purge System

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Each pre-purge manifold system consists of a vacuum pump and compressed nitrogen gas supply tube connected to a ten position manifold. The manifold is made of 1/4" 316 stainless steel and Teflon tubing with stainless steel or brass fittings. Pressure is read with an in-line pressure gauge. The canister valve fitting is attached to the manifold using a brass Swagelok nut and ceramic-filled Teflon ferrule.

The purge cycles are controlled automatically with a three-way solenoid valve; one position connects to the nitrogen source, the second position connects to the pump, and the third position connects to the manifold. The canisters are pre-cleaned by serial evacuation and dilution with nitrogen. The solenoid valve is connected to a relay timer that can be programmed with the desired evacuation and fill time intervals and number of cycles. Programming is done using the keypad on the controller. The instruction for programming and starting the controller are found at the canister purge station.

8.3 Canister Cleaning System

The canister cleaning system is comprised of cleaning manifolds, nitrogen source with humidifiers, high vacuum pumps, digital vacuum/pressure gauges (in millitorr), electric heating bands/ovens, and electronic control units each described below.

8.3.1 Canister Manifolds

Currently, there are six cleaning manifolds. The cleaning manifolds are split into three groupings with the sample type or size serving as the source of the manifold designation for cleaning.

Cleaning Manifolds #1 and 2	(10) Source Canisters Each
Cleaning Manifold #3	(14) Ambient Canisters
Cleaning Manifold #4 (oven)	(15 total) 1L Canisters
Cleaning Manifold #5	(16) Ambient Canisters
Cleaning Oven #6	(6) Ambient/Source Canisters
Cleaning Oven #7 and 8	(12) Source Canisters Combined

The manifolds are constructed of 1/2" 316 stainless steel tubing and fittings, except for manifold 4 which uses ¼" stainless steel tubing. Canisters are attached to the manifold with a Swagelok brass nut and ceramic-filled Teflon ferrule or canisters are attached to the oven manifold with a Swagelok brass nut or Swagelok stainless steel nut with aluminum finger-tight body and graphite vespel ferrule. The proper fittings reduce the chance of damaging the stainless steel canister valve. Each manifold has its own pump and vacuum gauge, but shares a common gas source.

8.3.2 Gas Source with Hydrocarbon Trap and Humidifier

The manifolds use the gas from the auxiliary vapor outlet on the bulk liquid nitrogen tank. It is plumbed into the building with copper pipe, filtered through a hydrocarbon trap (if necessary), reduced in pressure, humidified, and connected to the gas inlet valves. Tubing is ¼" copper, stainless steel, or PFA Teflon. The fittings, humidifier, and single-stage regulator are 304 or 316 stainless steel. The vapor from the bulk tank ranges from 50 to 80psig, and is dropped to between 10 and 15psig before entering the humidifier via a

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regulator. The hydrocarbon trap may not be needed since this gas is typically ultra-clean.

8.3.3 Vacuum Pumps

The vacuum pumps are dual-stage direct driven rotary-vane type, made by Alcatel Co. and Edwards Mfg. Co. Each is rated at 285 liters per minute and is capable of achieving a vacuum of <10mtorr. An activated alumina filled foretrap is used to protect vacuum pump oil from system impurities and to prevent oil vapors from backstreaming into the canisters. The exhaust port of each pump is vented to the hood with Tygon tubing.

Manifold #5 and the Entech 3100 systems for manifolds 4a and 4b, 6, 7, and 8 use a two-stage oil-less pumping system comprised of a molecular drag pump backed by a Teflon diaphragm roughing pump. The rough pump evacuates the system to less than 2psig before activating the drag pump. The system on manifold #5 is a DriVac model BH200 with integrated vacuum controller. The Entech systems use a Vacuubrand roughing pump and an Alcatel MDP 5011 drag pump combination.

8.3.4 Conditioner Vacuum Gauges

The gauges used to read the system pressure on each manifold are the Pirani digital type, capable of measuring between 0 and 2000 millitorr.

8.3.5 Electric Ovens and Heating Bands

During the cleaning process, each canister is heated to 120˚C to 140˚C using an electric oven or heating band. The bands are made of fiberglass-reinforced silicone rubber, and range in size depending on the canisters to be cleaned. They are rated at 5 watts per square inch, and operate on 120 VAC.

8.3.6 Controller Unit

The canister cleaning manifolds are controlled through a remote I/O board and a custom designed PC-based software program. All electronic components of the system (except the power leads of the heating bands) are connected to an I/O board including solenoid valves, thermocouples, and vacuum gauges. The board is housed in a computer-style chassis with a 120 VAC, 60 Hz power supply. It is connected to the PC via a serial data port, and the interface software allows the operator to enter all the parameters necessary for automatic control of the cleaning process.

8.3.7 Laboratory Information Management System (LIMS)

The system (LIMS) has a number of essential functions including container history and maintenance, canister status (ready to be cleaned, etc.), cleaning set-up, post analysis information, and reports.

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8.4 Pressure/Vacuum Gauge

A pressure/vacuum gauge that is calibrated and capable of measuring -14.7 psig (30 inHg) to 100psig is used to provide a more accurate reading of vacuum/pressure, so that the initial leak check reading can be monitored and recorded.

9) Standards, Reagents, and Consumable Materials

9.1 Edwards 450g Charge Activated Alumina
Purchased from Scientific Instrument Services

9.2 Vacuum Pump Oil #19
Purchased from VWR

9.3 Hydrochloric Acid

10) Preventive Maintenance

10.1 Preventative maintenance is one of the best ways to keep the conditioning systems running optimally. Maintenance log entries made into LIMS must be made each time work is performed on a system no matter the extent. Types of maintenance activities include: changing pump oil, changing in-line foretrap activated alumina media, and replacing worn ferrules.

LIMS stores system maintenance information which must remain current. An entry shall be made into LIMS every time maintenance is performed (no matter the extent). The log entry must include:

(a) The date of maintenance
(b) Who did the maintenance
(c) Description of the maintenance
(d) Proof that the maintenance activity was successful

A notation of a successful QC may serve as proof that the maintenance is complete and the system is in working order. The extent of the maintenance is not important, however, it is important that a notation be included for each maintenance activity.

10.2 Vacuum System

The mechanical vacuum pumps used in the canister cleaning system require periodic maintenance. The pump oil must be changed regularly, every *three months* or when the pump will no longer evacuate the system efficiently, whichever is more frequent. The pumps may also need to be returned to the manufacturer for rebuilding if they fail to perform to the necessary specifications.

Preventative Maintenance Schedules are as follows;

Vacuum Pumps/Change Oil.....3 MONTHS
Foretraps/Change activated alumina media.....3 MONTHS

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10.2.1 Procedure for Changing Pump Oil

- Make sure there is a sufficient amount of fresh pump oil before the oil change begins.
- Wear safety glasses and gloves when changing oil.
- Set the control software to "EVAC ON"
- Open one position on the manifold so that air is pulled through the pump.
- Turn vacuum pump power switch to the off position and remove the power cord from the electrical outlet.
- Vent the pump from above the foreline trap.
- Detach the inlet and exhaust tubing from the pump.
- Place pump on a sturdy level surface and remove both fill and drain plugs.
- The pump oil is extremely hot and can cause severe burns. If possible, turn the pump off and let it sit for approximately 20 minutes to let the heat dissipate from the pump.
- Drain the oil into an appropriate hazardous waste container. Lift the motor end of the pump to get most of the used oil from the pump housing.
- Check for oil leaks at the pump seals and gaskets (*between the motor and the pump*), also at the drain and fill plugs.
- Install the drain plug and slowly refill with fresh vacuum pump oil.
- The vacuum pumps use a sight glass to measure the amount of oil in the pump. Fill to the designated mark on the sight glass and install the fill plug.
- Apply vacuum and exhaust tubing. Plug in the power cord into the electrical outlet and turn on power switch.
- The vacuum pump will take about an hour to prime to full vacuum.

10.2.2 Procedure for Changing Activated Alumina

- Make sure there is a sufficient amount of fresh activated alumina before the change begins.
- Wear gloves when handling the fore trap and the activated alumina.
- Set the control software to "EVAC ON"
- Open one position on the manifold so that air is pulled through the pump.
- Turn vacuum pump power-switch to the off position and remove the power cord from the electrical outlet.
- Vent the manifold from one of the canister positions with the evacuation valve open.
- Detach the inlet and exhaust tubing from the pump.
- Unscrew the cap from the fore trap and extract the activated alumina media holder. Unscrew the wing nut and remove the cover.
- Dispose of the used activated alumina as hazardous waste and refill the holder with fresh media.
- Install the cover and tighten the wing nut to the top of the holder. Place the media holder back inside of the fore trap and tighten the cap to the fore trap.
- Apply vacuum and exhaust tubing.
- Plug in the power cord into the electrical outlet and turn on power switch.
- The vacuum pump will take about an hour to prime to full vacuum.

10.3 Oil Contamination

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When canisters are cleaned on the conditioning manifolds, contaminates tend to remain in the pump oil and the activated alumina fore trap. This is normal and will not affect the normal workings of the pump and the conditioner. Occasionally, a canister will be conditioned that has high concentrations of a compound and will overwhelm the conditioning system. When this contamination occurs, the pump oil and the fore trap media must be changed. Additional conditioning cycles may be needed to purge the remaining contamination from the system. High contamination scenarios must be entered into LIMS.

10.4 Gas Source with Hydrocarbon Trap and Humidifier

- The step down pressure from the bulk nitrogen tank should be set to a minimum of 15psig.
- The water level in the humidifier should always be visible in the tubing and filled with ASTM Type II water or equivalent, such as boiled deionized water. When the water is low it shall be filled to the point where at least one inch of water is showing in the tubing.

11) Procedure

11.1 IMPORTANT:

1. Canister Conditioning Technicians Canister barcodes are *not* to be hand entered into LIMS unless the barcode cannot be scanned, in which case a new barcode label must be generated. If this procedure is unavoidable the hand entered canister barcode must be verified by a secondary reviewer. Additionally, complete comments must be entered for all changes and/or additions (after the fact) made in LIMS.

2. Analysts and Canister Conditioning Technicians If during analysis or attempted analysis, a canister is identified as leaking refer to Section 11.7.3 for the specific requirements.

11.2 Release of Canisters for Cleaning/Disposal

Canisters are grouped by job number and are shelved accordingly. Canisters ready for cleaning and disposal are released according to the *SOP for Laboratory Storage, Analysis, and Tracking*. Each canister in a group of canisters must include at least the concentrations of the primary or highest level compound(s) or a short description of the sample matrix.

11.3 Pre-Purging

All canisters, except canisters returned to the laboratory as unused, are evacuated and purged prior to cleaning. When pre-purging is performed, the operator selects a group of released canisters of similar analyte levels and places them on the pre-purge manifold. The pre-purge process may be performed according to the following procedure.

- Use a 9/16" wrench to attach the canisters to the manifold
- When the timer is off, the manifold is connected to the pump. Turn the manual three-way valve to the 'down' position to open the manifold to the solenoid valve.

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- Turn on the pump and timer power strips.
- Open the canister valves, and the canisters will begin to evacuate.
- Set the current time on the timer to 1 AM (or whatever time has been programmed as the start time). Hold the CLOCK button down and use the HOUR and MIN buttons to set the time.
- Use the MANUAL button to select AUTO OFF. This activates the timer. The canisters will evacuate and fill according to the program entered into the timer.
- When the cycles have finished, close the canister valves.
- Turn the manual three-way valve to the 'closed' position.
- Turn off the pump and timer power strips.
- Remove the canisters from the purge manifold and place them onto the cleaning manifold and follow the procedure detailed in 11.4 below.

11.4 Canister Cleaning

11.4.1 Cleaning Cycles

The number of cleaning cycles needed to sufficiently clean a canister is dependent on the concentration of the sample previously in the canister. The exact number of cleaning cycles is not significant as a canister is only accepted as clean upon passing the QC check criteria outlined in section 12.1.

Canisters are typically cleaned for sixteen cycles as this is the number of cycles that can be performed during the overnight cleaning session. Shorter cleaning cycles are sufficient for low level canisters as these canisters do not require extensive cleaning in order to meet the QC check criteria. The following are examples of canisters where a minimum of five cleaning cycles may be sufficient:

- Canisters used for ambient air sampling
- Canisters that have been cleaned for sixteen cycles but still contain low levels of compounds
- Standard canisters
- Canisters returned by clients as unused

Cleaning cycles may also be cut short as long as the QC check criteria are met. Twenty-four cycles are used for weekend canister conditioning to minimize the time canisters sit evacuated on the conditioner following the procedure.

If a QC canister fails to meet the QC requirements stated in Section 12.1 another set of conditioning cycles must be performed on the entire batch of canisters. The operator and/or supervisor should determine if the problem is due to a high level canister in the set and decide whether to remove that canister (or canisters) before proceeding. Following the second reconditioning process, the canister that initially failed the QC must be analyzed unless that canister was removed from the set in which case the second highest canister must be analyzed. If the batch fails a second time, then either a third set of cleaning cycles may be performed or the entire batch taken off the conditioner and tagged for maintenance and manually cleaned. See Attachment C for a QC canister flow-chart, Section 12.1 for QC canister acceptance criteria, and Section 16.2 for additional corrective action details.

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11.4.2 Evacuation and Fill Setpoints

Typical cycle setpoints are 20 minutes for evacuation time and 10 minutes for fill time. The evacuation and fill times combined with the number of cycles necessary to sufficiently clean a canister are dependent on the sample previously in the canister. The evacuation and fill times may vary as long as the batch passes the QC check criteria in Section 12.1. Weekend setpoints will typically be longer as to minimize the time canisters sit evacuated on the conditioner following the procedure. Shorter setpoints may be used for the following:

- Canisters used for ambient air sampling
- Canisters that have been cleaned for sixteen cycles but still contain low levels of compounds
- Standard canisters
- Canisters returned by clients as unused

11.4.3 Quality Control Canister Selection and Analyses

The following are strict guidelines that must be followed in order to ensure cleaning approval and that all documentation is complete and accurate.

- All canisters used for standards must be cleaned separately and the highest concentration standard selected as the QC canister.
- The canister or canisters, depending on the number of canisters within the batch (minimum of one per batch of 16 or fewer; two per batch of 20), selected for QC, must contain the highest level of contaminants as indicated on the back of the sample identification tag. When analyte concentrations in different canisters are within approximately 10%, the canister containing the analyte with the highest boiling point should be chosen as the QC canister. See Attachment B for TO-15 compounds listed in order by boiling point. If compound concentrations in different canisters are greater than approximately 10%, then the canister with the highest individual analyte concentration should be selected as the QC canister.
- Based on the analysis to be requested by the client, as may be indicated on the media request, the batch may be analyzed specifically for TO-15, sulfur compounds, methane (% level), or other analytes or methods not covered by TO-15. However, if the canisters are to be used for TO-15 analyses, they must be checked using this method and the acceptance criteria listed in Section 12.1 (as specified for the specific canister request, LIMS "Approved Container Orders").
- If the TO-15 analysis covers another method compound list, the QC canister may be analyzed by TO-15 for those compounds.
- A notation of the method and analytes or specific reference to the compound list that the batch is approved for must be noted in the log (*TO-15 is the default method; therefore, any method utilized other than TO-15 must be specified).*
- Make a notation in LIMS (Section 11.4.4) as to the canister or canisters selected as a QC canister.

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- Make a notation on the sample identification tag that that canister was selected as the QC can, the manifold number and LIMS batch number.
- A hard copy of the quantitation report and chromatogram along with the acceptability of the batch (example, 75 + TICS states that the canister batch passed for the TO-15 75 compound list as well as tentatively identified compounds) must be kept by the laboratory and be available for review. IMPORTANT - A failing or passing result is to be entered into batch comments with name of data file as well.

Note: Refer to Section 12.1 for alternative uses for canisters that pass for specific compound lists.

11.4.4 LIMS

Once canisters are ready to be cleaned, they shall be placed on a conditioning manifold and the following steps taken to set-up and complete the cleaning procedure with respect to the Laboratory Information Management System (LIMS).

The container cleaning section in LIMS is located under:

-Under "Resource Manager" on console, select "Materials Manager"
-Select "Reusable Containers" on console
-Select "Container Cleaning" button on "Reusable Containers" screen
-Under the "Build/Update Batch" tab; select "Summa Canister" and the "New Batch" tab

1) Select container type
2) User
3) Conditioner
4) # of cleaning cycles (Section 11.4.1)
5) Scan canister barcodes in order of position on manifold
6) Select QC canister (Section 11.4.3)
7) Enter contaminants of QC canister
8) Add any additional comments (per canister)
9) Add any batch comments by selecting comments "Batch Comments" button on right of screen
10) Select "Submit" button on right of screen, which creates a unique sequential batch ID (based on the total batches created)

11.4.5 Manifold Cleaning

Upon placement on the cleaning manifold, the canisters must be entered into LIMS.

Perform the following procedure to clean canisters:

- Remove canisters from the pre-purge manifold and secure on the cleaning manifold with a 9/16" wrench.
- Tighten the brass swagelok nuts ¼ turn past finger tight
- Wrap a heating band around the middle of the canister and secure.

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- Close manifold valves on any unused positions.
- Perform a leak check prior to proceeding by:
  - Make sure the canister valves are closed; evacuate the manifold by using the manual control buttons on the main software window.
  - Activate the evacuation solenoid valve by clicking the "Evacuate" button, which will remain on until the "Evacuate Off" button is clicked.
  - With this valve on, the gauge reading should rapidly decrease to less than 100 millitorr within a few minutes. If the vacuum does not drop there is either a leak somewhere in the system, or there is a problem with the vacuum pump.
  - The problem must be isolated and fixed before proceeding. If there is a leak found refer to Section 11.7 for the appropriate maintenance activity.
  - If it is necessary to remove a canister (or cap) from the manifold, turn off the evacuation valve first.
  - If the leak check is successful, power on the canister heaters and open the canister valves.
  - Start the automated sequence at this point.
- Enter the parameters to be used for the automated sequence.
- Start the program by using the shortcut on the Windows 95 desktop or by locating and running the "clean.exe" file on the local hard drive. This should be located in the c:\vivid subdirectory. The setpoint window is accessed by clicking the "Show Dialog Window" button in the lower left corner of the main window.
  - Step 1 is the vacuum reading down to which the manifold pumps before going on to step 2. This should be set to less than 100 millitorr (as long as the requirements of this document are met the vacuum may be less).
  - Step 2 is the time (using a 24 hour clock) that the program will wait before starting the cleaning cycles. This is used when a humidification step is necessary. Make sure the PC clock time is set accurately.
  - Step 3 includes the number of fill/evacuate cycles to be performed. It also includes the times (in minutes) for each fill and evacuation period. Refer to Section 11.4.2 for this information.
  - Step 4 is the final evacuation vacuum reading to be achieved, typically <100 millitorr. These setpoints are based on past experience, but other combinations may also be used if they can be demonstrated to achieve the QC criteria used to evaluate the canisters after cleaning.
- Start the sequence by clicking the "start/stop" button on the main window.
- After the cycles are completed and the canisters have been evacuated down to less than 100 millitorr, close the canister valves.
- Stop the sequence by clicking the "start/stop" button again.
- Follow the QC canister and leak check procedures detailed in Section 12.1 and 12.2 of this document.

11.4.6 <u>Manual Cleaning of Contaminated Canisters</u>

If a canister is deemed too *highly contaminated* to be cleaned on a conditioner, or if it will not meet the QC cleaning criteria by the standard procedure, it may be necessary to manually rinse the can with acidified water. The analyst will make this determination from the analytical data obtained for a canister. The manual rinse is followed by a drying step, before once again placing it on the cleaning manifold. This method will help remove semi-volatile compounds and

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particulates that may have entered the canister during sampling, as well as to dissolved salts which could have been deposited from condensed vapors. It may also help remove surface "lacquer" formed when the canisters are heated and contain condensed vapors.

1. Prepare a slightly acidic aqueous solution by diluting 3 or 4 drops of concentration hydrochloric acid (HCl) in a liter of distilled water. The pH will be approximately 3.
2. Using the appropriate pre-purge manifold, evacuate the canister to be cleaned (25"Hg is sufficient).
3. Attach a length of ¼" PFA Teflon tubing to the first canister using a brass nut and ferrule. It may be a removable type ferrule, such as Teflon or M-4.
4. The amount of acidified water used should be about 10% of the canister volume, i.e., 600mL for a 6L canister.
5. Place the free end of the tubing into the water, then open the can valve until the desired volume has be drawn into the can. Close the valve and leave under partial vacuum.
6. Remove the tubing and shake the can for at least one minute, making sure the entire inner surface come in contact with the liquid.
7. Place the canister in an oven set to approximately 80°C overnight (or at least 12 hours).
8. Remove the can from the oven and pressurize to about 20psig with zero air, nitrogen or helium.
9. Hold the canister upside down over the sink and open the valve, allowing the pressure to blow out the liquid.
10. If liquid is discolored or there were a lot of particulates, repeat steps 4 through 6 using non-acidified distilled water.
11. Repeat step 8 and 9 until no more water comes out of the canister
12. Evacuate the can on the pre-purge manifold, leaving it under vacuum for at least 30 minutes.
13. Purge the canister two more times, which should remove enough residual liquid to allow the canister to be placed on the cleaning manifold.
14. Put the canister through the normal cleaning cycles. QC each canister that was manually cleaned.

11.5 <u>Leak Check</u>

The leak check is an individual canister certification. The two procedures listed in this section must be completed following the cleaning procedure detailed in Section 11.4.

- While on manifold, close QC can and pressurize remaining canisters with Nitrogen to 10-15 psig.
- Read pressure from calibrated digital pressure/vacuum gauge (section 8.4) and record the initial reading and time the reading was taken in LIMS under the "Initial Vacuum" tab of "Build/Update Batch". Additionally, refer to Section 11.7.1 if a stable reading is not achieved.
- Close all canister valves.
- Disconnect all of the canisters from manifold.
- *Place the caps on the valves of all canisters (except the QC canister) with only a recommended ¼ turn to ensure that the leak check takes into account any issue with the valve closure.*

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- Pressurize the QC canister(s) with humidified zero grade air (>20% RH) using the pressure/vacuum gauge to achieve the appropriate pressure (15psig).
- Alternatively, these canisters may be pressurized utilizing a canister zero grade air fill station with humidifier.
- Submit the QC canister(s) for analysis.

Note 1: If a stable reading is not achievable, then each canister pressure must be evaluated individually and the reading recorded accordingly.

- Allow each canister to sit for a minimum of 24 hours (Refer to Note 2 below).
- Measure the final pressure (using the calibrated pressure/vacuum gauge on the manifold) and record the reading (under the "Final Vacuum" tab of "Build/Update Batch" in LIMS) as well as the time of the measurement.
- Acceptance criteria – a vacuum change of <2.0psig.
- If any canister is deemed to be leaking per this procedure then follow the steps in Section 16.3.

Note 2: All canisters including the QC canisters must be leak checked. If the leak check procedure is either not performed or cut short on any canister, it must done with the prior approval of the appropriate Project Manager. The person authorizing this departure from stated procedures as well as the specific reason for the departure must be noted in LIMS.

11.6 Batch Approval

Refer to Section 12.1 for information on batch approval requirements and approval with conditions.

Once the QC results have been submitted by the analyst, access the "QC Results" tab under "Build/Update Batch" in LIMS.

1) Select Batch
2) Select User
3) QC results button
   a) Select the appropriate passed for components
   b) Add any necessary comments
   c) Select the applicable update (batch or individual canisters)
   d) Select the canister IDs, where appropriate
   e) Select the analytes, where appropriate
   f) Next
4) Add QC results, where appropriate
5) Update QC results

11.7 Troubleshooting

11.7.1 Leaking Conditioning Manifold

If a leak develops in the canister cleaning manifold, and it is not due to a leaking canister, then the manifold must be systematically checked until the leak is found. The most obvious symptom of leaking manifold is when the

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system will not pump down to its normal ultimate vacuum and the pirani gauge reading rises steadily after the pump isolation valve is closed.

- With a full set of canisters attached, close all the canister valves tightly. Open the pump isolation valve (Evac. on). The gauge should read <100mtorr. If it does not, close the manifold valves one at a time while watching the gauge display. If the reading drops quickly after one valve is closed, the leak is at that position; proceed to the next step.
- Recheck the canister valve to be sure it is tightly closed. If the vacuum still does not go down to <100mtorr when that manifold valve is opened, the most common problem is a bad canister valve.
- Remove the canister from the leaking position and cap the end with a brass cap. Reopen the manifold valve and watch the gauge display. If it drops to <100mtorr, the problem was the canister valve. If it still does not drop, proceed to the next step.
- Close the manifold valve and replace the ferrule at the end of the flex hose. Recap the end and then open the valve. If this does not solve the problem, close the valve, remove the flex hose, and plug the valve fitting with a brass plug.
- Reopen the valve. If the vacuum still does not drop, replace the valve. Otherwise replace the flex hose and brass nut.

If the system will not pump down even with all of the manifold valves closed, the leak must be in the manifold itself or the problem may be a faulty pressure isolation valve. This is an extremely rare situation. Diagnose the problem using the following steps:

- Disconnect the gas inlet tubing from the manifold. This will eliminate the solenoid valve from the system. Open the pump isolation valve and check the vacuum gauge. If it pumps down to <100mtorr, replace the pressure isolation valve.
- If the manifold still does not pump down, carefully tighten each fitting on the manifold using the appropriate wrenches. If this does not solve the leak, it may be necessary to reconnect the gas source and replace the nitrogen gas with helium. Pressurize the manifold with helium and use an electronic leak detector to check each fitting. Replace any leaking pieces with new tubing and ferrules.

11.7.2 <u>Contaminated Conditioning Manifold</u>

- Clean the manifold by closing all of the manifold valves and clicking the “fill on” button on the screen. This will pressurize the system with UHP Nitrogen gas and can purge the stainless steel lines of the contaminant.
- Next use a heat source, i.e. heat gun, and start heating the fittings and the tubing at position one (1).
- Open the manifold valve at position one (1).
- Leave open for about one (1) minute and keep heating the tubing and the fittings surrounding the valve. This process will purge the contaminants from that valve’s outlet and the heat will help volatilize the compounds from the inner surface of the stainless steel tubing.
- Close valve and move to the next valve.

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- Continue this process for valves two (2) through remaining valves on the manifold in question.
- Refer to Section 11.3 for the pre-purging and canister cleaning procedure.

11.7.3 "Leaking" Canisters (Identified by Analysts or Other Such Sources)

This section is for those canisters that are suspected of leaking as identified by analysts using the AUTOCAN, during pressurization, or by other means and other individuals. This section is NOT for those canisters identified by the Canister Conditioning Department during the leak check or identified as leaking on the conditioner as those are required to go straight on Maintenance Hold.

Identified canisters are to be handled according to the following instructions and number 1 is to be completed first. This is regardless of the PM's or client's instructions to proceed.

1) Obtain a leaking canister or maintenance tag, place it on the canister and label as leaking, add initials and date.
2) Initiate a NCAR and write the NCAR # on the tag.
3) Also, make a note on the tag whether or not the analysis proceeded (e.g. "Analyzed" or "Canceled")
4) If canceled, then the Canister group can proceed and place the can on Maintenance Hold to perform a formal leak check or clean and perform the required 24hr leak check.
5) If analyzed, then the canister should be placed with the other canisters in the job and when cleaning is to take place then put the canister in question on Maintenance Hold.

IMPORTANT: All canisters that are suspected of leaking MUST have a tag stating such and NO canister that is deemed to be leaking is to go without the *full* 24hr leak check.

11.8 Documentation

11.8.1 Canister Conditioning

Information regarding each cleaning batch is maintained in LIMS. A notation is entered into LIMS for each cleaning batch regarding its QC status (Pass, Fail, etc.).

11.8.2 Canister Maintenance

The LIMS container tracking system must be utilized to track canister maintenance. An entry must be made to place a canister on maintenance hold, the maintenance to be performed and when maintenance is performed. The information must include the bar code number of the canister, date for the repair, a detailed description of the repair, and the initials of the repairer.

11.8.3 Manifold Maintenance

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Manifold maintenance is tracked through LIMS. Information must be entered into LIMS for scheduled and non-scheduled projects.

12) Quality Assurance / Quality Control Requirements

12.1 QC Canister Criteria

Once the cans have gone through the cleaning process, it is necessary to verify that all previous contaminants have been removed. This is accomplished by selecting a minimum of one canister per manifold/batch (10 or 16 or fewer) and two for a 20 canister batch (or fewer) to be analyzed for VOCs by GC/MS. However, all canisters that are to be analyzed for TO-15 (SIM mode) are individually analyzed/QC checked for the Client's specific compound list.

No analyte concentration may be at or exceed the canister cleaning requirement of 0.2ppbv or if above the required reporting limit (if lower than 0.2ppbV, also refer to the important note below) in order for the canister to be considered acceptable.

Analyte Exceptions	
Acetone	<1.26ppbv
Vinyl acetate	<0.85ppbv
Ethanol	<1.59ppbv
IPA	<0.31ppbv
1-Butanol	<0.30ppbv
2-Butanone	<1.0ppbv
Carbon Disulfide	<0.96ppbv
Acrolein	<0.65ppbv

- There must be no compound detected as a tentatively identified compound above the level specified in the associated TO-15 method SOP.
- If the canister has target contaminants above 0.2 ppbv or the MRL (if lower than the canister cleaning requirement), the entire batch is rejected and the cleaning process repeated.
- All canisters selected as QC canisters (all canisters for TO-15 SIM) for a cleaning batch must meet the requirements detailed above in order for the batch to be certified as "clean".

IMPORTANT NOTE: Method Reporting Limit – A client may request a certain method reporting limit for TO-15 that is lower than the 0.2ppbV canister cleaning requirement. In this case, it must be conveyed to the analyst. All TO-15 SIM reporting limits are lower than this canister cleaning requirement; therefore, they are all individually analyzed and approved.

Passed with Conditions

Depending on what the QC canister fails for, the laboratory may be able to use it or the batch for other media requests. For example, if the canister (SIM) or batch (SCAN) passes for BTEX only it may be entered into LIMS as "passed with conditions" and

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labeled accordingly. In this case, the canisters may be used to fulfill a media request for canisters for the BTEX compound target analyte list.

- A notation must always be made in LIMS if the QC canister(s) passed or failed.

12.2 Leak Check Criteria

There is no defined requirement for the initial reading; however, the initial reading for the vacuum should not be less than –28.0"Hg (i.e., 27.9, 27.8, etc.). The change in the initial and final readings must be <2.0"Hg.

13) Data Reduction and Reporting

13.1 All applicable data from this procedure must be documented through LIMS, and may also be found in physical logbooks.

14) Method Performance

14.1 Not Applicable

15) Pollution Prevention and Waste Management

15.1 Activated Alumina and pump oil is considered hazardous waste and must be disposed of in accordance with the *SOP for Waste Disposal.* Air purged from canisters via the vacuum pumps is discharged to an outside hood.

16) Corrective Actions for Out-of-Control Data

16.1 All corrective action plans must be developed in accordance with the *SOP for Nonconformance and Corrective Action*.

16.2 Corrective Action for Failed QC Canister

If one QC canister has failed to meet the QC requirement stated in Section 12.1, or the batch may not be used for another analytical method, another set of conditioning cycles must be performed on the entire batch of canisters in question in accordance with Section 11.4.1. Follow the corrective action guideline detailed in the flow chart in Attachment C. The operator and/or supervisor should determine if the problem is due to a high level canister in the set and decide whether to remove that canister (or canisters) before proceeding. This second run should clean the batch sufficiently. However, following the reconditioning process the canister that initially failed the QC must be analyzed (if it has not been removed from the set, in which case the second highest must be evaluated). If the batch fails again, then either all of the canisters are taken off of the conditioner and put in a "Maintenance Hold" position, where they are tagged as needing to be manually cleaned or a third set of cleaning cycles is performed. Otherwise, if the canisters pass the QC check, the batch is released for use.

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If it is suspected or determined that the conditioning manifold is contaminated follow the cleaning procedure detailed in Section 11.7.2 and the corrective action in Section 16.4.

Note 1: The requirements listed above are the minimum actions that must be taken. There must be sufficient evidence (number of QC canisters analyzed within a single batch) to ensure that the entire batch of canisters meets the analyte concentration requirement of this document.

Note 2: No batch (SCAN) or canister (SIM) may be released unless all QC canisters have passed the analyte concentration requirements or it is safely selected for use for another method and analyte list.

16.3 Corrective Action for Failed Leak Check

Once a canister has failed the leak check, it must be placed on maintenance hold in the container tracking system until repaired. All canister maintenance must be noted in LIMS as detailed in Section 11.8.2.

The most common cause is a leaking valve seat, which may be caused by repeated over-tightening or debris falling into the valve and preventing it from closing. Another possibility is that the valve is not sufficiently tightened onto the canister. It may be necessary to find the exact position of the leak by utilizing one or more of the following procedures:

- It possible to find leaks by pressurizing the canister to 20 psig and submerging it in water and looking for a stream of air bubbles.
- A leaking canister may also be detected by pressurizing the canister to 20psig with UHP Helium gas and using a Helium gas leak detector to “sniff” out a leak. The operator will use the detector probe around the valve head, valve seat, valve body, valve stem and the fastener nut fitting of the canister to allow the detector to find the leak.

16.3.1 Corrective Action for Leaking Valve

If it is determined that a canister valve is leaking and needs to be replaced, a Nupro "H" Series stainless steel bellows seated valve should be used. The valve body has 1/4" male Swagelok end fittings and the only tools needed for changing a valve are a 13/16" and a 9/16" wrench.

16.3.2 Corrective Action for Leaking Valve Stem

If the leak is found at the valve stem, it may be replaced.

- Remove the valve body with a 9/16” wrench.
- Remove the valve stem with 11/16” wrench from the canister inlet nut.
- Use jewelers pick to remove any residual Teflon tape from the threads of the inlet nut.
- Wrap the new valve stem with Teflon tape, about 4 turns.
- Install the valve stem to the canister inlet nut and tighten with an 11/16” wrench.

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- If the leak is from the center seam weld, the canister must be sent out for repair and the supervisor notified.

16.3.3 Corrective Action for Leaking Summa Canister

If a leaking valve is identified, it should be replaced with an identical or equivalent valve. The canister is put in the "Maintenance" section in LIMS and tagged as such and repairs are specified on the tag. The laboratory supervisor should be contacted regarding this type of repair. After replacing the valve, the canister shall be put in "to be cleaned" mode in the tracking database. It must be part of a cleaning set and pass batch QC before being put back in service.

16.4 Corrective Action for Contaminated Manifold

If it is determined that a manifold is contaminated, changing the pump oil and foreline trap adsorbent will usually solve the problem. After maintenance is preformed on the system (Section 11.7.2), the canister or set of canisters which caused the contamination should be removed and cleaned by the procedure described in Section 11.4.1. A minimum of two canisters shall be QC checked and meet the criteria in Section 12.1 before that set may be certified and further batches cleaned on that manifold.

16.5 Corrective Action for Leaking Manifold

If there is a leaking manifold, it must be repaired and any canister affected shall be reconditioned and a QC check performed. A leak check shall be performed (Section 11.7.1) following any maintenance to determine if the maintenance was successful.

Replacement fittings and tubing may be obtained in house. However, if parts must be ordered for the manifold, the unit will be in a "Lock Out" or unusable mode and a notation of the "Lock Out" status entered into LIMS.

17) Contingencies for Handling Out-of-Control or Unacceptable Data

17.1 In the case of unacceptable data being produced on a batch or specific canister, it shall be re-cleaned or put on maintenance hold for repair. In certain cases a canister may become reclassified for use (ex. ambient to source).

18) Training

18.1 Training shall be conducted in accordance with the *SOP for Training Policy*. The training plan, which includes all of the responsibilities and requirements included in this SOP, must be completed for each trainee. In addition, an initial demonstration of proficiency (IDP) shall be performed prior to independent performance of the procedures detailed in this document. The IDP must consist of two successful QC canisters, from two separate batches utilizing the same acceptance criteria specified in this document.

19) Method Modifications

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19.1 Not Applicable

20) Summary of Changes

20.1 SOP updated using ALS SOP Template – New cover page, document tracking/signature page, and table of contents page. Header/footer and font revised throughout SOP to align with ALS SOP template specifications. Additionally, sections were reorganized as outlined in the *SOP for Establishing Standard Operating Procedures* and section references updated as necessary. Section titled "Instrument Specific Addendum" removed (Section did not contain any information and had "Not Applicable" listed.

Section 1	Updated section heading to "Scope and Applicability"
Section 2	Updated section heading to "Summary of Procedure"
Section 4	Was Section 10 in previous SOP revision
Section 5	Was Section 4 in previous SOP revision
Section 6	Was Section 5 in previous SOP revision
Section 6.2	Updated second sentence
Section 7	Was Section 6 in previous SOP revision
Section 7.1	Removed Meriter 2.4-liter passivated stainless steel canisters from list
Section 8	Was Section 7 in previous SOP revision
Section 8.3	Included "ovens"
Section 8.3.1	Updated last paragraph
Section 9	Was Section 8 in previous SOP revision
Section 10	Was Section 9 in previous SOP revision
Section 11.3	8th bullet – changed "up" to "closed"
Section 11.4.3	7th bullet – removed analyst requirement to include LIMS batch numbers as part of QC sample identification
Section 11.5	1st bullet – updated to "10-15psig"
Section 16.1	Updated SOP title
Section 18	Was Section 19 in previous SOP revision Renamed section heading to "Training"
Section 18.1	Updated SOP title
Section 19	Was Section 20 in previous SOP revision
Section 20	Was Section 22 in previous SOP revision Renamed section heading to "Summary of Changes"
Section 21	Was Section 18 in previous SOP revision
Section 21.3	Updated SOP title and ID
Section 21.4	Updated SOP ID
Section 21.5	Updated SOP title and ID
Section 22	Was Section 23 in previous SOP revision
Attachment A	#3 - updated 1st and 3rd SOP titles

21) References

21.1 *SOP for Waste Disposal,* SOP Code: DSP-WASTE

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21.2 *SOP for Determination of Volatile Organic Compounds in Air Samples Collected in Specially Prepared Canisters and Gas Collection Bags by Gas Chromatography/Mass Spectrometry (GC/MS)*, SOP Code: VOA-TO15
21.3 *SOP for Nonconformance and Corrective Action*, SOP ID: CE-QA008
21.4 *SOP for Preventive Action*, SOP ID: CE-GEN004
21.5 *SOP for Training Policy*, SOP ID: CE-QA003
21.6 *SOP for Laboratory Storage, Analysis, and Tracking*, SOP Code: ADM-LabSAT

22) Attachments

22.1	Attachment A	Training Plan
	Attachment B	TO-15 Compounds Ordered by Boiling Point
	Attachment C	QC Canister Corrective Action Flow Chart

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Attachment A

Training Plan

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Training Plan for Cleaning and Certification of Summa and Other Specially Prepared Canisters

Trainee _____ Trainer _____ Date _____

1. Read SOP — Trainer ____ Trainee ____ Date _____

2. Demonstrated understanding of the scientific basis of the Conditioning and Certification of Canisters — Trainer ____ Trainee ____ Date _____

3. Demonstrated familiarity with related SOPs — Trainer ____ Trainee ____ Date _____
   - SOP for Making Entries Onto Analytical Records
   - SOP for Laboratory Storage, Analysis, and Tracking
   - SOP for Nonconformance and Corrective Action

4. Observe performance of SOP — Trainer ____ Trainee ____ Date _____
   - ___Canister selection
   - ___Pre-purging of highly concentrated samples
   - ___Conditioning batches and placement of canisters
   - ___LIMS batch setup
   - ___Using conditioning manifold and conditioning program
   - ___QC canister selection and documentation
   - ___Batch certification
   - ___Leak check procedure and documentation
   - ___LIMS approval and labeling
   - ___Logbook documentation
   - ___Racking canisters back into available inventory

5. Perform SOP with supervision — Trainer ____ Trainee ____ Date _____
   - ___Canister selection
   - ___Pre-purging of highly concentrated samples
   - ___Conditioning batches and placement of canisters
   - ___LIMS batch setup
   - ___Using conditioning manifold and conditioning program
   - ___QC canister selection and documentation
   - ___Batch certification
   - ___Leak check procedure and documentation
   - ___LIMS approval and labeling
   - ___Logbook documentation
   - ___Racking canisters back into available inventory

6. Independent performance of the SOP — Trainer ____ Trainee ____ Date _____
   - ___Canister selection
   - ___Pre-purging of highly concentrated samples
   - ___Conditioning batches and placement of canisters
   - ___LIMS batch setup
   - ___Using conditioning manifold and conditioning program
   - ___QC canister selection and documentation
   - ___Batch certification
   - ___Leak check procedure and documentation
   - ___LIMS approval and labeling
   - ___Logbook documentation
   - ___Racking canisters back into available inventory

7. Instrument operation and maintenance — Trainer ____ Trainee ____ Date _____
   - ___Manual canister cleaning
   - ___Canister maintenance and logbook entries
   - ___Vacuum pump and manifold maintenance and logbook entries

8. Troubleshooting — Trainer ____ Trainee ____ Date _____
   - ___Finding leaks in manifold system
   - ___Detecting leaks in canisters

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Attachment B

TO-15 Compounds Ordered by Boiling Point

TO-15 Compounds Ordered by Boiling Point					
1	Propene	33	Ethyl tert-Butyl Ether	65	Styrene
2	Dichlorodifluoromethane (CFC 12)	34	1,2-Dichloroethane	66	o-Xylene
3	Chloromethane	35	1,1,1-Trichloroethane	67	n-Nonane
4	1,2-Dichloro-1,1,2,2-tetrafluoroethane (Freon 114)	36	Isopropyl acetate	68	1,1,2,2-Tetrachloroethane
5	Vinyl Chloride	37	1-Butanol	69	Cumene
6	1,3-Butadiene	38	Benzene	70	alpha-Pinene
7	Bromomethane	39	Carbon Tetrachloride	71	n-Propylbenzene
8	Chloroethane	40	Cyclohexane	72	3-Ethyltoluene
9	Ethanol	41	tert-Amyl Methyl Ether	73	4-Ethyltoluene
10	Acetonitrile	42	1,2-Dichloropropane	74	1,3,5-Trimethylbenzene
11	Acrolein	43	Bromodichloromethane	75	alpha-Methylstyrene
12	Acetone	44	Trichloroethene	76	2-Ethyltoluene
13	Trichlorofluoromethane	45	1,4-Dioxane	77	1,2,4-Trimethylbenzene
14	Isopropyl Alcohol	46	Isooctane	78	tert-Butylbenzene
15	Acrylonitrile	47	Methyl Methacrylate	79	n-Decane
16	1,1-Dichloroethene	48	n-Heptane	80	Benzyl Chloride
17	tert-Butanol	49	cis-1,3-Dichloropropene	81	1,3-Dichlorobenzene
18	Methylene Chloride	50	4-Methyl-2-Pentanone	82	1,4-Dichlorobenzene
19	Allyl Chloride	51	trans-1,3-Dichloropropene	83	sec-Butylbenzene
20	Trichlorotrifluoroethane	52	1,1,2-Trichloroethane	84	p-Isopropyltoluene
21	Carbon Disulfide	53	Toluene	85	1,2,3-Trimethylbenzene
22	trans-1,2-Dichloroethene	54	2-Hexanone	86	1,2-Dichlorobenzene
23	1,1-Dichloroethane	55	Dibromochloromethane	87	d-Limonene
24	Methyl tert-Butyl Ether	56	1,2-Dibromoethane	88	n-Butylbenzene
25	Vinyl Acetate	57	Butyl Acetate	89	1,2-Dibromo-3-Chloropropane
26	2-Butanone (MEK)	58	n-Octane	90	n-Undecane
27	cis-1,2-Dichloroethene	59	Tetrachloroethene	91	1,2,4-Trichlorobenzene
28	Diisopropyl Ether	60	Chlorobenzene	92	Naphthalene
29	Ethyl Acetate	61	Ethylbenzene	93	n-Dodecane
30	n-Hexane	62	m- & p-Xylene	94	Hexachlorobutadiene
31	Chloroform	63	Bromoform		
32	Tetrahydrofuran	64	Cyclohexanone		

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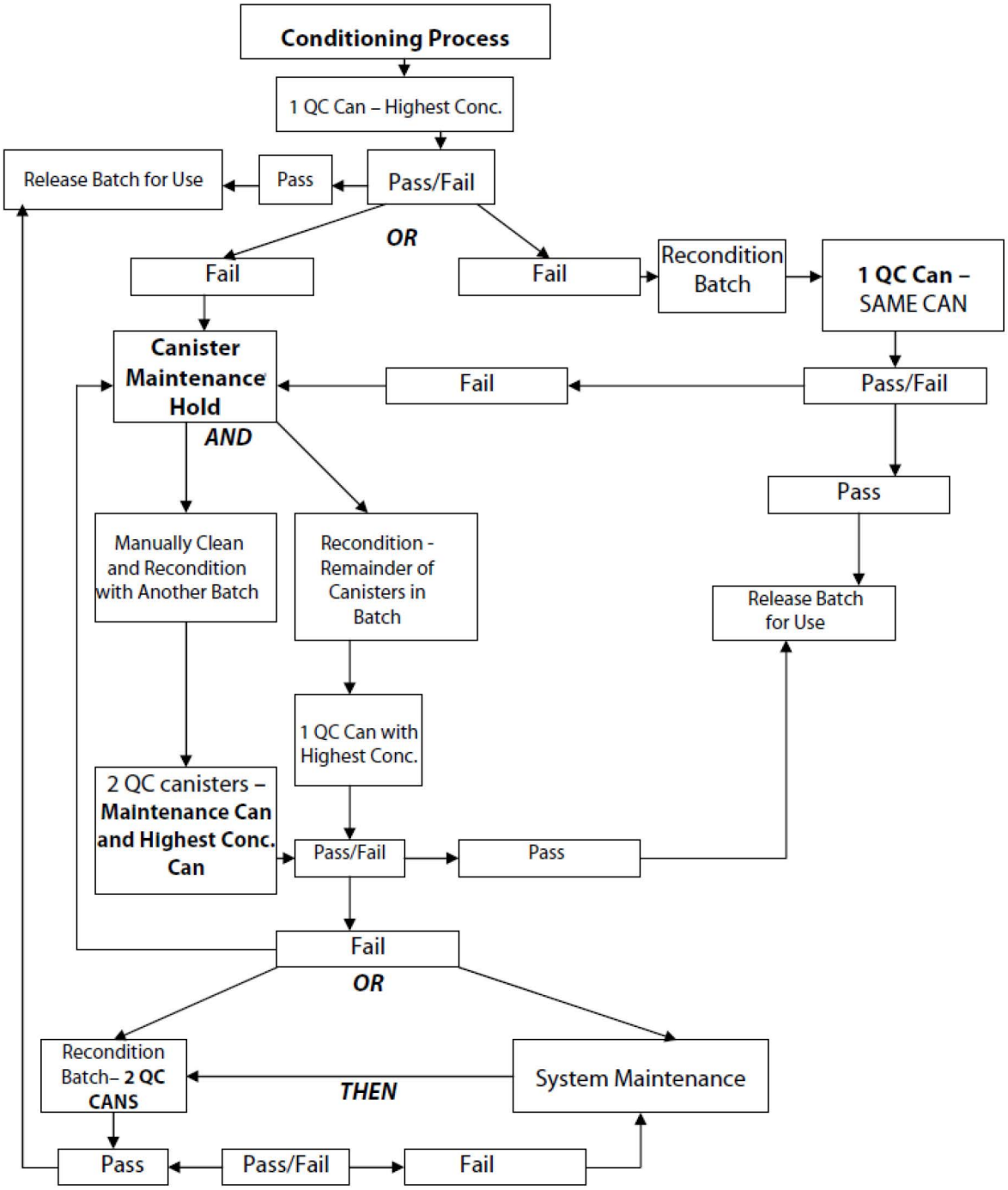
Attachment C

QC Canister Corrective Action Flow Chart



Note: This flow chart is designed for SCAN analyses, for SIM the number of QC canisters must include all of the canisters designated for SIM reporting. The 24 hour leak check is not included in this chart; however, the batch is to be released after all of the canisters have passed this check. Any canister failing the leak check is to be placed on maintenance hold in LIMS.

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APPENDIX E

ALS ENVIRONMENTAL
STANDARD OPERATING PROCEDURE FOR EVALUATION AND PRESSURIZATION OF SPECIALLY PREPARED STAINLESS STEEL CANISTERS

DOCUMENT TITLE:	*EVALUATION AND PRESSURIZATION OF SPECIALLY PREPARED STAINLESS STEEL CANISTERS*
REFERENCED METHOD:	*N/A*
SOP ID:	*SMO-CAN_PRESS*
REV. NUMBER:	*11.0*
EFFECTIVE DATE:	*01/26/2013*

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EVALUATION AND PRESSURIZATION OF SPECIALLY PREPARED STAINLESS STEEL CANISTERS

SOPID:	SMO-Can_Press	Rev. Number:	11.0	Effective Date:	01/26/2013

Approved By: _____ Date: 1/11/13
Operations Manager – Chris Parnell

Approved By: _____ Date: 1/11/13
QA Manager – Chaney Humphrey

Approved By: _____ Date: 1/11/13
Laboratory Director – Kelly Horiuchi

Archival Date: _____ Doc Control ID#: Non-Controlled Editor: _____

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EVALUATION AND PRESSURIZATION OF SPECIALLY PREPARED STAINLESS STEEL CANISTERS

1) Scope and Applicability

1.1 This document describes the procedure for performing pressure/vacuum evaluations and the pressurization of canisters upon laboratory receipt. The procedure detailed herein is necessary to ensure consistency with respect to documentation, method, gas, and gauge type and use. This SOP is applicable to all samples submitted in specially prepared canisters and must be followed for each pressurization process performed.

2) Summary of Procedure

2.1 The initial (upon receipt) pressure/vacuum reading is taken using a calibrated digital pressure/vacuum gauge. The canister is usually pressurized to approximately 3.5psig (may be dependent on the altitude sampled) and the final pressure recorded. The gas (either humidified zero grade air, nitrogen or helium) utilized in this process is dependent on the analyses requested; therefore, the analyst must ensure that the selection of the pressurizing gas is chosen in accordance with the guidelines of this document.

3) Definitions

3.1 Summa Canister A stainless steel air sampling canister whose inner surface has been electropolished by the proprietary "Summa" process to provide chemical inertness.

3.2 Service Request (SR) Form An internally generated document used to transmit sample and sample-analysis information to laboratory personnel. The service request number is a unique laboratory code which is assigned to a sample or group of samples submitted (at the same time) by the client representing one job or project.

3.3 psig Pounds per square inch gauge

3.4 psia Pounds per square inch absolute

3.5 in-Hg Inches of Mercury

4) Responsibilities

4.1 It is the responsibility of all laboratory personnel who perform the duties described in this document to comply with the all documentation and procedural requirements.

5) Interferences

5.1 Sample Cross Contamination

It is possible for sample canisters to cause cross-contamination in subsequent canisters if the lines are not sufficiently purged between pressurizations; therefore, a minimum of 5 seconds is recommended.

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5.2 Balance Gas Contamination

The pressure in the balance gas cylinders should not be allowed to go below 500psi. This is due to the fact that certain heavier contaminants such as argon settle and may be admitted into the sample upon pressurization.

6) Safety

6.1 The use of gases from high pressure gas cylinders is required for this procedure. Care should be taken when moving cylinders. All gas cylinders must be secured to a wall or a fixed lab counter with a chain or cylinder clamp at all times.

7) Sample Collection, Containers, Preservation, and Storage

7.1 Not Applicable

8) Apparatus and Equipment

8.1 Bubbler - Model ZAB2 (Lab Commerce, Inc. – San Jose, CA)

8.2 Purifier - Standard activated charcoal-filled hydrocarbon trap

8.3 Digital Compound Gauges - Vacuum/Pressure Gauge

All digital compound gauges shall be used within manufacturer recommended specifications and undergo annual calibration service. Gauge calibration range requirements are -30 in-Hg (-14.73 psig) to 100 psig.

8.4 Miscellaneous Equipment

SS Tubing / Toggle Valve
9/16" Wrench
Small Flat-Head Screwdriver
Pressurized Gas Regulators - C580 (nitrogen and helium), C590 (air)

9) Standards, Reagents, and Consumable Materials

9.1 UHP Nitrogen - 99.999% purity

9.2 UHP Helium - 99.999% purity

9.3 Zero Grade Air - <0.1ppm total hydrocarbons

9.4 DI Water – Laboratory produced

9.5 Certificates and purity verifications must be maintained on file.

Note: Certificates of Analysis from Airgas may be found online at http://www.airgas.com/certs.

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10) Preventive Maintenance

10.1 Digital Compound Gauges

All gauges in use shall maintain current calibration status and be evaluated every 6 months by performing calibration checks. This calibration shall be in compliance with the *SOP for Calibration and Use of Laboratory Support Equipment* and be documented.

10.2 Replacing Cylinders

The balance gas cylinder should be replaced before the pressure drops below 500psi. Make sure that the cylinder valve is closed prior to disconnecting the current cylinder. Attach the regulator to the new tank and turn cylinder valve on sufficiently in order to purge the connection. Allow the gas to flow for at least 10 minutes before filling a clean, evacuated canister for QC. Refer to section 12.3 for instructions on certifying balances gases.

10.3 Gas Source with Hydrocarbon Trap and Humidifier

- The water level should always be visible in the tubing and filled with DI water. When the water is low it shall be filled to the point where at least one inch of water is showing in the tubing.
- Replace the hydrocarbon trap with new carbon whenever the quality check of a gas source indicates a problem (Section 12.3.1). Note the carbon replacement on the trap label.

11) Procedure

11.1 The pressurization step is an integral portion of the laboratory procedure as it affects final results. Reporting limits (RLs) are a function of the analytical system, the pressurization of the sample canister in the laboratory, and the concentration of target and non-target compounds.

Sample RL = Base RL * Pressurization Dilution Factor (DF) * Analytical DF

Pressurization Dilution Factor: The pressurization step performed by the laboratory at the time of receipt dilutes the sample and results in an increase in the base reporting limit. The reporting limit is calculated by multiplying the base reporting limit by the pressurization dilution factor. The pressurization dilution factor is simply the ratio of the final canister pressure and the initial (as received) sample pressure. Therefore, the more sample volume collected (lower vacuum reading), the lower the dilution factor and reporting limit and the lower the final pressure due to the pressurization step, the lower the dilution factor. The final pressure is typically dependent on the canister size. *A 6L canister is typically pressurized to ~3.5 psig, while a 1L canister is typically pressurized to ~5.0 psig to allow for sufficient volume for multiple analytical runs.* However, for certain applications, alternative final pressures may be performed to minimize dilution and meet a lower reporting limit. Refer to all associated documentation to determine if alternative final pressures are required.

In addition, canisters may require additional pressure in order for further analyses to be conducted. In this case, follow the procedure above. Gas usage, upon re-

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pressurization, must be consistent and every process must be properly documented in accordance with Section 11.4.

11.2 Initial Pressure Check (Upon Receipt)

This initial check must be performed on each submitted canister sample and should be performed at the time of receipt and must be recorded on the corresponding Service Request Form. Refer to the *SOP for Sample Receipt, Acceptance, and Log-in* for specific procedures for the receipt of samples. The pressure/vacuum of all canisters must be evaluated at sample receipt and canisters should be pressurized that same day and all values (initial and final) recorded in LIMS. The reading should be recorded to two decimal places. If the sample canister is not pressurized immediately after the initial pressure check, use the subsequent readings (immediately before and after pressurization) for calculating the pressure dilution factor. This is important because the temperature of the can may change in the interim which would result in a deviation from the initial reading. The before and after pressures must be taken under the same conditions. See sections 12.6.2 and 16.3 for the procedure to deal with a discrepancy between the two readings.

11.2.1 Pressure/Vacuum Requirements

Upon evaluation, readings for ambient and source canisters should be as follows:

Ambient/Indoor Air - Grab samples should be near ambient pressure (-0.5 in-Hg or -0.2 psig to 0.5 psig).

Time Integrated Samples - Should be slightly negative (down to –5 in-Hg or -2.5 psig).

Source - Varies with application but typically ranges from 5 psig to -5 in-Hg.

11.2.2 Initial Pressure/Vacuum Evaluation (Sample Receipt)

A digital pressure gauge with an up-to-date calibration verification may only be used, as indicated on the calibration sticker located on the gauge. There must be separate gauges for checking ambient and source canister samples as a precaution against cross-contamination. The person performing the checks must examine the chain of custody and check the tag on each canister before proceeding.

- Select the correct gauge (ambient or source). Zero the digital pressure gauge with a small screwdriver (if needed) and attach to canister with wrench.
- Open the valve on the canister and take a reading (allow to stabilize).
- A TIF\Cole Parmer gauge will read in inches of Mercury (in-Hg) if the canister has a negative reading, this value will need to be converted, however if the initial pressure reading is positive then the pressure reading is given in psi. To obtain the conversion from in-Hg to pounds per square inch (psi), a conversion chart is located in this SOP as Attachment A. If an APG digital pressure gauge is used, conversion will not be needed.
- Record the psig value on the back of the sample tag.

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- For all canisters, use the vacuum pump line to purge the gauge several times before checking each sample. Turn on the pump and attach the flexible silicone tubing connector to the gauge fitting for several seconds. Repeat this at least two more times before going on to the next canister.

11.3 Pressurization for Analysis

There are a few very important guidelines that must be followed when pressurizing canisters submitted to the laboratory for analysis. The same pressurization gas must be used each time a canister is pressurized. Refer to the sample tag and Service Request form in LIMS for the gas utilized. A helium tag should be placed on each canister that requires pressurization with helium; however, all documentation must be reviewed.

IMPORTANT: The pressurization of samples that are known to be trip blanks must be witnessed by a second person. Both individuals must initial and date the appropriate section on the back of the canister's sample tag.

Note 1: The Service Request form in LIMS must be opened and the requested analyses must be checked to ensure that the proper pressurization gas is selected. If there is any question as to the required analyses, contact the Project Manager prior to pressurization.

Note 2: If the canisters are backfilled, the gas used to pressurize must be the same.

Pressurization Gas Guidelines (See Attachment C)

1. If NEITHER TO-15 or EPA Method 3C (fixed gases) analyses are requested, pressurize with either humidified zero air or helium (Section 11.3.1 or 11.3.2).
2. If EPA Method 3C (fixed gases) is requested and TO-15 is NOT requested; the canisters must be pressurized with helium (Section 11.3.1).
3. If TO-15 is requested and EPA Method 3C (fixed gases) is NOT requested; pressurize with humidified zero grade air (Section 11.3.2).
4. If BOTH TO-15 AND EPA Method 3C (fixed gases) are requested, then one of the following must be observed.

    a. Two canisters with the same client sample identification must be submitted; one for TO-15 (Zero Air) and one for fixed gases (Helium).
    b. Pressurize with helium and note the pressurization gas on the tag and SR form. A notation of the pressurization gas used must be indicated so that method TO-15 is reported to the client as modified.

    If neither of these is observed then the person filling the canisters must contact the Project Manager so that the client may be contacted and appropriate corrective action taken, where necessary.

IMPORTANT: It is possible for sample canisters to cause cross-contamination in subsequent canisters if the lines are not sufficiently purged between pressurizations. A minimum of 5 seconds is recommended.

11.3.1 Helium Pressurization

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This gas is primarily used for backfilling and the pressurization of backfilled canisters and canisters deemed for analysis by EPA methods 3C and 25C.

- Make sure the helium tank regulator is open and reads positive pressure of at least 500 psig. If the pressure is under 500 psig then the tank will be replaced.
- Turn the gas supply valve open to ≤40psig and allow to flow for approximately 15 seconds.
- Zero the digital pressure gauge by pressing the Zero button (or with a small screwdriver for TIF gauges), if needed.
- Attach the tube fitting connector to the canister valve connector and tighten the fitting finger tight.
- Turn off the valve and ensure there is a good seal (i.e. the pressure does not drop).
- Open the valve on the canister and take a reading. If the value is negative and in in-Hg units, compare the reading observed to the chart located on the wall beside the tank and obtain the value in psig. Record the pressure (psig) on the Service Request Form under Can Pressurization Section: P_i (pressure initial - *psig*).
- Open the gas supply valve and allow the canister to fill to a reading of about 3.5 and close the gas supply valve.
- Wait about 15 seconds for the pressure to equalize and record this value on the Service Request Form under Can Pressurization Section: P_f (pressure final - *psig*).
- Close the canister valve and disconnect the digital compound gauge.
- Enter the readings on the Service Request form in LIMS. Also, record the initial and final pressure readings on the back on the sample identification tag and pressurization gas utilized, where necessary.

11.3.2 Humidified Zero Grade Air Pressurization

- Make sure the zero air tank regulator is open and reads positive pressure of at least 500 psig. If the pressure is under 500 psig then the tank will be replaced.
- Turn the gas supply valve open to ≤40psig and allow to flow for approximately 15 seconds.
- Zero the digital pressure gauge by pressing the Zero button (or with a small screwdriver for TIF gauges), if needed.
- Attach the tube fitting connector to the canister valve connector and tighten the fitting finger tight.
- Turn off the valve and ensure there is a good seal (i.e. the pressure does not drop).
- Open the valve on the canister and take a reading. If the value is negative and in in-Hg units, compare the reading observed to the chart located on the wall beside the tank and obtain the value in psig. Record the pressure (psig) on the Service Request Form under Can Pressurization Section: P_i (pressure initial - *psig*).
- Open the gas supply valve and allow the canister to fill to a reading of about 3.5 and close the gas supply valve.

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- Wait about 15 seconds for the pressure to equalize and record this value on the Service Request Form under Can Pressurization Section: P_f (pressure final - *psig*).
- Close the canister valve and disconnect the digital pressure gauge.
- Enter the readings on the Service Request form in LIMS. Also, record the initial and final pressure readings on the back on the sample identification tag and pressurization gas utilized, where necessary.

11.3.3 Nitrogen Pressurization

This pressurization gas is utilized at the request of a client. If this gas is used, a reference to the gas must be made on the SR form.

- Make sure the nitrogen tank regulator is open and reads positive pressure of at least 500 psig. If the pressure is under 500 psig then the tank will be replaced.
- Turn the gas supply valve open to ≤40psig and allow to flow for approximately 15 seconds.
- Zero the digital pressure gauge by pressing the Zero button (or with a small screwdriver for TIF gauges), if needed.
- Attach the tube fitting connector to the canister valve connector and tighten the fitting finger tight.
- Turn off the valve and ensure there is a good seal (i.e. the pressure does not drop).
- Open the valve on the canister and take a reading. If the value is negative and in in-Hg units, compare the reading observed to the chart located on the wall beside the tank and obtain the value in psig. Record the pressure (psig) on the Service Request Form under Can Pressurization Section: P_i (pressure initial - *psig*).
- Open the gas supply valve and allow the canister to fill to a reading of about 3.5 and close the gas supply valve.
- Wait about 15 seconds for the pressure to equalize and record this value on the Service Request Form under Can Pressurization Section: P_f (pressure final - *psig*).
- Close the canister valve and disconnect the digital pressure gauge.
- Enter the readings on the Service Request form in LIMS. Also, record the initial and final pressure readings on the back on the sample identification tag and pressurization gas utilized, where necessary.

11.4 Documentation

Each time the canister pressure/vacuum is evaluated/pressurized the reading and the gas utilized must be recorded. The same pressurization gas must be used each time. The sample identification tag should be used in conjunction with the LIMS form but not in lieu of this form.

Refer to Attachment B for the in-Hg to psig conversion chart.

1. Initial pressure/vacuum (as received) reading, P_i (in-Hg or psig) under "Initial Reading". All in-Hg readings must be converted to psig (refer to Section 12.4).
2. Initial pressure/vacuum (prior to pressurization); P_i (in-Hg or psig) under "Can Pressurization". Record the reading to two decimal places.

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3. Final pressure (following pressurization); P_f (psig) under "Can Pressurization"
4. Subsequent pressurization under "Can Pressurization, PF_2" on the SR form.
5. Pressurization gas utilized if EPA Method 3C and/or TO-15 methods are requested.
6. Sample tag – initial and final pressures, initials of employee and dates of readings; trip blank witness initials and date.

12) Quality Assurance / Quality Control Requirements

12.1 The difference between the initial (upon receipt) and the initial (upon pressurization) should be reviewed to ensure that there are no apparent canister leaks, however changes in temperature of the canister will cause small discrepancies in these two readings. The calculation may be found in Section 12.6.2.

12.2 Pressure/Vacuum Gauge Evaluation and Calibration

All of the gauges must be within calibration tolerances of the range of use; this shall be found on the certificate and kept on file with the QA department. In addition, each gauge shall be evaluated biannually in accordance with the *SOP for Calibration and Use of Laboratory Support Equipment.*

12.3 Balance Gas Certification

All balance gas tanks received by the laboratory shall be traceable to a certificate produced from the vendor. This certification must include, product, grade, cylinder number, batch number, QC type (batch or individual cylinder), and impurities. The laboratory must be able to provide evidence of traceability to a valid certificate whenever necessary.

12.3.1 Quality Check In addition, each cylinder utilized for canister pressurization must be quality checked prior to use. A cleaned/certified Summa canister shall be pressurized with the gas in question to at least 3.5psig and analyzed by TO-15 for target compounds *and* tentatively identified compounds. No results for any analyte may exceed 0.2ppbV for the cylinder to be acceptable for use.

12.3.2 Documentation Analytical documentation must include at least the Summa canister identification, type of gas being quality checked (helium, zero air), gas cylinder identification and pass/fail based on the requirements listed above. All analytical documentation and associated tag for each quality check must be kept on file.

Analytical Documentation:
- Lot number
- Cylinder number
- Gas type
- Initials of analyst
- Results
- Date of quality check acceptance determination
- Pass or fail

Tag:
- Date changed
- Lot number

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Cylinder number
QC date
Instrument
Initials and date
Pass or Fail*

*If the cylinder fails, it must be tagged with "*FAILED QC CHECK DO NOT USE*" and it should be sent back to the vendor.

12.4 Pressure/Vacuum Conversion

The vacuum readings entered on the Service Request will be converted to psig automatically. If needed, the conversion chart may be used to convert in-Hg (vacuum) to pounds per square inch (psig). The following is the formula used for this conversion (refer to Attachment B).

In-Hg * 0.491154 = psig

If this conversion is performed using a macro program, it must be validated and the documentation retained on file.

12.5 Service Request Form

All pressure/vacuum applicable evaluations including the balance gas utilized must be recorded on the Service Request form in LIMS. This documentation is imperative in providing information for ensuring that sampling occurred, the valve and/or canister are not leaking and confirming that the correct balance gas was used.

12.6 Initial Pressure/Vacuum Verification and Dilution Factors

12.6.1 Dilution Factors Dilution factors (DF) are calculated using the P*i* (initial pressure) and P_f (final pressure). Calculate the DFs using the following equation.

Dilution Factor (DF)

$$DF = \frac{14.7 + P_f}{P_i + 14.7}$$

where:	DF	Dilution factor, psia
	P_f	final pressure
	P_i	initial pressure

Note: If there are multiple pressurizations then multiply the two DF together.

If a canister was backfilled then the canister dilution factor is calculated using the following equation.

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$$DF = \frac{14.7 + P_f}{P_i + 14.7} x \frac{14.7 + P_i}{\left|P_i - P_{backfilled}\right|}$$

12.6.2 Initial Pressure/Vacuum Verification *Dilution factors for determining analytical concentrations shall be taken from the initial pressure reading taken at the time the sample canister is pressurized, not the reading recorded at login.* If the reading taken during the initial pressurization is substantially different from the reading taken during sample receipt, make an effort to determine the cause. See section 16.3. A significant difference would constitute ≥10% difference (see equation below) between the calculated pressure dilution factors (DFs) using the equation in Section 12.6.1 and both P*i* readings and the P_f following pressurization. Refer to Section 16.3 for corrective action.

Relative Percent Difference (RPD)

$$\frac{\left|DF_1 - DF_2\right|}{\left(\frac{DF_1 + DF_2}{2}\right)} x100$$

where: DF_1 First measurement value
DF_2 Second measurement value

13) Data Reduction and Reporting

13.1 Not Applicable

14) Method Performance

14.1 Not Applicable

15) Pollution Prevention and Waste Management

15.1 Not Applicable

16) Corrective Actions for Out-of-Control Data

16.1 This section is designed to provide a course of action for handling erroneous, out of control situations detailed in the preceding sections. Corrective actions include discrepancies at the initial pressure/vacuum evaluation, secondary pressure/vacuum check and pressurization as well as the possible causes. The *SOP for Nonconformance and Corrective Action* shall be followed, where applicable when determining course of action. *The client must be contacted and informed of the non-conformity, regardless of the information that is available at the time.*

It is important to determine if the deviation was due to a system problem within the laboratory, human error, etc. This is imperative to ensure continuous improvement within the laboratory quality system.

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16.2 Initial Pressure/Vacuum Does Not Meet Criteria

If the initial pressure/vacuum of a sample taken at sample receipt does not meet the guidelines detailed in Section 11.2.1 of this document the Project Manager must be contacted immediately. The reading shall be recorded as usual and the PM must contact the client for instructions on the fate of the sample.

In addition, the laboratory must determine the cause, whenever possible, which could be a digital compound gauge / flow controller problem, sampler error or a leaking canister or valve. All determinations must be fully documented and retained in the corresponding job file. The outcome of any conversation with the client regarding the sample(s) in question must be documented and retained in the file. Review the following as a means to determine the possible/probable root cause of the pressure/vacuum reading error and the course of action, which must be taken.

16.2.1 Flow Controller

The first step is to evaluate the flow controller calibration (if used). If a flow controller was used in the sampling process, the laboratory must determine the controller serial number used and review the calibration records. A post calibration shall be performed in accordance with the *SOP for Flow Controllers and Critical Orifices*. If it is determined that the flow controller is out of calibration tolerance, the reason for the deviation must be investigated. This includes the evaluation of both the fine and coarse adjustments located on the controller. The client must be contacted and told of any problem with the sample volume and all resolutions shall be filed in the associated job file. It may be necessary to qualify the data, if reported.

If the calibration is consistent, then the flow controller may be ruled out as a possible cause and Sections 16.2.2 and 16.2.3 must be considered.

16.2.2 Leaking Valve or Canister

It is unlikely but possible that the canister developed a leak after being shipped from the laboratory. If the valve was securely closed and the cap tightened after sampling, and a leak is suspected, the canister leak check documentation must be reviewed and another leak check may be performed following any analyses, where necessary. The leak check procedure is detailed in the *SOP for Cleaning and Certification of Summa Canisters and Other Specially Prepared Canisters*.

If the cap was not secured, a leaking or improperly closed valve is the most common problem. The Project Manager must be contacted in accordance with Section 16.2 above.

16.2.3 Sampler Error

Unless the sampling event is well documented in the field, it will be difficult to attribute a particular problem to sampler error. If the sampler does not record the canister pressure after sampling, the lab cannot know for certain what the reading should be upon receipt.

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The most common problem is that the valve may not have been completely closed (refer to Section 16.2.2). The client must be notified and the data for any analyses may need to be qualified.

16.3 Relative Percent Difference Does Not Meet Criteria

The basic procedure for the investigation process is described in Section 16.2. In addition, the gauges used to take the two readings (if different gauges were used) may be evaluated against each other to determine if a difference exists. Another problem may be an erroneous recording. In either case, another gauge shall be used to verify the actual reading. If there was a significant temperature change in the canister between the two readings the pressure will change. This change will not introduce error in the calculations because the dilution factor is calculated from the initial pressure reading taken at the time of pressurization. If the samples are logged in and pressurized in the SMO room there should be very little difference because the ambient temperature does not vary greatly, unless the canisters were not allowed to equilibrate to room temperature before checking initial pressures at login.

A full investigation must be initiated to determine the root cause. If it is determined that there was an error on the part of the laboratory (leaking valve or canister, flow controller error, etc.) a NCAR must be generated in accordance with the *SOP for Nonconformance and Corrective Action.* The client must be contacted immediately and all documentation retained on file including any training, system revisions, etc.

17) Contingencies for Handling Out-of-Control or Unacceptable Data

17.1 Not Applicable

18) Training

18.1 Laboratory personnel shall have read, understood and agreed to perform the procedure detailed in this document including all documentation requirements, investigations and corrective actions described herein. The training plan (Attachment A) shall be used to properly document this training, and will be retained on file.

19) Method Modifications

19.1 Not Applicable

20) Summary of Changes

20.1 SOP updated using ALS SOP Template – New cover page, document tracking/signature page, and table of contents page. Header/footer and font revised throughout SOP to align with ALS SOP template specifications. Additionally, sections were reorganized as outlined in the *SOP for Establishing Standard Operating Procedures* and section references updated as necessary. Section titled "Instrument Specific Addendum" removed (Section did not contain any information and had "Not Applicable" listed.

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Section 1	Renamed "Scope and Applicability"
Section 2	Renamed "Summary of Procedure"
Section 4	Previously Section 10
Section 5	Previously Section 4
Section 6	Previously Section 5
Section 7	Previously Section 6
Section 8	Previously Section 7
Section 9	Previously Section 8
Section 9.5	Reworded
Section 10	Previously Section 9
Section 11.2	Revised to specify that readings should be recorded to 2 decimal places
Section 11.2.1	Corrected the range listed
Section 11.3.1	3rd bullet - updated
Section 11.3.2	3rd bullet – updated
Section 11.3.3	3rd bullet – updated
Section 11.4	#2 – Added last sentence
Section 16.1	Updated SOP title
Section 16.3	Updated SOP title
Section 18	Previously Section 19 Renamed "Training"
Section 19	Previously Section 20
Section 20	Previously Section 22 Renamed "Summary of Changes"
Section 21	Previously Section 18
Section 21.1	Updated SOP title and ID
Section 22	Previously section 23

21) References

21.1 *SOP for Nonconformance and Corrective Action*; CE-QA008
21.2 *SOP for Calibration and Use of Laboratory Support Equipment*; ADM-SupEQ
21.3 *SOP for Sample Receiving, Acceptance, and Log-in*; SMO-SMPL_REC
21.4 *SOP for Cleaning and Certification of Summa Canisters and Other Specially Prepared Canisters*; SMO-Can_Cert
21.5 *SOP for Flow Controllers and Critical Orifices*; ADM-CNTRL

22) Attachments

22.1 Attachment A – Training Plan for Canister Pressurization
Attachment B – in-Hg to psig Conversion Chart
Attachment C – Pressurization Gas Flowchart

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Attachment A

Training Plan

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Training Plan for Canister Pressurization

Trainee _____ Trainer _____ Completion Date _____

1. Read SOP — Trainer ____ Trainee ____ Date _____

2. Demonstrated understanding of: — Trainer ____ Trainee ____ Date _____
   - Units
   - Equipment utilized
   - Equipment Requirements
     - Bubbler
     - Pressurization gas cylinders / certification
     - Gauges

3. Observe performance of SOP — Trainer ____ Trainee ____ Date _____
   - ___Pressurization Gas Guidelines
   - ___Contamination Prevention
   - ___Initial Pressure Verification Requirements
   - ___Conversions
   - ___LIMS and Tag

4. Perform SOP with supervision — Trainer ____ Trainee ____ Date _____
   - ___Pressurization Gas Guidelines
   - ___Contamination Prevention
   - ___Initial Pressure Verification Requirements
   - ___Conversions
   - ___LIMS and Tag

5. Independent performance of the SOP — Trainer ____ Trainee ____ Date _____
   - ___Pressurization Gas Guidelines
   - ___Contamination Prevention
   - ___Initial Pressure Verification Requirements
   - ___Conversions
   - ___LIMS and Tag

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Attachment B

In-Hg to psig Conversion Table

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in-Hg to psig Conversion Table

in-Hg	psig	in-Hg	psig	in-Hg	psig	in-Hg	psig	in-Hg	psig	in-Hg	psig
0.0	0.0	-5.0	-2.5	-10.0	-4.9	-15.0	-7.4	-20.0	-9.8	-25.0	-12.3
-0.1	0.0	-5.1	-2.5	-10.1	-5.0	-15.1	-7.4	-20.1	-9.9	-25.1	-12.3
-0.2	-0.1	-5.2	-2.6	-10.2	-5.0	-15.2	-7.5	-20.2	-9.9	-25.2	12.4
-0.3	-0.1	-5.3	-2.6	-10.3	-5.1	-15.3	-7.5	-20.3	-10.0	-25.3	-12.4
-0.4	-0.2	-5.4	-2.7	-10.4	-5.1	-15.4	-7.6	-20.4	-10.0	-25.4	-12.5
-0.5	-0.2	-5.5	-2.7	-10.5	-5.2	-15.5	-7.6	-20.5	-10.1	-25.5	-12.5
-0.6	-0.3	-5.6	-2.8	-10.6	-5.2	-15.6	-7.7	-20.6	-10.1	-25.6	-12.6
-0.7	-0.3	-5.7	-2.8	-10.7	-5.3	-15.7	-7.7	-20.7	-10.2	-25.7	-12.6
-0.8	-0.4	-5.8	-2.8	-10.8	-5.3	-15.8	-7.8	-20.8	-10.2	-25.8	-12.7
-0.9	-0.4	-5.9	-2.9	-10.9	-5.4	-15.9	-7.8	-20.9	-10.3	-25.9	-12.7
-1.0	-0.5	-6.0	-2.9	-11.0	-5.4	-16.0	-7.9	-21.0	-10.3	-26.0	-12.8
-1.1	-0.5	-6.1	-3.0	-11.1	-5.5	-16.1	-7.9	-21.1	-10.4	-26.1	-12.8
-1.2	-0.6	-6.2	-3.0	-11.2	-5.5	-16.2	-8.0	-21.2	-10.4	-26.2	-12.9
-1.3	-0.6	-6.3	-3.1	-11.3	-5.6	-16.3	-8.0	-21.3	-10.5	-26.3	-12.9
-1.4	-0.7	-6.4	-3.1	-11.4	-5.6	-16.4	-8.1	-21.4	-10.5	-26.4	-13.0
-1.5	-0.7	-6.5	-3.2	-11.5	-5.6	-16.5	-8.1	-21.5	-10.6	-26.5	-13.0
-1.6	-0.8	-6.6	-3.2	-11.6	-5.7	-16.6	-8.2	-21.6	-10.6	-26.6	-13.1
-1.7	-0.8	-6.7	-3.3	-11.7	-5.7	-16.7	-8.2	-21.7	-10.7	-26.7	-13.1
-1.8	-0.9	-6.8	-3.3	-11.8	-5.8	-16.8	-8.3	-21.8	-10.7	-26.8	-13.2
-1.9	-0.9	-6.9	-3.4	-11.9	-5.8	-16.9	-8.3	-21.9	-10.8	-26.9	-13.2
-2.0	-1.0	-7.0	-3.4	-12.0	-5.9	-17.0	-8.4	-22.0	-10.8	-27.0	-13.3
-2.1	-1.0	-7.1	-3.5	-12.1	-5.9	-17.1	-8.4	-22.1	-10.9	-27.1	-13.3
-2.2	-1.1	-7.2	-3.5	-12.2	-6.0	-17.2	-8.4	-22.2	-10.9	-27.2	-13.4
-2.3	-1.1	-7.3	-3.6	-12.3	-6.0	-17.3	-8.5	-22.3	-11.0	-27.3	-13.4
-2.4	-1.2	-7.4	-3.6	-12.4	-6.1	-17.4	-8.5	-22.4	-11.0	-27.4	-13.5
-2.5	-1.2	-7.5	-3.7	-12.5	-6.1	-17.5	-8.6	-22.5	-11.1	-27.5	-13.5
-2.6	-1.3	-7.6	-3.7	-12.6	-6.2	-17.6	-8.6	-22.6	-11.1	-27.6	-13.6
-2.7	-1.3	-7.7	-3.8	-12.7	-6.2	-17.7	-8.7	-22.7	-11.2	-27.7	-13.6
-2.8	-1.4	-7.8	-3.8	-12.8	-6.3	-17.8	-8.7	-22.8	-11.2	-27.8	-13.7
-2.9	-1.4	-7.9	-3.9	-12.9	-6.3	-17.9	-8.8	-22.9	-11.3	-27.9	-13.7
-3.0	-1.5	-8.0	-3.9	-13.0	-6.4	-18.0	-8.8	-23.0	-11.3	-28.0	-13.8
-3.1	-1.5	-8.1	-4.0	-13.1	-6.4	-18.1	-8.9	-23.1	-11.3	-28.1	-13.8
-3.2	-1.6	-8.2	-4.0	-13.2	-6.5	-18.2	-8.9	-23.2	-11.4	-28.2	-13.9
-3.3	-1.6	-8.3	-4.1	-13.3	-6.5	-18.3	-9.0	-23.3	-11.4	-28.3	-13.9
-3.4	-1.7	-8.4	-4.1	-13.4	-6.6	-18.4	-9.0	-23.4	-11.5	-28.4	-14.0
-3.5	-1.7	-8.5	-4.2	-13.5	-6.6	-18.5	-9.1	-23.5	-11.5	-28.5	-14.0
-3.6	-1.8	-8.6	-4.2	-13.6	-6.7	-18.6	-9.1	-23.6	-11.6	-28.6	-14.0
-3.7	-1.8	-8.7	-4.3	-13.7	-6.7	-18.7	-9.2	-23.7	-11.6	-28.7	-14.1
-3.8	-1.9	-8.8	-4.3	-13.8	-6.8	-18.8	-9.2	-23.8	-11.7	-28.8	-14.1
-3.9	-1.9	-8.9	-4.4	-13.9	-6.8	-18.9	-9.3	-23.9	-11.7	-28.9	-14.2
-4.0	-2.0	-9.0	-4.4	-14.0	-6.9	-19.0	-9.3	-24.0	-11.8	-29.0	-14.2
-4.1	-2.0	-9.1	-4.5	-14.1	-6.9	-19.1	-9.4	-24.1	-11.8	-29.1	-14.3
-4.2	-2.1	-9.2	-4.5	-14.2	-7.0	-19.2	-9.4	-24.2	-11.9	-29.2	-14.3
-4.3	-2.1	-9.3	-4.6	-14.3	-7.0	-19.3	-9.5	-24.3	-11.9	-29.3	-14.4
-4.4	-2.2	-9.4	-4.6	-14.4	-7.1	-19.4	-9.5	-24.4	-12.0	-29.4	-14.4
-4.5	-2.2	-9.5	-4.7	-14.5	-7.1	-19.5	-9.6	-24.5	-12.0	-29.5	-14.5
-4.6	-2.3	-9.6	-4.7	-14.6	-7.2	-19.6	-9.6	-24.6	-12.1	-29.6	-14.5
-4.7	-2.3	-9.7	-4.8	-14.7	-7.2	-19.7	-9.7	-24.7	-12.1	-29.7	-14.6
-4.8	-2.4	-9.8	-4.8	-14.8	-7.3	-19.8	-9.7	-24.8	-12.2	-29.8	-14.6
-4.9	-2.4	-9.9	-4.9	-14.9	-7.3	-19.9	-9.8	-24.9	-12.2	-29.9	-14.7

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Attachment C

Pressurization Gas Flowchart

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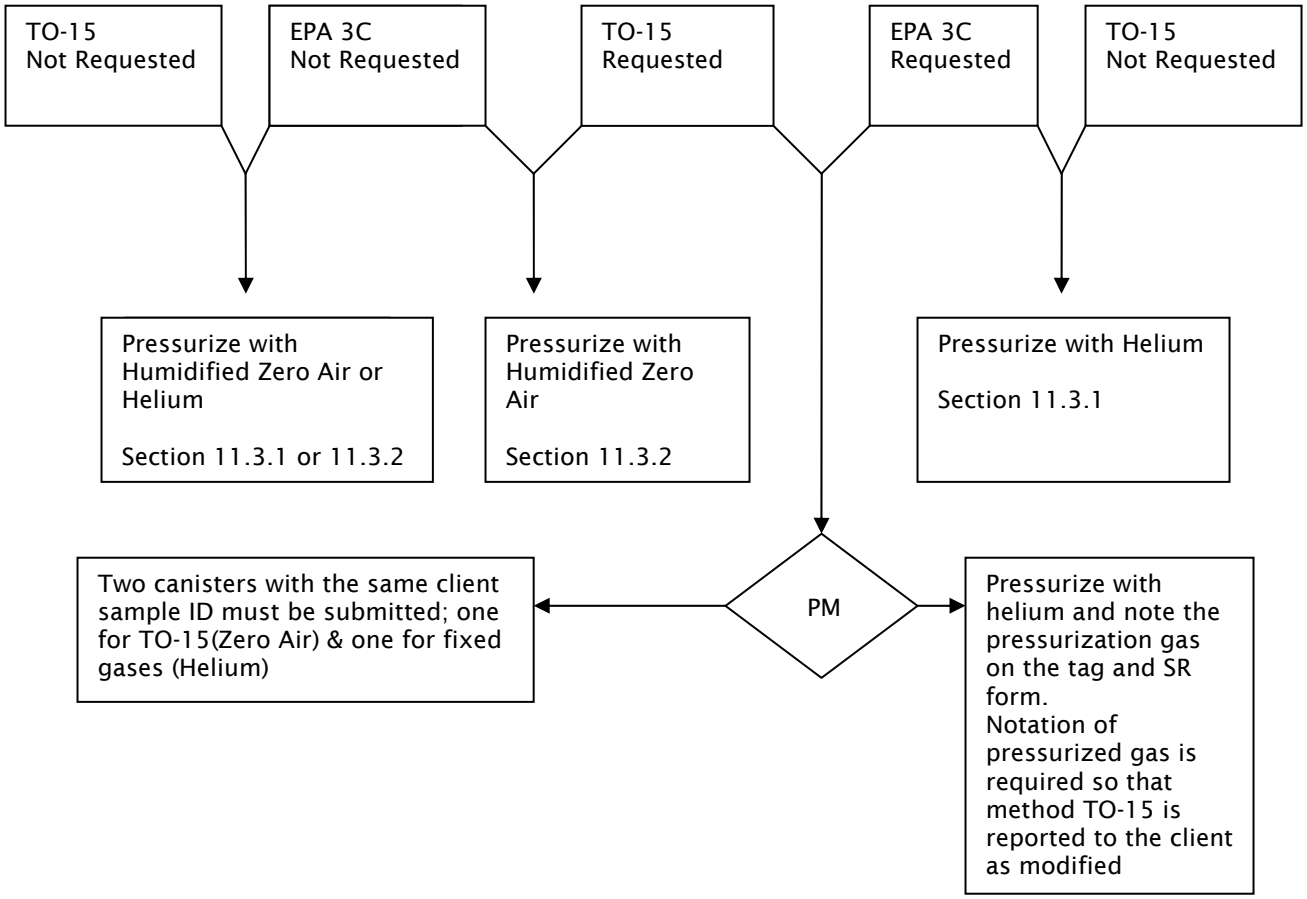
Pressurization Gas Flowchart



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APPENDIX F

ALS ENVIRONMENTAL

STANDARD OPERATING PROCEDURE FOR DETERMINATION OF VOLATILE ORGANIC COMPOUNDS IN AIR SAMPLES COLLECTED IN SPECIALLY PREPARED CANISTERS AND GAS COLLECTION BAGS BY GAS CHROMATOGRAPHY/MASS SPECTROMETRY (GC/MS)

DOCUMENT TITLE:	*DETERMINATION OF VOLATILE ORGANIC COMPOUNDS IN AIR SAMPLES COLLECTED IN SPECIALLY PREPARED CANISTERS AND GAS COLLECTION BAGS BY GAS CHROMATOGRAPHY/MASS SPECTROMETRY (GC/MS)*
REFERENCED METHOD:	*EPA TO-15*
SOP ID:	*VOA-TO15*
REV. NUMBER:	*20.0*
EFFECTIVE DATE:	*01/07/2013*

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DETERMINATION OF VOLATILE ORGANIC COMPOUNDS IN AIR SAMPLES COLLECTED IN SPECIALLY PREPARED CANISTERS AND GAS COLLECTION BAGS BY GAS CHROMATOGRAPHY/MASS SPECTROMETRY (GC/MS)

EPA TO-15

SOPID:	VOA-TO15	Rev. Number:	20.0	Effective Date:	01/07/2013

Approved By: Wida Ang — Date: 12/14/12
Team Leader (VOA GC/MS) - Wida Ang

Approved By: Chris Parnell — Date: 12/14/12
Technical Manager (VOA GC/MS) - Chris Parnell

Approved By: Chaney Humphrey — Date: 12/14/12
QA Manager - Chaney Humphrey

Approved By: Kelly M Horiuchi — Date: 12/17/12
Laboratory Director - Kelly Horiuchi

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DETERMINATION OF VOLATILE ORGANIC COMPOUNDS IN AIR SAMPLES COLLECTED IN SPECIALLY PREPARED CANISTERS AND GAS COLLECTION BAGS BY GAS CHROMATOGRAPHY/MASS SPECTROMETRY (GC/MS)

1) Scope and Applicability

1.1 This procedure is based on and incorporates the requirements detailed in EPA Compendium Methods TO-15 and TO-14A and is used to quantify a wide range of volatile organic compounds (VOCs) in gaseous matrices collected in gas collection bags (method modification) and specially prepared stainless steel canisters. This method typically applies to ambient concentrations of VOCs 0.50ug/m^3 (down to 0.10ug/m3 for low level ambient analyses) and above for the SCAN mode and 0.010ug/m^3 and above for the SIM mode; however, refer to Tables 3 and 3A for the specific laboratory initial calibration ranges for each target compound. The method requires VOC enrichment by concentrating up to one liter of a sample volume, with a virtually unlimited upper concentration range using dilutions from source level samples.

In this document, Tables 2 and 2A (see Note 1 below) list compounds that can be determined by this procedure along with their corresponding laboratory method reporting limits (MRLs) and method detection limits (MDLs). The reported MRL may be adjusted higher; however, the capability of achieving lower MRLs for specific project requirements must be thoroughly demonstrated (by an acceptable initial calibration and method reporting limit check standard) and documented as long as the MRL is higher than the current method detection limit for each compound. Additional compounds may be analyzed according to this procedure as described in the referenced methods as long as the requirements of this document are adhered to; however, if a compound is not listed in the TO-15 method, refer to Note 1 below. The number of samples that may be analyzed in a 24-hour period is about twenty. The number of sample results that may be reduced in an eight-hour day is approximately twenty.

Note 1: A number of compounds selected as target analytes are not included in the TO-15 Method; therefore the Florida Department of Health (FL DOH) required the laboratory to reference this Standard Operating Procedure (SOP) when reporting results for these analytes. However, the FL DOH disapproved of the SOP Code for this purpose, so CASS TO-15/GC-MS shall be referenced. Consequently, VOA-TO15 is otherwise known as CASS TO-15/GC-MS and shall be referenced, where necessary (on quantitation reports).

2) Summary of Procedure

2.1 The analytical method involves using a high-resolution gas chromatograph (GC) coupled to a mass spectrometer (MS). The GC/MS utilizes a linear quadrupole system, which allows for it to be operated by either continuously scanning a wide range of mass to charge ratios (SCAN mode) or by Select Ion Monitoring mode (SIM), which consists of monitoring a small number of ions from a specified compound list.

An aliquot of an air sample is concentrated on a solid adsorbent trap (either cryogenically or fan cooled glass beads or stronger adsorbents at higher temperatures) to collect the analytes of interest. To remove co-collected water vapor, the concentrated sample then goes through a water removal (dry purge) step. After the sample is pre-concentrated on a trap, the trap is heated and the VOCs are thermally desorbed onto a

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refocusing cold trap. The VOCs are then thermally desorbed onto the head of a capillary column once the cold trap is heated. The oven temperature (programmed) increases and the VOCs elute and are detected by the mass spectrometer.

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 method utilizes the internal standard calibration technique; refer to Section 3.16 for a complete definition.

3) Definitions

3.1 Cryogen A refrigerant used to obtain sub-ambient temperatures in the VOC concentrator and/or on front of the analytical column. Liquid nitrogen (cryogen) is used for this purpose and it has a boiling point of -195.8˚C.

3.2 Gauge Pressure Pressure measure with reference to the surrounding atmospheric (barometric) pressure, usually expressed in units of psig. Zero gauge pressure is equal to atmospheric pressure.

3.3 MS-SCAN Mass spectrometric mode of operation in which the gas chromatograph (GC) is coupled to a mass spectrometer (MS) programmed to SCAN all ions repeatedly over a specified mass range.

3.4 MS-SIM Mass spectrometric mode of operation in which the GC is coupled to a MS that is programmed to scan a selected number of ions repeatedly [i.e., selected ion monitoring (SIM) mode].

3.5 Analytical Sequence The analytical sequence describes exactly how the field and QC samples in an analytical batch are to be analyzed.

3.6 Neat Stock Standard A purchased, single component assayed reference material having a stated purity used to prepare working calibration standards.

3.7 Stock Standards Solution A concentrated solution of one or more target analytes at a known concentration purchased from a reputable commercial vendor. Stock standard solutions are used to prepare working calibration standards.

3.8 Intermediate Calibration Standard A solution of one or more target analytes at a known concentration prepared either from one or more neat stock standards or from one or more stock standards solutions.

3.9 Working Calibration Standard A solution of all the target analytes at a known concentration prepared either from one or more intermediate calibration standards and/or from one or more stock standard solutions.

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3.10 Calibration or Standard Curve A calibration or standard curve is a graph which plots the concentration of a compound (or an analyte) versus the instrument response to the compound.

3.11 Initial Calibration Verification (ICV) Standard A solution prepared in the laboratory containing known concentration(s) of analytes of interest. The solution is prepared from neat stock standards and/or stock standards solutions which are from a different source than the standards used to prepare the working calibration standards.

3.12 Continuing Calibration Verification (CCV) Standard A working calibration standard which is analyzed at specific intervals in order to verify that the instrument continues to meet the calibration criteria.

3.13 Field Sample A sample collected and delivered to the laboratory for analysis.

3.14 Manual Integration This term applies to a data file in which setpoints have been changed and reintegration has occurred under the changed setpoints; baselines have been adjusted; peak integration start and stop "ticks" have been changed; peak area, or peak height, are changed after the time of data collection and data file generation.

3.15 Batch Quality Control (QC) Batch QC refers to the QC samples that are analyzed in an analytical batch of field samples and includes the Method Blank (MB), Laboratory Control Sample (LCS) and Laboratory Duplicate (LD).

3.16 Internal Standard Calibration Compares the instrument responses from the target compound in the sample to the responses of specific standards (called internal standards), which are added to the sample or sample preparation prior to analysis. The ratio of the peak area (or height) of the target compound in the sample or sample preparation is compared to a similar ratio derived for each calibration standard.

3.17 May This action, activity, or procedural step is neither required nor prohibited.

3.18 Must This action, activity, or procedural step is required.

3.19 Shall This action, activity, or procedural step is required.

3.20 Should This action, activity, or procedural step is suggested, but not required.

3.21 SOP Standard Operating Procedure

3.22 Service Request A form generated, at the time of sample receipt, which details pertinent information such as client name, address, contact, client and laboratory sample identifications, sampling and receipt dates and times, requested analyses, sample type, canister pressures (initial and final), and the service request number (unique number for each submitted job) and serves as an inter-laboratory "custody" form which accompanies all samples throughout the laboratory.

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3.23 Selectivity Selectivity of a method refers to the extent to which it can determine particular analyte(s) in a complex mixture without interference from other components in a mixture. Another definition is the extent to which a particular method can be used to determine analytes under given conditions in the presence of other components of similar behavior.

3.24 Limit of Detection (LOD) The smallest amount or concentration of a substance that must be present in a sample in order to be detected at a high level of confidence (99%). At the LOD, the false negative rate (Type II error) is 1%. (DoD Clarification). For consistency purposes, the LOD may be referred to as the MDL once it is reported; however, full verification will be on file in the laboratory per the procedures detailed in this document.

3.25 Limit of Quantitation (LOQ) The lowest concentration that produces a quantitative result within specified limits of precision and bias. For DoD projects, the LOQ shall be set at or above the concentration of the lowest initial calibration standard. (DoD Clarification). For consistency purposes and since the LOQ and MRL are equivalent with regards to laboratory procedure, the LOQ will be referred to as the MRL in this document and once it is reported. Full verification will be on file in the laboratory per the procedures detailed in the document.

3.26 Detection Limit (DL) / Method Detection Limit (MDL) The smallest analyte concentration that can be demonstrated to be different from zero or a blank concentration at the 99% level of confidence. At the DL, the false positive rate (Type 1 error) is 1%. (DoD Clarification). For consistency purposes, the DL may be referred to as MDL. Also, as far as reporting is concerned the MDL will be raised up (where necessary) to the verified LOD per the procedures defined in this document and reported accordingly.

4) Responsibilities

4.1 It is the responsibility of the analyst to perform the analysis according to this SOP and to complete all documentation required for data review. Personnel in the laboratory who have demonstrated the ability to generate acceptable results utilizing this SOP may perform analysis, interpretation and peer review of the results. Data reduction and/or peer review may be performed by another qualified employee. This employee must be familiar with the analytical technique and have completed a data review training plan to ensure familiarity with specific analysis and requirements.

The supervisor/manager must also ensure that method proficiency is documented initially and whenever significant changes in the instrument type, personnel, and matrix or test method are made. The department supervisor/manager or designee shall perform final review and sign-off of the data.

5) Interferences

5.1 Summa Canisters

Canisters shall be stored in a contaminant free location and shall be capped tightly during shipment to prevent leakage and minimize any compromise of the sample. The pressure/vacuum is checked prior to shipment and upon receipt from the field. Any problems with the sample from the field are noted and the Project Manager contacted.

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Also, canisters must be cleaned and certified to be free from target analytes before being shipped to the field for sample collection. The procedure is described in detail in the *SOP for Cleaning and Certification of Summa Canister and Other Specially Prepared Canisters* (refer to this procedure as well as Section 12.5 for the acceptance criteria).

Current laboratory practice entails the segregation of 6L canisters into ambient (low) level and source levels. All the ambient canisters are used for low level (indoor air, ambient air) projects and not intentionally for soil gas, SVE monitoring, or other higher level applications. It may be necessary to "retire" an ambient canister and re-assign for source level use if high concentrations are encountered. This decision will be made by management based on analytical concentrations and what compounds were encountered at these levels. If the level of any analyte is detected above 5,000ug/m*3* in the ambient can, then the supervisor/team leader must be contacted to determine if the canister(s) is to be retired. If retirement is decided upon, make a notation on the sample tag (or other color coded tag) of each canister in question. The notation must contain the analyte, threshold levels and retirement from ambient use (initial and date notation) so that the canister conditioning/management department may properly execute the retirement.

5.2 Analytical System

The analytical system must be demonstrated to be free from contamination under the conditions of the analysis by running humidified zero air blanks. The use of non-chromatographic grade stainless steel tubing, non-PTFE thread sealants, or flow controllers with buna-N rubber components must be avoided.

5.3 Carbon Dioxide

Excessive levels of carbon dioxide present in a sample may interfere with analysis by freezing up the cryogenic trap. A smaller aliquot must be analyzed to eliminate this problem, or the sample should be analyzed using the higher temperature multi-adsorbent trapping technique which allows carbon dioxide to pass.

5.4 Gas Collection Bags

This procedure covers the use of gas collection vessels such as Tedlar® or Mylar® bags. However, due to the nature of these types of bags it is not recommended that clients use this option for ambient air samples. Sample collection bags made out of ®Tedlar have contaminants that are inherent to the manufacturing process. The two main contaminants are phenol and N,N-Dimethylacetamide. However, this only becomes a problem when the concentration levels in the sample are low ppbv such as ambient air monitoring samples where more of the sample usually has to be concentrated and analyzed. To minimize the loss of sample integrity, a 72-hour hold time has been incorporated into the procedure.

5.5 Glassware

Interferences caused by contaminants in solvents, reagents, glassware, and other sample processing hardware results in discrete artifacts and/or elevated baselines in the detector profiles should be minimized. All glassware associated with this method must be scrupulously cleaned to avoid possible contamination. The cleaning shall be

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performed in accordance with the procedure outlined in the *SOP for Glassware Cleaning.* The use of high purity water, reagents, and solvents helps to minimize these problems.

6) Safety

6.1 Each compound, mixture of compounds, standards, and surrogates, as well as samples, should be treated as a potential health hazard. Exposure to these chemicals should be reduced to the lowest level possible through the use of gloves (to minimize absorption through the skin) and hoods (to minimize inhalation). Refer to the laboratory's Environmental, Health and Safety Manual as it makes reference to the safe handling of chemicals, MSDS location, and the laboratory waste management plan for the safe disposal of chemicals and samples.

6.2 Material Safety Data Sheets (MSDS)

The analyst should consult MSDS for compounds being handled in the course of this procedure, and be familiar with proper safety precautions to be followed when handling hazardous chemicals. Care should be taken when handling standard material in a neat or highly concentrated form.

6.3 Liquid Nitrogen

Liquid nitrogen can cause serious tissue damage (frostbite) with only a few seconds of contact. The valves on the cryogen dewars should be opened slowly so leaky fittings can be identified. Neoprene or leather gloves should be worn when turning valves and handling tubing and fittings that have been in contact with the cryogen.

6.4 Protective Clothing

Personal protective clothing (safety glasses, gloves and lab coat) are required when preparing standards and handling standard material in neat form.

6.5 Pressurized Gases

The use of pressurized gases is required for this procedure. Care should be taken when moving cylinders. All gas cylinders must be secured to a wall or an immovable counter with a chain or a cylinder clamp when not in use. The regulator should never remain on small "D" size cylinders following use. Sources of flammable gases (i.e. pressurized hydrogen) should be clearly labeled.

6.6 Syringes

The proper use of syringes should be part of employee training for this SOP. Care should be taken to avoid personal injury as a result or improper handling techniques.

7) Sample Collection, Containers, Preservation, and Storage

7.1 Air samples are collected in the field and delivered to the laboratory and shall be collected in either a specially prepared, leak-free, stainless steel pressure vessel (with valve) of desired volume (e.g., 6L) or a sample collection bag (Tedlar). Canister samples may either be grab or time integrated (using a variable flow controller, refer to the *SOP*

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for Flow Controllers and Critical Orifices) utilizing the canister vacuum to draw the sample. Bags require the use of an upstream pump or a "lung machine."

7.2 There are no special preservation requirements for either canisters or bags. However, bags should be stored in an environment free from puncture or deterioration sources (by hanging them from clips), labeled with the specific service request number, in accordance with the *SOP for Laboratory Storage, Analysis and Tracking*. Canisters should be stored on the appropriate shelves until they are to be analyzed.

7.3 Sample collection bags must be analyzed within 72 hours from the confirmed time of sampling. Samples received by the laboratory shall be analyzed within 30 days of sampling or sooner if project specific requirements dictate. Programs, which have shorter recommended or required hold times, include the Department of Toxic Substances Control (DTSC), which advises a 72 hour hold time. The Minnesota Pollutions Control Agency (MPCA) and EPA Region 9 both require a 14 days hold time. Additionally, the MPCA does not allow the use of Tedlar bags for sampling or sample dilution. The DTSC requirement is an advisory notice, but the laboratory shall make every effort to comply. However, the following statement shall be added to each report where sample analyses do not meet the 72 hour hold time and the client project is intended to comply with DTSC requirements. *"The recommended 72-hour hold time for the analysis of TO-15 was exceeded per the DTSC and LARWQCB Advisory – Active Soil Gas Investigations document dated January 28, 2003; however, this specific hold time statement is advisory and not considered as regulation. In addition, the samples were analyzed within the EPA Method TO-15 stated requirement of 30 days."*

8) Apparatus and Equipment

8.1 Additional instruments and/or differing models may be utilized as long as they are equivalent and meet the minimum requirements of this document.

8.2 Gas Chromatograph (GC)

An instrument capable of temperature programming, with a column oven that may be cooled to sub-ambient temperature at the start of the gas chromatographic run to result in the resolution of the VOCs.

Hewlett Packard 5890 Series II Plus
Hewlett Packard 6890 Series
Hewlett Packard 6890A Series
Agilent 6890N Series
Agilent 7890A Series

8.3 Autosampler

Tekmar-Dohrmann AUTOCan Autosampler:	14-ACAN-074
Concentrating Trap (cryogenic trap, built-in):	14-6938-020
Cryofocusing Module w/split valve:	14-6520-A00
GAST Vacuum Pump:	DOA-P104-AA or equivalent

8.4 Mass Spectrometer (MS)

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A MS capable of scanning from 34 to 350 amu every second or less, using 70 volts (nominal) electron energy in the electron impact ionization mode. The mass spectrometer must be capable of producing a mass spectrum for Bromofluorobenzene (BFB) which meets all of the criteria when 50ng or less of BFB is injected onto the GC/MS system.

Hewlett Packard 5972 Series
Hewlett Packard 5973 Series
Agilent 5973N
Agilent 5973 *inert*
Agilent 5975B *inert*
Agilent 5975C *inert*

8.4.1 Ionization Gauge Controller

- Agilent: 59864B
- Granville-Phillips 330 Ionization Gauge Controller: 330001/2/3
- Hewlett Packard Ionization Gauge Controller: 59864B

8.5 Analytical Column

Any analytical column capable of separating the compounds of interest may be used. The capillary column should be directly coupled to the source of the mass spectrometer. The following are suggested columns; an alternative column may be used as long as sufficient peak resolution and separation is achieved.

- Restek Rxi-1ms Fused Silica Capillary Column; 30m x 0.25mm ID 1.0µm film thickness

  OR

- Restek Rxi-1ms Fused Silica Capillary Column; 60m x 0.25mm ID 1.0µm film thickness

8.6 Data Systems

IBM-compatible PC with Windows 95/98/NT/XP/7 (Microsoft Office EXCEL version 2003 or newer) and Hewlett Packard Chemstation software including EnviroQuant with Extracted Ion Current Profile (EICP), National Institute of Standards and Technology (NIST) library (2002 version or newer) or equivalent.

8.7 Canister Pressurization Station

Vacuum/Pressure Gauge [0 to –30 inHg; 0-90 or 100 psig]

8.8 Canister Sampling Devices

Refer to the *SOP for Flow Controllers and Critical Orifices* for specific calibration and other pertinent information.

- VICI Condyne Model 300 Flow Controller

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- Critical Orifices (Laboratory manufactured)

8.9 Gas Collection Devices

- Lab Commerce, Aerosphere Model S6L, 6.0L Summa Passivated Canisters or equivalent
- Lab Commerce, Stabilizer Model 22.4L, 2.4L Canisters or equivalent
- Restek Corporation, #24203, 3.0L Silco Canisters or equivalent
- Tedlar bags – 0.5L, 1L, 3L, 5L, 10L, 25L, and 40L (other sizes are available; however, the volumes that are listed encompass the majority of the bags supplied and the samples submitted to the laboratory).

8.10 Dynamic Dilution System

- Entech Dynamic Diluter Model 4620A
- Toshiba laptop computer Model 2210CDT/6.0 and Software NT460

9) Standards, Reagents, and Consumable Materials

9.1 Reagents and Equipment

9.1.1 UHP Grade Helium (99.999%) (GC carrier gas, preconcentrator purge/sweep gas, pressurization gas)
9.1.2 Cryogen - Liquid nitrogen from bulk tank or 50 psig dewars (used to cool preconcentrator traps)
9.1.3 UHP/Zero Grade Air (canister pressurization)
9.1.4 ASTM Type II Water, DI water or equivalent
9.1.5 UHP Grade Nitrogen (99.999%) (additional pressurization gas, based on other methods requested – modification to method)

9.2 Standards

Standards are prepared for both SCAN and Selective Ion Monitoring (SIM) modes according to the procedures detailed in this section. The preparation of standards for the analysis of air samples is carried out by following the procedure, "Preparation of Gas Phase Standards for Ambient Air Analysis", Application Note, Spring 96, Vol. 6.5, *Tekmar*-DOHRMANN AutoCan Users Manual. Neat standards that are used for making trace gas standards must be of high purity; generally a purity of 98 percent or better is commercially available.

9.2.1 Instrument Performance Check, Internal Standard and Surrogate Spiking Mixture
Prepare a standard solution of p-Bromofluorobenzene (BFB-used as both a tune check and surrogate compound), bromochloromethane, chlorobenzene-d5, and 1,4-difluorobenzene, 1,2-dichloroethane-d4(surrogate), and toluene-d8(surrogate) at 500µg/m^3 each in humidified zero air (Section 9.2.1.2). Prepare this standard according to the procedure outlined in Volume 6.5 of the *Tekmar*-DOHRMANN Application Note. This standard may also be prepared from a neat cocktail as in Section 9.2.2.2.1 or as stated in Section 9.2.1.3.

9.2.1.1 An intermediate standard is prepared from neat compounds in a glass static dilution bottle (SDB). After the volume of the SDB is determined, calculate the mass of each compound to be spiked to achieve a final concentration of 5.0µg/mL. Then use the density of each neat compound

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to calculate the microliter amount to be spiked into the SDB. The SDB is then heated for a minimum of one hour at ~60°C to completely volatilize all components.

Concentration of the intermediate standard prepared in a SDB is 5.0µg/mL. The amount required to achieve this concentration is determined through the use of the following equation.

$$A = \frac{(C)(V)}{D} \qquad \text{(Equation 1)}$$

Where:

A Amount of each compound required to achieve the desired concentration of the standard in the SDB (µL)
C Desired concentration of SDB (µg/mL)
V Actual volume of the SDB (mL)
D Density of the compound in question (µg/µL)

Example:

Calculate the amount of neat bromochloromethane needed to achieve the final concentration of 5.0µg/mL of that compound in the SDB.

V = 2010mL
D = 1934.4µg/µL
C = 5.0µg/mL

$$A = \frac{\left(5.0\frac{\mu g}{mL}\right)2010mL}{1934.4\frac{\mu g}{\mu L}} = 5.2\mu L$$

Density (µg/µL)	Compound
1934.4	Bromochloromethane
1170.1	1,4-Difluorobenzene
1157	Chlorobenzene-d5
1307	1,2-Dichloroethane-d4
943	Toluene-d8
1593	BFB

9.2.1.2 The Working standard is prepared in a Summa canister by spiking an aliquot of the stock SDB standard (Section 9.2.1.1) using a heated gastight syringe. Connect a cleaned, evacuated Summa canister to a source of pure diluent gas (humidified zero air) using a Teflon line with a stainless steel tee directly above the canister valve. One port of the tee is fitted with a septum. Spike the SDB stock and following removal of syringe a small flow of diluent gas to flush the spike into the can.

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Pressurize the can to positive 83.3 psig with humid zero air, and allow the contents to equilibrate for approximately 24 hours before using.

Concentration of the working standard prepared in a Summa canister is 500ng/L. The final pressure of the canister is 83.3psig; therefore, the pressurized volume is 40L, which is obtained through the use of the following equation.

PV = PDF(V) (Equation 2)

Where:

PV Pressurized canister volume (L)

PDF Pressure Dilution Factor, where PF = $\frac{P_{atm} + P_f}{P_{atm} + P_i}$

P_f Final Canister Pressure

P_i Initial Canister Pressure

V Volume of canister at 1atm

P_{atm} Atmospheric Pressure = 14.7psig

Example:

$$\frac{14.7 + 83.3}{14.7 + 0}(6L) = 40L$$

In order to prepare the canister with a concentration of 500ng/L, it must be determined how much of the intermediate standard is required. This is achieved through the use of the following equation.

$$A = \frac{(F)(V)}{(C)\left(1000\frac{ng}{\mu g}\right)} \quad \text{(Equation 3)}$$

Where:

F Desired concentration of working standard (ng/L)

V Pressurized Volume of Canister (L)

C Concentration of prepared SDB (µg/mL)

A Amount of standard (mL) of the SDB required to obtain the desired working standard concentration

Example:

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$$A = \frac{500\frac{ng}{L}(40L)}{\left(5.0\frac{\mu g}{mL}\right)\left(1000\frac{ng}{\mu g}\right)} = 4mL$$

9.2.1.3 Currently the working standard is purchased in a cylinder at a certified concentration of 500ng/L (prepared by Spectra Gases Inc, Alpha, NJ).

9.2.1.3.1 For SCAN analyses, the working standard is filled directly into a summa canister to a pressure of 70 to 80 psig.

9.2.1.3.2 For SIM analyses, the working standard is diluted and pressurized with humid zero air to the desired concentration using Equation 2 in Section 8.2.1.2. Typical concentrations will be 20ng/L, 40ng/L or 50ng/L.

9.2.2 Initial Calibration (ICAL) Standard Prepare the primary source calibration standards in Summa canisters with nominal concentrations of 4ng/L (optional), 20ng/L and 200ng/L for analyses in SCAN mode and 0.1ng/L and 5.0ng/L, 20ng/L for analyses in Selective Ion Monitoring (SIM) mode for each of the target analytes. Differing injection volumes will create the standard concentrations listed in Tables 3 (SCAN) and 3A (SIM) of this document. The full list of analytes which are analyzed according to this method can also be found in Tables 2 (SCAN) and 2A (SIM).

Standards are prepared by diluting the stock standard with humid zero air into a Summa canister. The stock standard is a certified custom-blended cylinder (prepared by Spectra Gases Inc, Alpha, NJ). Refer to Tables 3 and 3A for the list of analytes and certified concentrations in the purchased cylinder.

9.2.2.1 Working standards are prepared into Summa canisters using the Entech Dynamic Diluter. Turn on the power to the diluter one hour prior to using to allow for the components to come to thermal equilibrium. Connect the computer and start the software. Connect a Zero Air source to the humidification chamber (flow controller #1). Connect stock standard cylinder#1 to flow controller #2 inlet. Open the cylinder valves. Adjust the inlet pressures to 50 to 60psig.

Standard Concentration Selection: The concentration of the two working standards prepared in Summa canisters should be 200ng/L and 20ng/L or 200ng/L, 20ng/L and 4ng/L (depending on the dynamic range of the initial calibration-include 4ng/L if a 0.1ng on column standard is desired or this standard may be used for the 0.5ng/L concentration as well) for SCAN and 0.1ng/L and 5.0ng/L, 20ng/L for SIM.

Position 1 – Total Air Flow (Zero Air)
Position 2 – Standard Flow (Purchased Standard One)
Position 3 – Standard Flow (Purchased Standard Two if Applicable)
Position 4 – Total Air Flow (Zero Air) (utilized if preparing a two dilution standard)

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Position 5 – Diluted Standard Flow (utilized if preparing a two dilution standard)

Step1: Determine the required flow rate of the stock standards (positions #2 and #3). The range must be from 5 to 50sccm (standard cubic centimeters per minute, same as ml/min). The flows listed below are guidelines to be used for the default standard flow (based on the desired standard concentration) and were chosen based on the ultimate final dilution required and limitations of the Dynamic Diluter (flows must be from 150 to 2000ml/min.).

Desired Standard Conc.	Default Standard Flow
200ng/L	50ml/min
100ng/L	50ml/min
20ng/L	20ml/min
5.0ng/L	10ml/min
4.0ng/L	8ml/min
0.1ng/L	10ml/min (See Note 1 below)

Note 1: For the 0.1ng/L standard (or another two dilution standard), a slightly different procedure is performed. In order to prepare this standard, two dilutions must be performed. Unscrew the cover of the dilutor and connect the first mass flow controller as well as the tubing to re-route the first dilution output from the final standard Summa canister to the 2nd dilution chamber. Refer to example 2 for the calculation guidelines to prepare a two dilution standard.

Example 1: Prepare a 200ng/L working standard. The concentration of each stock standard is 1000ng/L.

Step 2: Determine the required dilution factor for each stock.
Dilution factor = Stock Conc. (ng/L) / Desired Standard Conc. (ng/L)
Dilution Factor = 1000ng/L / 200ng/L = 5

Step 3: Calculate Total Flow
Total Flow= (stock std. flow-see table above)*(Dilution Factor)
Total Flow=50ml/min*5 = 250ml/min

Step 4: Calculate Diluent Air Flow
Air Flow=Total Flow-(Sum of stock std. flows-purchased cylinders)
Air Flow=250ml/min-(50+50)ml/min = 150ml/min

Example 2: Prepare a 0.1ng/L working standard. The concentration of each stock standard is 1000ng/L.

Step 2: Determine the required total dilution factor for the 0.1ng/L standard.
Dilution factor = Stock Conc. (ng/L) / Desired Standard Conc. (ng/L)
Dilution Factor = 1000ng/L / 0.1ng/L = 10,000

The two dilutions must be performed which total the dilution factor calculated above. Since the flow for the Diluter is restricted to a

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maximum of 2000ml/min, the total flow (as calculated in Step 3 below) cannot exceed 2000ml/min; therefore, the dilutions must be chosen accordingly.

Step 3: Calculate Total Flow
Total Flow = (stock std. flow-see table above)*(Dilution Factor)
Total Flow (Dilution 1) = 10ml/min*200 = 2000ml/min

For the 2nd dilution take the stock standard flow selected for dilution 1 for the two purchased cylinders (10ml/min each based on the desired final concentration) and add them together (10ml/min + 10ml/min for 20ml/min) to get the stock standard flow for the 2nd dilution.

2nd Dilution Factor Needed = Total Dilution/1st Dilution
2nd Dilution Factor = 10000/200(1st dilution) = 50
Total Flow (Dilution 2) = 20ml/min*50 = 1000ml/min

Step 4: Calculate Diluent Air Flow
Air Flow=Total Flow-(Sum of stock std. flows-purchased cylinders)
Air Flow=2000ml/min-(10+10)ml/min = 1980ml/min (Dilution 1)
Air Flow=1000ml/min-20ml/min = 980ml/min (Dilution 2)

Position 1 = 1980ml/min
Position 2 = 10ml/min
Position 3 = 10ml/min
Position 4 = 980ml/min
Position 5 = 20ml/min

Step 5: Enter flow rates in the appropriate fields in the Entech software. Start flows by clicking the "GO" button in the top right of the window. Allow flows to equilibrate for at least fifteen minutes, then attach an empty canister to the outlet port and open the valve. The outlet pressure will be displayed in the lower right of the window, in units of psia. Close the canister valve when the pressure reaches 30psia. There is a relief valve on the diluter that will open when the pressure reaches 35psia, so the canister will still be usable if the valve is not closed in time.

9.2.2.2 When analysis of additional (extra) compounds are requested which are not in the purchased stock cylinders, the following preparation instructions should be used. In addition, the internal standard / surrogate standard may also be prepared in this manner (Sections 9.2.2.2.1 – 9.2.2.2.2) as mentioned in Section 9.2.1.

9.2.2.2.1 *Equi-mass "soup"* (contains compounds in equal mass amounts) or *cocktail* prepared from the neat compounds for a large number of components. If additional SIM compounds are requested, the same cocktail may be used.

Cocktail Preparation:
Step 1: This cocktail is prepared by combining 25mg of each neat compound into a small glass vial. Use a microliter syringe to transfer each compound, cleaning with solvents in between.

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Put the vial in the freezer between aliquots to minimize volatilization. Take the density of each compound into account to determine the actual amount of each compound to spike into the cocktail by using the following equation.

$$S = \frac{A}{D} \qquad \text{(Equation 4)}$$

Where:
S Actual spike amount (µL)
A Desired amount for each compound (mg)
D Density (mg/µL); refer to Table 2 for the density

Example: The actual volume of acrolein to add to the cocktail is calculated by the following.

$$S(\text{Acrolein}) = \frac{25mg}{\left(0.840\frac{mg}{\mu l}\right)} = 29.8\mu L$$

Step 2: The concentration of each compound in the cocktail is determined by the following equation.

$$C = \frac{A}{V}\left(1000\frac{\mu g}{mg}\right) \qquad \text{(Equation 5)}$$

Where:

C Concentration of cocktail (µg/µL)
A Amount of each compound (mg)
V Final volume of cocktail (total spike volumes of each compound) (µL)

Example:

$$C = \frac{25mg}{631.8\mu L}\left(1000\frac{\mu g}{mg}\right) = 39.569\mu g/\mu L$$

8.2.2.2.2 *An intermediate standard* is prepared from neat compounds by spiking individual compounds into a glass static dilution bottle (SDB) as described in Section 9.2.1.1 or spiking an aliquot of a cocktail into the SDB. The spike amount of a cocktail is determined by using the following equation.

$$S = \frac{C_1 V}{C_2} \qquad \text{(Equation 6)}$$

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Where:

S Spike amount required in order to obtain the desired concentration (µL)

C_1 Desired concentration of SDB (µg/mL)

C_2 Concentration of cocktail (µg/µL)

V Volume of SDB (L)

Example: Determine the spike amount of the cocktail required to achieve the desired intermediate standard concentration.

$$S = \frac{\left(1\frac{\mu g}{ml}\right)(2010ml)}{27.81\frac{\mu g}{\mu L}} = 72.28\mu L$$

9.2.2.2.3 *Intermediate Standard Preparation (Gaseous Compounds)* As an alternative to the glass SDB method, if the extra compounds needed to be analyzed are gases at room temperature, use a gastight syringe to prepare an intermediate standard in a 1L Tedlar bag filled with humidified zero-grade air. Use the molecular weight of the compound to calculate the microliter amount to be spiked into the bag to achieve desired concentration. The spike amount is determined by using the following equation.

$$S = \frac{C * V * 24.46}{M * \left(1000\frac{ng}{\mu l}\right)}$$

S Spike amount required in order to obtain the desired concentration (µl)

C Desired concentration (ng/L)

V Volume of the Tedlar Bag (1L)

M Molecular Weight of the compound

24.46 Molar Volume of gas at 25°C, 1atm

Example:

Make a 100,000ng/L intermediate standard of Chloro-difluoromethane (Freon22) in a Tedlar Bag, where M=86

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$$S = \frac{100{,}000\frac{ng}{L} * 1L * 24.46}{86 * \left(1000\frac{ng}{\mu l}\right)} = 28.44\mu l$$

9.2.2.2.4 *The Working standard* for extra compounds is prepared in a Summa canister by spiking an aliquot of the intermediate standard (glass SDB or Tedlar bag) using a heated gastight syringe. The preparation of these standards shall follow the instructions detailed in Section 9.2.1.2. The concentrations for working standards are usually 20 and 200ng/L, however different concentrations can be chosen which work best for a particular project.

9.2.3 Initial Calibration Verification (ICV) - (Laboratory Control Sample - LCS) Prepare a secondary source standard (either a different manufacturer or different lot from the same manufacturer as the initial calibration standard) using the same procedures as the primary source. The ICV/LCS working standard should contain each target analyte present in the calibration working standard. Prepare the ICV/LCS working standard at a concentration of 200ng/L. Differing injection volumes account for the allowed concentrations listed in Table 4 for SCAN and 4A for SIM. The preparation of this standard shall follow the instructions detailed in Section 9.2.2, using the certified second-source standard cylinder.

9.2.4 Continuing Calibration Verification (CCV) Standard The CCV is the same as the initial calibration working standards detailed in Section 9.2.2.

9.2.5 Screening Standards Recommended procedure: Prepare a 0.5ug/mL and/or a 3.0ug/mL concentration standard so that the GC may be calibrated utilizing a few levels (may include approximately 0.5ng, 150ng and 600ng). However, other concentrations can be prepared depending on the desired range.

Any of the desired standard concentrations (primary and secondary) may change as long as the equations and the appropriate densities remain the same.

9.3 Storage and Expiration Dates

All standards that are to be stored in a freezer shall be stored at ≤-10˚C for DoD projects.

- Neat Stock Liquids are stored at < -10˚C (-10˚C to -20˚C) as specified by the manufacturer or for a period of five years.
- Equi-Mass Primary Stock Standard is a cocktail or soup of neat compounds (containing compounds in equal mass amounts) used to in preparing intermediate gas phase standards and shall be stored in the freezer at < -10˚C (-10˚C to -20˚C) for up to six months. This is assuming that the soup is sealed with a septum-containing screw cap or Mininert™ valve. The selection of the compounds for the soup should be performed in accordance with the guidelines in Volume 6.5 of the *Tekmar*-DOHRMANN Application Note.

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- Purchased Stock Standards Cylinders must be stored at laboratory temperature for a period of 2 years or as specified by the manufacturer before vendor re-certification or purchase of new standards.
- Intermediate Calibration Standards prepared by static dilution must be stored in an oven at a temperature of approximately 60°C to ensure analyte vaporization. Every time a standard is prepared from the static dilution bottle (SDB), the concentration changes. To increase the useful lifetime of an SDB standard, remove volumes of 25mL or less. The volume removed can be manipulated by increasing the SDB concentration or by adjusting the canister final volume/pressure. Depending upon the volume removed, an SDB intermediate standard is stable for approximately two months as long as new working standards made from this standard continue to meet acceptance criteria. These bottles must be in the oven for a minimum of one hour prior to use in preparing working standards. The guidelines for the storage and expiration date for the intermediate calibration standards are stated in Volume 6.5 of the *Tekmar*-DOHRMANN Application Note.
- Prepared Stock / Intermediate Calibration Standards prepared in Summa canisters (1000ng/L) may be stored at laboratory conditions for up to three months in an atmosphere free of potential contaminants. Upon preparation, canister standards should be allowed to sit for approximately 24 hours prior to use in order for equilibration to take place. Shorter equilibration periods may be necessary and acceptable as long as performance criteria are met.
- Calibration or Working Calibration Standards prepared in canisters may be stored at laboratory conditions for one month in an atmosphere free of potential contaminants. Upon preparation, canister standards should be allowed to sit for approximately 24 hours prior to use in order for equilibration to take place. Shorter equilibration periods may be necessary and acceptable as long as performance criteria are met.

10) Preventive Maintenance

10.1 A maintenance log will be kept documenting maintenance performed on each analytical system. The serial numbers of each instrument shall be recorded, and each log entry must include a description of the maintenance performed and be initialed by the analyst performing or observing/authorizing maintenance by an outside contractor.

The instrument maintenance log must be kept current. An entry shall be made in the appropriate log every time maintenance is performed (no matter the extent). The entry in the log must include:

(a) The date of maintenance
(b) Who did the maintenance
(c) Description of the maintenance
(d) Proof that the maintenance activity was successful

A notation of a successful tune and continuing calibration or initial calibration and the file number that accompanies the data will serve as proof that the maintenance is complete and the instrument is in working order.

The extent of the maintenance is not important, however, it is important that a notation be included for each maintenance activity such as changing a column, tuning the instrument, changing the pump oil, cleaning the source, ordering a part. In addition, a

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notation should be made in the logbook stating that no samples were analyzed during the days that the instrument was down and no active maintenance was being conducted (i.e., where no other notation was made in the logbook for those days).

10.2 Concentrating Trap

Routine maintenance includes periodic solvent cleaning of the Silco steel lines in the valve oven if contamination is suspected. Also, periodic replacement of the multi-sorbent or partial replacement of the trap if analyte specific deterioration is detected is required. For specific trap information refer to the instrument maintenance logbook and electronic method manual.

After repacking, the trap should be baked at 265°C for a minimum of two hours (or until a clean blank is generated) and a partial repacking requires baking (at 265°C) the trap for a minimum of 20 minutes (or until a clean blank is generated).

10.3 GC System

Column performance is monitored by observing both peak shapes and column bleed. Over time, the column will exhibit a poor overall performance, as contaminated sample matrices are analyzed. The length of time for this to occur will depend on the samples analyzed. When a noticeable decrease in column performance is evident and other maintenance options do not result in improvement, the column should be replaced (see Section 7.4). Whenever GC maintenance is performed, care should be taken to minimize the introduction of air or oxygen into the column.

Clipping off a small portion of the head of the column often improves chromatographic performance. When cutting off any portion of the column, make sure the cut is straight and "clean" (uniform, without fragmentation) by using the proper column-cutting tool. When removing any major portion of the column, which will affect the retention times and elution characteristics, a change in instrument conditions may be required to facilitate nominal analytical activity.

Declining performance can also be due to ineffective column ferrules, which should be replaced when a tight seal around the column is no longer possible. This can be detected with the use of a leak detector.

10.4 Mass Spectrometer

The Mass Selective Detector (MSD) ion source requires periodic cleaning to maintain proper performance. Symptoms of a dirty ion source include difficulty keeping the MSD in tune and fluctuating internal standard areas. The vacuum system should be serviced every six months, including changing the pump oil and checking the molecular sieve in the back-streaming trap.

10.5 Instrument Tuning

The instrument is tuned with guidance from the procedure described in the HP Operations Manual, when necessary.

10.6 Computer Troubleshooting

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Computer care and troubleshooting is conducted by the IT department. Refer to Section 8.5 for the computer hardware and software requirements.

Computers are selected to meet or exceed operating system and or acquisition software requirements. Periodic upgrades of memory are performed to maintain or improve system performance and reliability. Upgrades may be performed on systems until instrument hardware configurations become the limiting factor.

Basic Troubleshooting Outline:

1) Document occurrence and severity in IT Log
2) Interview user(s)
3) Investigate any available logs (Event Logs, Acquisition Logs, etc.)
4) Determine if problem is isolated (single user or acquisition) or widespread (multi user or network).
5) If multiple possibilities exist for cause, then eliminate in systematic manner.
6) Hardware issues are addressed with component replacement (beginning with most suspect portion).
7) Software issues are addressed first with internet investigation (user blogs, software source updates/findings).
8) Network issues are investigated from the Server, to Switch, to Network Card; utilizing all available managed devices to help discover possible failure points.
9) In some cases, system corruption may require reload or complete system replacement.
10) Finalize documentation in IT Log with actions taken
11) Perform periodic follow-up with User and review any log found to have suspect events that suggested source of issue.

11) Procedure

11.1 Sufficient raw data records must be retained on file of all laboratory analyses described in this document including passing QC canister checks, tune checks, instrument calibrations, verifications, sample analyses and dilutions, QC checks, and method detection limit studies. The information that is required includes: analysis/calibration date and time, test method, instrument, sample identification, analyte identification, analyst's initials, concentrations and responses, as well as standards used for the analysis and calibrations, all manual calculations including sample dilutions and manual integrations to permit reconstruction of analyses. Information entered and reported on the quantitation report and instrument run log must be complete and accurate. All data shall be obtained following defensible and ethical practices in accordance with the most recent Quality Assurance Manual and the *SOP for Laboratory Ethics and Data Integrity*.

Note: All data records must explicitly connect data to the initial instrument calibration. This includes all samples, continuing calibrations and QC samples.

11.2 Sample Preparation

The pressure/vacuum is checked and the canister pressurized upon receipt by the laboratory, as needed. When necessary, canisters shall be pressurized with humidified zero grade air. However, if the samples are to be analyzed in accordance with EPA Method 3C then the samples must be pressurized with UHP Helium (refer to Section 11.12 for additional information). The client must be made aware of this in advance and

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given the option of either submitting two canisters for analysis or receiving a report with qualified results (TO-15 Modified).

Depending on the size of the canister and location of sampling and as specified in the SOP below, samples may be pressurized to approximately 1.0psig to 3.5psig. Additional information may be found in the *SOP for Evaluation and Pressurization of Specially Prepared Stainless Steel Canisters*. Initial and final pressures are recorded in LIMS and should be repeated on the back of the sample tag. The dilution factor created by filling the sample canister is calculated using equation number 12 in Section 13.8.

11.3 Screening

The analyst must screen a sample or subset of samples if the source is of unknown origin. Typically, if the source is known to be indoor or ambient outdoor air, no screening is necessary. However, if screening is required make sure that the instrument is calibrated. A single point calibration is sufficient; however, the instrument may be calibrated utilizing a two point calibration. The ICAL points are recommended to be at approximately 0.5ng, 150ng and/or 600ng spanning the desired dynamic range. Refer to Section 9.2.5 for additional information.

Inject a 1mL or smaller aliquot of each sample into a GC/flame ionization detector (FID) system that has been calibrated with a standard containing a subset of the target analytes. This subset represents the most commonly found compounds in air samples, such as acetone, trichloroethylene, and toluene. Use the results to determine the maximum volume of sample to be analyzed by TO-15 by utilizing the following equation. Dilutions may be prepared as necessary according to Section 11.12.1.

$$I = \frac{C}{H}$$ Where:

I Injection volume (mL)
C Maximum calibration level (ng on column)
H Compound screening concentration (ng/mL)

Example: Select the compound with the highest concentration (toluene = 1.0ng/mL). If the upper calibration level is 100ng on column, then the following calculation determines the maximum injection volume to analyze.

$$\frac{100ng}{1.0ng/mL} = 100\text{mL maximum injection volume}$$

11.4 Analytical Sequence and Data System Setup

11.4.1 Data System For the Tekmar AUTOCAN, fill in the sequence log of the Teklink program with the appropriate information. Refer to the Section 11.5.1 for the operating parameters.

For HP Chemstation, load the appropriate acquisition method for the GC/MS in the top window of the Chemstation program. Suggested GC/MS operating parameters are given in Section 11.5.2.

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11.4.2 Analytical Sequence The analytical sequence must be completed for the analysis of ≤20 (19 samples including dilutions with one laboratory duplicate) field samples. A method blank (MB) shall be run to monitor for laboratory introduced contamination. There must be at a minimum a laboratory duplicate (LD) analyzed in each batch to access batch precision. The following generalized analytical sequence is to be followed:

Analytical Sequence Guideline

With Calibration	Tune Check[1] Calibration Standards (5 Standards Minimum) ICV Standard[2] (Acts as the ICV and LCS) QC Canister Checks[6] MB[7] Sample(s) – 1-19 Laboratory Duplicate[4]
With Continuing	Tune Check[1] CCV Standard[5] QC Canister Checks[6] MB[7] LCS[3] MRL Check Standard[8] Sample(s) – 1-19 Laboratory Duplicate[4]

[1] The instrument performance check solution must be analyzed initially and once per 24 hour (or as specified by the project) time period (sequence / tune window) of operation. All analyses for a sequence must be initiated (injected) prior to the expiration of the tune window.

[2] In this scenario, the ICV may also be evaluated as the LCS (differing acceptance criteria).

[3] An LCS shall be analyzed at a rate of 1 in 20 or fewer samples. The LCS is the second source calibration check standard analyzed at the lower end of the calibration curve (below the midpoint).

[4] A laboratory duplicate must be analyzed at a rate of 1 per 20 or fewer samples. The duplicate must be rotated among clients, whenever possible. Also, a duplicate laboratory control sample may be analyzed to assess precision to meet project requirements or due to sample matrix effects.

[5] A CCV must be analyzed at the beginning of every analytical sequence.

[6] Any number of QC check canisters may be analyzed in the sequence to determine a canister cleaning batch or batches acceptability.

[7] Any of the QC Check Canisters may serve as the method blank as long as the minimum requirements detailed in this document are met. A method blank shall be analyzed at a rate of 1 in 20 or fewer samples.

[8] A MRL check standard may be analyzed with each batch of 20 or fewer samples (when an initial calibration is not analyzed within the same batch). Additional information is included in Section 11.18.

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Note: Client project batch specifications may require certain modifications to the analytical sequence; however, a batch may not be more lenient than that which is specified in this document.

11.5 Conditions

11.5.1 Sample Collection Conditions The suggested settings and system parameters are as follows:

Adsorbent Trap

Set Point:	35˚
Sample Volume:	up to 1L
Dry Purge:	300mL
Sampling Rate:	100mL/min (utilize for a sample injection volume of >100mL); 40mL/min (utilize for a sample injection volume of 25-100mL)
Desorb Temp.:	230˚C to 240˚C
Desorb Flow Rate:	8-10mL/min He
Desorb Time:	3.0 minutes

Refocusing Trap

Temperature:	-180˚C
Injection Temp.:	160˚C
Injection Time:	1.0 min

Adsorbent Trap Reconditioning Conditions

Temperature:	265°C
Initial Bakeout:	2 hours or until clean blank is obtained
After each run:	5-8 minutes

Sample Run Time

Each analytical run is approximately 20 minutes long; the total cycle time is about 30 minutes between injections.

11.5.2 GC/MS System

Optimize GC conditions for compound separation and sensitivity.

Item	Condition
Carrier Gas	Helium
Flow Rate	1.0-1.5mL/minute
Temperature Program	Initial Temperature: ~20˚C Initial Hold Temperature: 3 minutes Ramp Rate: 5˚C/min to 80˚C 2nd Ramp: 10˚C/min to 160˚C 3rd Ramp: 20˚C/min to 240˚C for 5 min hold
Detector B (MSD *Interface)*	260˚C

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Electron Energy	70 Volts (nominal)
Mass Range (Scan mode)	34 to 280 amu
Mass Range (SIM mode)	Scan masses corresponding to the target analytes
Scan Time	To give at least 10 scans per peak, not to exceed 1 second per scan.

Note: The instrument may be operated in Selective Ion Monitoring (SIM) mode if requested by the client.

11.6 Instrument Performance Check

Since the BFB tuning compound is included in the internal standard and surrogate standard canister and an autosampler is used, it is necessary to establish that a given GC/MS meets tuning and standard mass spectral abundance criteria prior to the reduction and approval of any data collection. The 24-hour time period for GC/MS instrument performance check and standards calibration (initial calibration or continuing calibration verification criteria) begins at the injection of the BFB, which shall be documented in laboratory records. Upon completion of the successful BFB tune, the tune report must be printed and retained on file for future reference.

The mass spectrum of BFB must be acquired in the following manner.

- Inject 50ng or less (on column)
- Three scans (peak apex scan and the scans immediately preceding and following the apex) are acquired and averaged.
- Background subtraction is conducted using a single scan prior to the elution of BFB.
- 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.
- The ion abundance criteria must not be changed from the requirement stated in this document (TO-15 or TO-14A, as requested).

All subsequent standards, samples and QC samples associated with a BFB analysis must use identical instrument conditions.

11.7 Initial Calibration

The initial calibration is performed to determine instrument sensitivity and the linearity of the GC/MS response for the target compounds.

Initial calibration requirements are as follows:

1. A minimum of 5 concentrations must be used to calculate the calibration curve.
2. An initial calibration must be performed at a minimum initially per instrument, annually thereafter or whenever the continuing calibration verification standard does not meet the acceptance criteria.
3. Highest concentration, together with the lowest concentration, defines the calibration range.
4. The method reporting limit for any reported analyte must be at >/= the lowest calibration point.
5. The initial calibration event may not be interrupted by maintenance.
6. Only one value per concentration may be used.
7. Analyze calibration standards from lowest to highest concentration.

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8. All ICAL analyses must be completed within the 24-hour tune window.
9. If 5 calibration standards are in the ICAL, one standard may be re-analyzed. If 6 to 10 calibration standards are in the ICAL, two calibration standards may be re-analyzed.
10. One of the calibration points from the initial calibration curve must be at the same concentration as the continuing calibration verification standard.
11. The upper end of the calibration range must not exhibit any peak saturation for any analyte or the range must be lowered accordingly.
12. The initial calibration model must be linear calibration using average of response factors and cannot be changed for any reason.
13. Point dropping policy
    - Minimum of 5 consecutive concentrations must be used to calculate the calibration curve.
    - Lowest concentration must be at or below the MRL (LOQ) and may not be dropped unless the MRL is changed to the concentration of the remaining lowest standard.
    - Points at the high end may be dropped, but doing so lowers the calibration range.
    - Points may not be dropped from the interior of the curve unless an assignable cause (i.e., gross dilution error, missing internal standards, purge malfunction, standard preparation error, or instrument malfunction) is accounted for and documented. In these instances, all the analytes in that calibration standard must be dropped from the calibration curve as the corrective action (the reason must be documented and the results maintained with the documentation for the final ICAL).
    - Dropping individual compound points from the upper or lower end of the calibration range to improve linearity is not considered an error correction. The reason for dropping these points does not need to be documented but the ICAL documentation must state the revised calibration range if the MRL must be adjusted or the calibration range is lowered for a particular compound. This must be documented on the ICAL Review Checklist.
    - A calibration standard may be re-analyzed if the first analysis of the standard has been dropped and other requirements in this policy are met (i.e., still within 24 hours).
    - Once the ICAL has been used to calculate and report sample results it MUST not to be changed for any reason.
    - It is recommended that if an analyte has a higher MRL than the lowest concentration analyzed that the low standard be automatically dropped from the curve (i.e., acetone MRL is 5, drop at least the 0.5ng point).

11.7.1 <u>Calibration Points</u> Analyze the calibration standards (analyze low to high) that span the monitoring range of interest of the samples. For SCAN, the range is typically 0.5ng-100ng on column; however, 0.1ng on column may be added if low level analyses are requested. For SIM, the range is 10pg on column to 20,000pg on column. The dynamic range is dependent on the sensitivity of a particular instrument as well as the required reporting limit for a given project and may be adjusted accordingly. Refer to Table 3 (SCAN) and Table 3A (SIM) for the concentrations of the compounds of interest in the initial calibration at each particular calibration concentration level.

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Note: Refer to the EXCEL TO-15 Standard Concentration templates, located on the network at Q:\\TO15 Std. Concentrations\Std. Conc. Templates for both the SIM and SCAN templates. These templates must be utilized for the documentation of the standard canister concentration selection, final ICAL level concentrations and the determination of the correct injection volumes for the selected standard canister concentrations. If the primary or secondary stock standard cylinder concentrations are revised (upon re-certification or new purchases), the EXCEL spreadsheet templates, injection amounts and the ICAL concentrations in each instrument method must be adjusted accordingly. Other templates may be employed as long as they are validated and provide at least the same information.

SCAN

1. Determine if the lower end of the calibration range is to be 0.1ng or 0.5ng on column. If the low end is 0.1ng, then the 4ng/L standard must be utilized.
2. Determine if the 4ng/L or 20ng/L standard canister is to be used for the 0.5ng on column point.
3. Follow the instructions in the spreadsheet and save the file under the correct instrument folder and the initial calibration method identification.
4. Print the final ICAL concentration sheets and place into the corresponding ICAL folder

11.7.2 Recalibration Each GC/MS system must be recalibrated following any instrument maintenance which may change or effect the sensitivity or linearity of the instrument, if the continuing calibration verification acceptance criteria are not met and at least annually. The following procedure must be followed when updating an initial calibration method.

1. Open the most recent method.
2. Save the method with the new ICAL method ID using the "Save Method As" option. Date used in the method ID must be the date files were analyzed.
3. Quantitate midpoint standard and check retention times and integrations. Update retention times if necessary using QEdit or Easy ID (Tools → Easy ID). Requant if any changes are made and verify all peaks are identified correctly. Print.
   a. While midpoint standard is loaded update reference spectra (Continuing Calibration → Update Reference Spectra).
   b. With midpoint standard loaded update qualifier ion ratios and retention times (Initial Calibration → Update Levels → Select Update Level and then select Retention Times (Replace) and Replace Qualifier Ion Relative Responses).
   c. If necessary adjust integration parameters prior to processing remaining ICAL points.
4. Quantitate remaining ICAL standards. Review each peak for retention time, integration, and print. Review low level standards for acceptable signal to noise ratios and high level standards for saturation.
5. All responses must be cleared from ICAL before updating (Initial Calibration → Clear All Calibration Responses).
6. Update responses for each standard level (Initial Calibration → Update Levels) or (Initial Calibration → Quick Levels Update). If Quick Levels Update is used do not requant datafiles.

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7. Save method.
8. Check Response Factor Report and evaluate whether any points should be dropped following the criteria outlined in this SOP.
9. Save method if any changes are made.
10. Verify calibration files listed on Response Factor Report are correct.
11. Verify file ID, acquisition time, quant time, update time, and last update information is correct on the Calibration Status Report.

11.7.3 Analytical Window If time remains in the tune window after meeting the acceptance criteria for the initial calibration, samples may be analyzed according to the procedure described in this document (see Section 11.4.2). If time does not remain in the analytical window, a new sequence shall commence with the analysis of the instrument performance check compound (BFB) and the continuing calibration verification standard.

11.7.4 Procedure The system should be operated using temperature and flow rate parameters equivalent to those in Section 11.5. Use the standard prepared in accordance with Section 9.2.2 of this SOP. Attach the calibration standard and internal standard/surrogate canisters to the designated inlets on the preconcentrator and open the canister valves. Analyzing different volume aliquots of the calibration standards produces differing concentrations.

Analyte responses (target ion areas) are tabulated and recorded using the Enviroquant program. Quantitation ions for the target compounds are shown in Table 2 and 2A and the primary ion should be used unless interferences are present, in which case the secondary ion may be used, but the reason documented in the initial calibration file and all subsequent quantitations utilizing that ICAL must be performed using the same ion selections. Refer to Section 13.3 for the required calculations and Section 12.3 for the acceptance criteria.

11.7.4.1 Additional Requirements The procedure for performing and generating a new initial calibration method must follow a few additional requirements.

1. If any analyte lacks the appropriate sensitivity (3 to 1 signal to noise ratio) at the low end of the calibration range, this point must be dropped from the curve and the MRL/LOQ raised accordingly.
2. No detector saturation may occur for any compound; the upper calibration level must produce no saturated peaks. Exhibited by:
   - The flattening of the response for the higher concentration standards as shown on the plot;
   - The presence of a reverse tail or rise on the front part of the peak;
   - The observed actual percent ratio of the secondary ion presence is lower than the expected percent ratio; or
   - The presence of a flat topped peak and again by the decline or saturation of the secondary ion compared with the expected % recovery.

11.7.4.2 LOQ Establishment, Verification and Acceptance Criteria

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1. The LOQ must be set within the calibration range (≥ low std. of the current passing ICAL) prior to sample analysis.
2. The LOQ for each analyte must be > the analyte's LOD.
3. Initially a passing demonstration of precision and bias must be performed at the LOQ.
4. Run CCV 2 times at LOQ and:
   a. Generate a duplicate report for precision using ±25% as the criteria.
   b. Check the %Rec using laboratory generated control limits.
   c. Check the signal to noise ratio (S/N) using the software. The S/N ratio must be at least 3:1 for each analyte.
   d. All ion abundances must be acceptable per the requirements set forth in this document.
5. If any compounds fail, verify at a higher level and notify reporting. Also, make a note in the ICAL documentation.
6. Turn in <u>all</u> LOQ verification data (quant reports and software reports/checks) to QA (regardless of pass/fail).
7. Verify the LOQ on each instrument quarterly.

11.7.5 <u>Initial Calibration Review</u> Analyst's calculation and assessment along with a peer review of all ICAL data and documentation as stated in Attachment B is required before the ICAL may be used to analyze samples. In the case where samples are placed on the autosampler and allowed to run overnight, the sample results may only be reported if the ICAL is reviewed and found to be acceptable. The ICAL checklist in Attachment B must be used to document the review and approval process.

Perform a review of specific aspects of the calibration which might compromise data quality such as inappropriate extension of the calibration range with detector saturation and/or a lack of sensitivity for any analyte. Analyte concentrations which do not meet the signal to noise ratio or exhibit saturation are not to be reported and must be eliminated from the initial calibration. These instances should be followed by a short explanation regarding the reason for the omission.

11.7.6 <u>Initial Calibration File</u> An ICAL file is to be created for each initial calibration performed per instrument into which is placed the following ICAL documents. The file shall remain in the laboratory and be filed by instrument and date.

- ICAL Checklist filled out, reviewed and approved
- BFB tune analysis report
- Calibration status report (aka Calibration History)
- Relative Response Factor Report / Percent Relative Standard Deviation
- Quantitation report for each calibration standard (including manual integration documentation – before and after manual integration)
- ICV quantitation report and % recovery report.
- TO-15 Standard Concentration Spreadsheet (exact ICAL level concentrations and ICV concentrations)
- Any manual integration documentation

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11.8 Initial Calibration Verification Standard

Verify the initial calibration by analyzing an initial calibration verification standard (ICV). This standard shall be obtained or prepared from materials acquired from a different manufacturer or lot from that of the initial calibration and prepared according to Section 9.2.3.

Analyze 50ng or less (refer to Table 4 for the secondary source standard concentrations) of the ICV standard depending on the dynamic range of a given instrument and refer to Section 13.5 for the required calculations.

11.9 Continuing Calibration Verification Standard

Verify the calibration each working day, where necessary (e.g., an ICAL was not analyzed or the tune window has closed) by analyzing a continuing calibration verification (CCV) standard from the initial calibration standard canister. The concentration of the calibration verification may be varied between the low calibration standard and the midpoint of the calibration range; however, the concentration must be at one of the levels analyzed in the initial calibration. Refer to Table 3 for the standard concentrations. Refer to Section 13.4 for the required calculations.

11.10 Canister Quality Control Check and Method Blank

The method blank must be a sample of a matrix similar to the batch of associated samples that is free from the analytes of interest and is processed simultaneously with and under the same conditions as samples through all steps of the analytical procedure, and in which no target or interferences are present at concentrations that impact the analytical results for sample analyses. Prepare a canister that has not left the building by pressuring with humidified zero air. Analyze an aliquot of one liter along with the same volume of internal standard and surrogate as standards and samples. Additionally, a blank must be analyzed whenever a high concentration sample is encountered and carryover is suspected.

A Quality Control (QC) check canister pressurized with humidified zero air may serve as a method blank as long as the analyte concentration requirements stated in the canister quality control check section (Sections 12.6 and 12.7) and other requirements (refer to Section 12.11 for internal standard requirements) are met. Assuming continuing failure, another QC canister or a new canister must be prepared and analyzed in order to verify that no system contamination exists. For tracking purposes the unique laboratory barcode given to a canister shall be the information included in the sample analysis identification.

11.10.1 Sampling Systems Section 7.1 and 8.4 of Method TO-15 describe the setup and certification procedure for a specific sampling apparatus that has been used by the EPA for several of its large air monitoring programs. These systems are rarely used for the types of projects that make up the bulk of the laboratory's work. The vast majority of samples analyzed by the laboratory are taken into Summa canisters either as grab samples or using a simple time integrated sampling device (flow controller), as in Section 8.2.1 of the method, so these procedures are not part of the typical protocol for providing sampling materials to clients. The laboratory has developed an SOP for the cleaning and certification of the materials it provides its clients for obtaining air samples to be analyzed by

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method TO-15. Refer to the *SOP for Cleaning and Certification of Summa Canisters and Other Specially Prepared Canisters* for additional information.

It is this laboratory's interpretation that the sampler system certification procedure described in Section 8.4.4 of the TO-15 method applies to the specific sampling apparatus described in the method and not to the sampling procedures used by our clients. The laboratory does not maintain a dynamic calibration manifold or canister sampler apparatus as described in the method and thus performance of the relative accuracy certification procedure described in section 8.4.4. is not possible.

11.11 Laboratory Control Sample

The laboratory control sample is a sample matrix, which is free from the analytes of interest and spiked with a standard containing known amounts of analytes. The laboratory control sample is an injection of the initial calibration verification standard. Inject the LCS (ICV) at concentrations below the midpoint of the calibration curve. Make sure that all of the pertinent information is included on the quantitation report including the sample identification (LCS), concentration, standard used, and analyst.

11.12 Sample Analysis

Prior to analysis, all sample containers (canisters and bags) should be at temperature equilibrium with the laboratory.

- Attach sample canisters to Tekmar AUTOCan using a 9/16" wrench.
- Before opening the valve, check for leaking fittings by running the leak check program in the Teklink software.
- If system is leak tight, open the canister valves and start the automated preconcentration procedure. Make sure the Chemstation data acquisition software has been readied.
- Maintain the trap at an elevated temperature until the beginning of the next analysis.

Check all target compounds using the QEdit routine in Enviroquant, making sure all extracted ion chromatogram peaks are integrated properly (see Section 11.16).

Note: *The secondary ion quantitation is only allowed if there is sample matrix interference with the primary ion. If the secondary ion quantitation is performed, document the reasons in the instrument run logbook and/or on the quantitation report (initial and date any notation).*

SCAN Mode - The instrument is normally operated in the SCAN mode, where the following procedure may be followed.

- Upon sample injection onto the column, the GC/MS system is operated so that the MS scans the atomic range from 34 to 270 amu. At least ten scans per eluting chromatographic peak should be acquired. Scanning allows identification of unknown compounds in the sample through searching of library spectra. See operating conditions in Section 11.5.
- Generate a quantitation report for each run.
- If reporting Tentatively Identified Compounds (TICs), refer to Section 11.12.2 for identification criteria.

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SIM Mode - When the client requests SIM mode, select SIM instead of SCAN mode and identify a minimum of two ions per analyte of interest. Also, a minimum of two ions for each internal standard and surrogate compound should be selected.

Helium Pressurization – If a canister is pressurized with helium, a correction factor is applied to sample volumes extracted from the canister via auto sampler. This is due to the difference in thermal properties between helium and air. A correction factor worksheet has been generated to determine the exact volume taken from a canister and may be found at J:\\A-GCMS\Helium Pressurization. Save file, print the sheet and include with the data. Refer to the instruction page in the template for all of the instructions and calculations including backfilled canisters.

AutoCAN Leak Checks – Canisters should be put on at least two different AutoCAN positions to confirm a "leak". In addition, the valve threads should be inspected for defects which may prevent a good seal with the AutoCAN. Once a canister has "failed" the leak check it must be tagged, an NCAR initiated, and the PM notified. Regardless of what the client or PM specifies as the fate of the sample, the canister must be put on maintenance hold to complete a full 24-hour leak check. A yellow sheet is to be completed in addition to, but not in lieu of an NCAR. This is a fixed QA procedure with no allowance for deviation.

11.12.1 Sample Dilution If any target analyte results are above the highest level of the initial calibration, a smaller sample aliquot should be analyzed. The dynamic range of volume aliquots for the automatic cryogenic concentrator is 20cc to 1L. If a volume smaller than 20cc is to be analyzed, a dilution should be made in a Tedlar bag, or the sample directly injected using a gastight syringe. Guidance in performing dilutions and exceptions to this requirement are given below.

- Refer to Section 11.5.1 (Adsorbent Trap Sampling Rate) for the required sampling rate if less than 100mL is to be analyzed.
- Use results of the original analysis to determine the approximate dilution factor required and get the largest analyte peak within the initial calibration range.
- The dilution factor must be documented (and included in the final report) and chosen in such a way as to keep the response of the analyte peak for a reported target compound in the upper half of the initial calibration range of the instrument.

Tedlar bag dilution:

- Make a dilution by filling a Tedlar bag with 1.0 liter of humidified zero air using a one-liter gas syringe.
- Calculate the volume of balance gas needed to obtain the required dilution.
- Remove the difference in the balance gas using a syringe.
- Add the calculated sample amount using a gastight syringe.

Direct injection:

- Make a direct injection by attaching a clean, humidified zero air filled Summa canister to the preconcentrator autosampler using 1/4" stainless steel or teflon tubing with a "tee" septum port. This canister should be the same canister that may be used as the method blank.

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- Inject the sample through the septum while the preconcentrator withdraws a 200cc aliquot from the canister.

11.12.2 Tentatively Identified Compounds When requested, a mass spectral library search may be made for the purpose of tentatively identifying sample components not associated with the calibration standards. The necessity to perform this type of identification will be determined by the purpose of the analyses being conducted. Data system mass spectral library search routines should not use normalization routines that would misrepresent the library or unknown spectra when compared to each other.

Certain programs may require the reporting of non-target analytes. Only after visual comparison of sample spectra with the nearest library searches may the analyst assign a tentative identification. The following guidelines are used for making tentative identifications.

- Relative intensities of major ions in the reference spectrum (ions greater than 10% of the most abundant ion) should be present in the sample spectrum.
- The relative intensities of the major ions should agree within ±20%. For example, for an ion with an abundance of 50% in the standard spectrum, the corresponding sample ion abundance should be between 30 and 70%.
- Molecular ions present in the reference spectrum should be present in the sample spectrum.
- Ions present in the sample spectrum but not in the reference spectrum should be reviewed for possible background contamination or presence of co-eluting compounds.
- Ions present in the reference spectrum but not in the sample spectrum should be reviewed for possible subtraction from the sample spectrum because of background contamination or co-eluting peaks. Data system library reduction programs can sometimes create these discrepancies.
- The concentration of the tentatively identified compound is estimated by assuming a response factor of 1.0 and comparing the response of the tentatively identified compound to the response of the nearest internal standard.
- If non-target analytes are not Q-deleted from the quant report, the analyst must evaluate whether these compounds should be reported as TICS.

11.13 Duplicate

A duplicate must be analyzed to assess laboratory precision and samples selected for duplicate analysis shall be rotated among client samples, where applicable. Some projects or sample matrix issues may require the analysis of a duplicate laboratory control sample (DLCS).

11.14 Internal Standard (IS)

The concentration of internal standard added to each standard, field sample and QC sample must be consistent from that of each current ICAL standard.

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11.15 Surrogates

Internal standards/surrogates must be added at the same volume for every standard, sample and QC sample. Surrogate compound recoveries are requested by a number of clients, but are more appropriately used as system monitoring compounds. This is due to the fact that the compounds are introduced directly into the analytical system and not into the canisters or bags. It is for this reason that they are not considered to be true surrogates and a fixed window is applied. Additionally, surrogates are not included in the ICAL because they are not required by the method and are only system monitoring compounds.

11.16 Manual Integration and Q Deletion

A list of abbreviations (codes) that may be used to give a reason for performing either of these procedures are listed in the *SOP for Data Review and Reporting*.

11.16.1 Manual Integration The integration for each peak must be legally defensible and shall be checked to ensure that it has been integrated properly and consistently between samples, standards and QC samples. All peak reviews and manual integrations must follow the requirements specified in the *SOP for Manual Integration Policy* and the *SOP for Laboratory Ethics and Data Integrity.* The requirements in the above stated procedure include when manual integrations are performed, raw data records shall include a complete audit trail for those manipulations (i.e., chromatograms showing both the integration prior to any manual integrations and those depicting the corresponding manually integrated peaks), and notation of rationale, date, and initials of person performing the manual integration operation. In addition, manual integrations must be reviewed and approved by a second reviewer and the manual integrations maintained in the appropriate job file.

Reporting Requirements Certain project requirements including samples which are submitted under the Department of Defense (DoD) QSM require that the case narrative include an identification of samples and analytes for which manual integration is required. Refer to project requirements to determine if this is necessary.

11.16.2 Q Deletion Q deleting may be performed to either delete a false positive or delete non-target compounds.

11.17 Detection Limits and Limits of Detection

The MDL study shall be performed annually for all target analytes on each instrument (with identical configurations) for which this method is performed. The MDL shall be performed in accordance with the procedure outlined in the *SOP for Performing Method Detection Limit Studies and Establishing Limits of Detection and Quantitation.* The detection limit shall be used to determine the LOD for each analyte.

Once determined on each instrument, the highest LOD (for each analyte from all instrument determinations) shall be used as the uniform LOD. However, if a lower detection limit is reported, then the samples must have been run on that specific instrument on which the lower LOD was determined.

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11.17.1 Performance and Acceptance Criteria

1. The MDL must be <0.5ppbV for each analyte (Method 11.11.1).
2. Perform Limit of Detection (LOD) verification on all instruments (performing this method) immediately following the MDL study. Spike the LOD at 1-4x the MDL; the spike level establishes the LOD.
3. LOD Acceptance
   - Analyte must be detected reliably and identified by the method-specific criteria (i.e, ion confirmation) and produce a signal that is at least 3 times the instrument's noise level (3:1 signal to noise ratio).
   - It is specific to each combination of analyte, matrix, method and instrument configuration.
   - The LOD must be verified quarterly on each instrument (spiked at LOD) using the criteria listed above.
4. If the LOD verification fails (per #3), repeat the detection limit determination and LOD verification at a higher concentration or perform and pass two consecutive LOD verifications at a higher concentration and set the LOD at the higher concentration.
5. The laboratory shall maintain documentation for all detection limit determinations and LOD verifications (regardless of pass or fail).

11.18 Method Reporting Limit Check Standard

It is recommended to analyze a MRL check standard at the current MRL or required MRL for the batch (per client requirements) of twenty or fewer samples if the CCV fails low for any target compound. A MRL check standard may also be required per client specifications.

This check standard can also serve as the LOQ verification if it meets the specific requirements listed in Section 11.7.4.2. Apply the requirements and retain all documentation accordingly. Refer to Attachment D for Minnesota specified MRL check standard criteria.

11.19 Storing Electronic Data

The initial calibration data must be stored in a quantitation method (on the server) using a unique filename and may not be overwritten at any time in order to maintain an accurate audit trail. There are multiple quantitation methods, which are subsets of the compound list in Table 2. Therefore, files will be named with an eight-character notation indicating the compound list and the date of the corresponding initial calibration. In addition, all data files including method blanks, continuing calibration verification, laboratory control samples and client submitted samples files are saved in a unique sub-directory on the server.

12) Quality Assurance / Quality Control Requirements

12.1 To the extent possible, samples shall be reported only if all of the quality control measures are acceptable. If a quality control measure is found to be out of control, and the data must be reported, all samples associated with the out of control quality control measure shall be reported with the appropriate data qualifier(s).

12.2 Instrument Performance Check

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Refer to Tables 1 and 1A for the required ion abundance criteria.

12.3 Initial Calibration

- The RRT for each target compound at each calibration level must be within 0.06RRT units of the mean RRT for the compound.
- The calculated %RSD for the RRF for each compound in the calibration standard must be less than 30% with at most two exceptions up to a limit of 40% (this may not be true for all projects).
- For each Internal Standard the area response (Y) at each calibration level must be within 40% of the mean area response $\overline{Y}$ over the initial calibration range.
- The retention time shift for each of the internal standards at each calibration level must be within 20s of the mean retention time over the initial calibration range for each internal standard.
- All of the following information must be retained to permit reconstruction of the initial instrument calibration: calibration date, test method, instrument, analysis date, analyte identification, analyst's initials, concentration and responses, and response factors.
- All initial instrument calibrations must be verified with an acceptable ICV.

12.4 Initial Calibration Verification Standard

The percent recovery for each compound in the ICV must be between 70%-130% for all analytes except vinyl acetate, which must be within 50-150%. Exceptions to this allowance for the vinyl acetate recovery are project specific requirements and any DoD type project, which shall adhere to the 70-130% requirement for all target compounds.

12.5 Continuing Calibration Verification Standard

All compounds must be evaluated prior to rounding. The percent difference for each target analyte must be within plus or minus 30% of the initial calibration average RRFs.

12.6 Canister Quality Control Check

The actual cleaning procedure, number of cans to select for analysis (to release a cleaning batch) and corrective actions are covered in the *SOP for Cleaning and Certification of Summa Canister and Other Specially Prepared Canisters* and are not covered in this section. However, the procedure for analyzing and certifying a cleaning batch is included. If a canister passes as a QC canister it meets all of the requirements for a method blank (Method, NELAC\TNI, and Department of Defense Quality Systems Manual – DoD QSM, etc.).

12.6.1 Scan Analyses A canister is considered "clean" for normal SCAN analyses if the analysis shows <0.2ppbv of any target analyte (analyte exceptions listed in table below). If a canister passes as a QC canister it meets all of the requirements for a method blank (Method, NELAC\TNI, and Department of Defense Quality Systems Manual - DoD QSM, etc.).

Low Level SCAN Analyses For those analytes with a MRL of 0.1ug/m3, the QC criteria of <MRL is acceptable; otherwise, <0.2ppbV is required (analyte exceptions listed in table below).

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SIM Analyses Results <MRL will be acceptable as this complies with the <0.2ppbV method requirement.

ANALYTE EXCEPTION LIST					
Compounds	ppbV	On Column (ng)	Compounds	ppbV	On Column (ng)
Target Analytes	0.2	0.50	Acrylonitrile	0.2	0.43
Chloromethane	0.2	0.41	Acetone	1.5	3.5
1,3-Butadiene	0.2	0.44	Ethanol	1.9	3.5
Acetonitrile	0.2	0.33	Vinyl acetate	0.99	3.5
Acrolein	0.65	1.5	1-Butanol	0.23	0.70
Isopropanol	0.28	0.70	Carbon Disulfide	1.1	3.5
2-Butanone	1.2	3.5			

Make a notation on the sample identification tag as to the status of the check and return the canister to the canister conditioning room. Additionally, if the check was found to be acceptable, the quantitation report must be kept on file for future reference

12.6.2 Tentatively Identified Compounds (TIC) If the batch of canisters are to be used for tentatively identified compounds (TIC) analysis, any non-target peaks present in the QC check canister analysis must be evaluated and determined to be less than the TIC reporting limit (10% of the internal standard). The concentration is estimated by assuming a RRF of 1.0 and comparing the response of the TIC to the response of the nearest internal standard.

12.7 Method Blank

- The concentration of a targeted analyte in the blank cannot be at or above the MRL, AND be greater than 1/10 of the amount measured in any associated sample. For any project that requires reported results less than the MRL, all associated measurements found in the MB should result in a qualifier; however, project requirements may differ and must be followed. Refer to DoD requirements listed below.
- The method blank should not contain additional compounds with elution characteristics and mass spectral features that would interfere with identification and measurement of a method analyte.
- For DoD samples, the method blank will be considered to be contaminated if:

  1. The concentration of any target analyte in the blank exceeds 1/2 the reporting limit and is greater than 1/10 the amount measured in any sample or 1/10 the regulatory limit (whichever is greater);
  2. The concentration of any common laboratory contaminant (acetone, ethanol, carbon disulfide, and methylene chloride) in the blank exceeds the reporting limit and is greater than 1/10 the amount measured in any sample or 1/10 the regulatory limit (whichever is greater); or
  3. The blank result otherwise affects the samples results as per the test method requirements or the project-specific objectives.

The laboratory shall evaluate whether reprocessing of the samples is necessary based on the above criteria.

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12.8 Laboratory Control Sample

Round all results to the nearest whole number prior to determining if the acceptance criteria have been met. The percent recoveries must be within the laboratory-generated limits and are referenced in the electronic TO-15 Method Manual. However, Arizona requires the percent recovery for each compound in the LCS to be 70%-130% (to match the ICV requirement). Therefore, the ICV exception for vinyl acetate stated in Section 12.4 requires the percent recovery for AZ samples to be 50-150%.

Note: Client project requirements, AFCEE and DoD requirements shall take precedence over the AZ requirement for AZ samples. Meaning if a sample is collected for a DoD project in AZ, DoD requirements specified in this document and the project specific QAPP (if supplied) are to be followed.

12.9 Sample Results

- Sample results must be quantitated from the initial instrument calibration and may not be quantitated from any continuing instrument calibration verification.
- Samples out of holding time must be handled according to Section 16.
- The field sample must be analyzed on a GC/MS system meeting the BFB tuning, initial calibration, initial calibration verification technical acceptance criteria described in this document.
- All target analyte peaks must be within the initial calibration range, diluted or reported with the appropriate data qualifier.

12.10 Laboratory Duplicate

The relative percent difference must fall within ±25%. This RPD criterion also applies to duplicate laboratory control samples (DLCS).

12.11 Internal Standards

The following acceptance criteria must be applied to each run (except the ICAL – see Section 12.3).

- The area response for each internal standard in the blank must be within ±40 percent of the area response for each internal standard in the most recent valid calibration. (CCV or mid-point from the initial calibration, whichever is most current).
- The retention time for each internal standard must be within ±0.33 minutes of the retention time for each internal standard in the most recent valid calibration. (CCV or mid-point from the initial calibration, whichever is most current).

12.12 Surrogates

Since the matrix precludes the use of true surrogates and there is no established method criterion, acceptable surrogate recoveries are based on a fixed window of 70 - 130%. This is the typical requirement from clients. Additionally, these limits are referenced in SW-846 for use as guidance in evaluating recoveries. These limits are sufficient for evaluating the effect indicated for the individual sample results.

12.13 Method Reporting Limit Check Standard

Per client requirements or if the CCV is biased low for any compound, then evaluate the MRL check standard. Analyte must be detected reliably and identified by the method-

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specific criteria (i.e, ion confirmation) and produce a signal that is at least 3 times the instrument's noise level (3:1 signal to noise ratio). Also, a percent difference +/-50% is recommended.

13) Data Reduction and Reporting

13.1 The essential information to be associated with analysis, such as computer data files, run logs, etc. shall include: Sample ID code, date and time (if the holding time is 72 hours) of analysis, instrument operating conditions/parameters (or reference to such data), analysis type, all manual calculations including dilutions and manual integrations, analyst's initials, sample preparation (pressure readings and balance gas if pressurized with helium), standard and reagent origin, receipt, preparation, and use, as well as calibration criteria, frequency and acceptance criteria, data and statistical calculations, review, confirmation, interpretation, assessment and reporting conventions.

13.2 This method has specific requirements including the use of canisters; any modification must be reported accordingly. All reports that fall under the laboratory's certificate of approval (in accordance with NELAC/TNI standards) must include a statement(s) clarifying any deviations from the scope of this certification. Refer to Section 13.11 for additional information and specific items, which require this clarification.

13.3 Initial Calibration

Tabulate each of the following:

13.3.1 Equation Number 1 - **Relative Response Factor (RRF):**

$$RRF = \frac{A_x C_{is}}{A_{is} C_x}$$ where:

A_x is the area response of the analyte quantitation ion.
A_{is} is the area response of the corresponding internal standard quantitation ion.
C_{is} Internal standard concentration, ng.
C_x Analyte concentration, ng.

Note: The equation above is valid under the condition that the volume of internal standard spiking mixture added in all field and QC samples is the same from run to run.

13.3.2 Equation Number 2 - **Average (or Mean) RRF:**

$$\overline{RRF} = \frac{\sum_{i=1}^{N} RRF_i}{N}$$ where:

RRF_i are the individual RRFs from each concentration level in the initial calibration curve.

N is the number of calibration concentration levels.

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13.3.3 Equation Number 3 - **Standard Deviation, SD:**

$$SD = \sqrt{\sum_{i=1}^{N} \frac{\left(RRF_i - \overline{RRF}\right)^2}{N-1}}$$ where:

RRF_i are the individual RRFs from each concentration level in the initial calibration curve.

$\overline{RRF}$ Average (or Mean) RRF of all concentration levels in the initial calibration curve.

N total number of calibration concentration levels

13.3.4 Equation Number 4 - **Percent Relative Standard Deviation, %RSD:**

$$\%RSD = \frac{SD}{\overline{RRF}}(100)$$ where:

SD Standard Deviation calculated in equation number 3

$\overline{RRF}$ Average or Mean RRF

13.3.5 Equation Number 5 - **Relative Retention Time (RRT):**

$$RRT = \frac{RT_C}{RT_{is}}$$ where:

RT_C Retention time of the target compound, seconds.

RT_{is} Retention time of the internal standard, seconds.

13.3.6 Equation Number 6 - **Mean Relative Retention Time ($\overline{RRT}$):**

$$\overline{RRT} = \sum_{i=1}^{n} \frac{RRT_i}{n}$$ where:

$\overline{RRT}$ Mean relative retention time (seconds) for the target compound for all initial calibration levels.

RRT_i Relative retention time for the target compound in level i.

n Number of calibration levels

13.3.7 Equation Number 7 - **Mean Area Response ($\overline{Y}$):**

$$\overline{Y} = \sum_{i=1}^{n} \frac{Y_i}{n}$$ where:

Y_i Area response for the primary quantitation ion for the internal standard for each initial calibration standard.

n number of calibration concentration levels

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13.3.8 Equation Number 8 - **Mean Retention Times ($\overline{RT}$):**

$$\overline{RT} = \sum_{i=1}^{n} \frac{RT_i}{n}$$ where:

$\overline{RT}$ Mean retention time, seconds
RT_i Retention time for the internal standard for each initial calibration standard, seconds.
n number of initial calibration levels

13.4 Continuing Calibration Verification

- Calculate the (RRF) of each target compound using equation number 1 (13.3.1).

13.4.1 Equation Number 9 - **Percent Difference, %D:**

$$\%D = \frac{RRFx - \overline{RRF}}{\overline{RRF}}(100)$$ where, for any given analyte:

RRF_x is the RRF from the CCV being evaluated.
$\overline{RRF}$ is the mean RRF from the current calibration curve.

13.5 Percent Recovery – ICV, LCS, Surrogates, MRL Check Standard

13.5.1 Equation Number 10 - **Percent Recovery (%R):**

%R = X/TV x 100

where
X = Concentration of the analyte recovered
TV = True value of amount spiked

13.6 Duplicate Analysis

13.6.1 Equation Number 11 - **Relative Percent Difference (RPD):**

$$\frac{x_1 - x_2}{\overline{x}}(100)$$ where:

x_1 First measurement value
x_2 Second measurement value
$\overline{x}$ Average of the two values

13.7 Internal Standards (IS)

- Calculate the mean area response $\overline{Y}$ for each internal standard using equation number 7 (Section 13.3.7).

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- Calculate the mean of the retention times for each internal standard using equation number 8 (Section 13.3.8).

13.8 Pressure Dilution Factor (PDF)

13.8.1 Equation Number 12 - **PDF**, for samples collected in Summa canisters:

$$\text{PDF} = \frac{P_{atm} + P_f}{P_{atm} + P_i}$$ where:

P_{atm} is the ambient atmospheric pressure, 14.7 psi at sea level.
P_f is the final sample canister pressure, in psig.
P_i is the initial sample canister pressure, in psig. This will most often be a negative value (sub-ambient initial pressure).

13.9 Results

If a canister has been pressurized with Helium and the Tekmar AutoCan was utilized, refer to Section 11.12.

13.9.1 Equation Number 13 - For calculating analyte concentrations in a sample, the starting point is the nanogram amount generated by the HP Enviroquant software, which appears on the quantitation report.

$$ng_x = \frac{A_x ng_{is}}{A_{is}\overline{RRF}}$$ where:

ng_x is the nanogram amount of analyte *x*.
A_x is the area response of the analyte's quantitation ion.
A_{is} is the area response of the corresponding internal standard's quantitation ion.
ng_{is} is the internal standard amount, in nanograms.
$\overline{RRF}$ is the average or mean RRFs

13.9.2 Equation Number 14 - The final analyte concentration, C_x, in units of micrograms per cubic meter (µg/m³), is then calculated from the following:

$$C_x = \left(\frac{ng_x PDF}{V}\right)\left(\frac{1\mu g}{1000ng}\right)\left(\frac{1000l}{1m^3}\right)$$ where:

V is the sample volume analyzed, in liters.
PDF is the sample canister pressure dilution factor.

13.9.3 Equation Number 15 - To convert to units of parts per billion volume (ppbv):

$$ppbv = \frac{\mu g/m^3}{MW} x24.46 \qquad \mu g/m^3 = \frac{ppbv}{24.46} xMW$$ where:

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MW is the molecular weight (Table 2) of the analyte, in g/mole.
24.46 is the molar volume of an ideal gas at 298 K (25 °C) and 760 mmHg (1 atm), in liters per mole (l/mol).

C_x the final analyte concentration in micrograms per cubic meter.

13.9.4 Equation Number 16 – Helium Pressurization (Injection Amount)

Applicable to canisters pressurized with helium and injected utilizing the mass flow controller of the AutoCAN. For full instructions and calculations, refer to the 1st tab of the template located at: J:\A-GCMS\Helium Pressurization\MFC_GCF _backfill.

13.10 Data Review

The analyst must review data on a real time basis for all calibration and QC data. The QC data must be evaluated by analytical sequence following the data review checklist in Attachment C. The data shall be reviewed and the sample results calculated and assessed by one analyst and reviewed by a second qualified analyst. The data review checklist is used to document the reviews and once it has been completed, initialed and dated it must be filed with each job file.

Initial calibrations must be reviewed in the same manner as QC data with all ICAL documentation retained in a separate file organized by instrument and date. Refer to the initial calibration checklist in Attachment B for the review guideline. The ICAL file must contain all the pertinent information stated in Section 11.7.7.

13.11 Reporting

The results of each test shall be reported clearly, unambiguously and objectively, and shall include all the information necessary for the interpretation of the test results and information required by this laboratory's policy, NELAC\TNI standards, DoD Manual (applicable version, see reference section), client projects, and the TO-15 method including modifications, observances, data qualifiers, and certification information.

If the project requires that results be reported below the MRL (LOQ), but above the LOD all of the requirements specified for normal reporting apply (3:1 S/N ratio and ion abundance). This is regardless of the fact that the results will be qualified as estimated.

13.11.1 Analysis Observations / Case Narrative Summary Form

This form, which is included in the *SOP for Laboratory Storage, Analysis and Tracking*, must be generated when there are specific sample composition information or analysis issues and/or observations. In addition, during the analysis, specific identification information or problems, interferences, calibration issues, flags, and additional/expanded explanation of flags should be added to the form. This form may be modified as long as the sections and basic concepts are reserved. All data qualifiers and flags should follow those listed in the most recent Quality Assurance Manual or as defined in any client requirements.

This form is necessary as a means for documentation. This form, among other information, will be reviewed when compiling the final report and case narrative.

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All information regarding the job shall remain in the file, in order that sufficient documentation is available to recreate the job from sample receipt through analysis, data reduction, and reporting.

13.11.2 NELAC\TNI Requirements

The following items do not comply with NELAC\TNI standard requirements and must be reported accordingly. A statement, however worded, must be included in the final report indicating that data reported does not fall under the laboratory's NELAC certificate of approval.

- Reporting any compound which is not included in the second source standard (ICV or LCS) does not meet NELAC requirements.
- In addition, a report that contains a compound not included on the NELAC certificate of approval must also include the statement listed above.

13.11.2.1 Modifications

Method modifications are also not allowed under NELAC\TNI standards; therefore, a statement, however worded, must be included in the final report indicating that data reported does not fall under the laboratory's NELAC certificate of approval. In addition, the following items are considered to be method modifications and must be reported accordingly.

- Sample collection in gas collection bags
- The pressurization of canisters with nitrogen or helium (if EPA Method 3C is requested) refer to Section 11.12.

13.11.3 Surrogates

Only report surrogates at the request of the client. If any surrogate is out of control, all samples results (with surrogates requested) associated with the surrogate must be reported with the appropriate data qualifier.

13.11.4 DoD Requirements

Report results with the appropriate data qualifiers, if samples cannot be reanalyzed for any reason. In addition and at a minimum, the following situations are to be noted in the case narrative: manual integrations, CCV out of control, and results exceeding the calibration range.

14) Method Performance

14.1 An on-going assessment of method performance is conducted in order to ensure that the laboratory is capable of reporting results which are acceptable for its intended use. Validation of the method is confirmed by the examination and provision of objective evidence that these requirements are met.

14.2 Method Detection Limit (MDL)

The procedure used to determine the method detection limits are as stated in the *Code of Federal Regulations* (40 CFR 136 Appendix B) as defined in the *SOP for Performing Method Detection Limit Studies and Establishing Limits of Detection and Quantitation*. The MDL is

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defined as the minimum concentration of a substance that can be measured and reported with 99% confidence that the value is above zero. The MDL concentrations are listed in Tables 2 and 2A for both SCAN and SIM modes and were obtained using spiked canisters prepared with humidified zero air, making at least seven replicate measurements of the compounds of interest, computing the standard deviation, and multiplying this value by the appropriate Student's t value for 99 percent confidence. The MDL actually achieved in a given analysis will vary depending on instrument sensitivity and matrix effects. All MDLs, regardless of the mode of operation, meet the method performance criteria of <0.5ppbV.

14.3 Accuracy and Precision

Refer to Section 11.4 in the referenced method for information on replicate precision criteria for method performance. Single laboratory accuracy is presented as the second source initial calibration verification standard, which meets the method performance criteria of 30%. Additionally, laboratory generated control limit data for LCSs are presented for the analytes of interest and may be referenced in the electronic TO-15 Method Manual. Refer to Section 11.7.4.2 for the accuracy and precision requirements for concentrations at the LOQ/MRL.

14.4 Selectivity

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 multidetector arrangement of these (see discussion in Compendium Method TO-14A). The use of both gas chromatographic retention time and the generally unique mass fragmentation patterns reduce the chances for misidentification.

It is necessary to establish that a given GC/MS meets tuning and standard mass spectral abundance criteria prior to initiating any data collection. Upon sample injection onto the column, the GC/MS system is operated so that the MS scans the atomic mass range from 35 to 300 amu. At least ten scans per eluting chromatographic peak must be acquired. Scanning also allows identification of unknown compounds in the sample by searching through library spectra.

The sample analysis using the GC/MS is based in part on a combination of retention times and relative abundances of selected ions. The retention time of each chromatographic peak should be ±0.10 minutes of the library/reference retention time of the compound. The acceptance level for relative abundance should be set at ±20% of the expected abundance. The data should be manually examined by the analyst to determine the reason for the # flag [(#) = qualifier out of range], if present and whether the compound should be reported as found or if there is matrix interference. A background subtraction may aid in this determination. Manual inspection of the qualitative results should also be performed to verify concentrations outside the expected range.

Specific selectivity information is provided in this section and document (such as relative retention time) as well as in the referenced method. Refer to the method for additional information on selectivity.

- Use NIST Library 98 or newer version

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- The *reference spectra updates* must be performed with every new ICAL utilizing the mid-level standard (minimum). If needed, the reference spectra may be updated sooner with the continuing calibration standard.
- *Retention time updates* must be performed using EasyID and not by updating to the method (InitCal \ Update Calibration). Refer to the Help selection of the software.

14.5 Demonstration of Capability

This laboratory has continuously performed this method since before July 1999. Therefore, ongoing demonstration of capable shall be performed and documented; however, the initial demonstration of method capability is not required.

14.6 Proficiency Testing (PT) Program

The laboratory shall participate in an air and emissions PT study for TO-15. The testing shall be performed in accordance with this document and meet the frequency and proficiency requirements detailed in the DoD QSM Version 4.2 (Requirement Box 43).

15) Pollution Prevention and Waste Management

15.1 All waste disposals shall be carried out in accordance with the requirements detailed in the *SOP for Waste Disposal.* In addition, canisters must be cleaned in accordance with the requirements detailed in the *SOP for Cleaning and Certification of Summa Canister and Other Specially Prepared Canisters.*

16) Corrective Actions for Out-of-Control Data

16.1 Corrective actions shall follow the procedures outlined in the *SOP for Nonconformance and Corrective Action*, where appropriate. Any maintenance which may alter instrument sensitivity or linearity must result in the re-analysis of the entire sequence including the tune compound, ICAL or CCV or any batch QC.

16.2 Tune Does Not Meet Criteria

Perform auto tune or manual tune and then re-analyze BFB. If the BFB acceptance criteria are still not met, the MS must be retuned according to the procedure outlined in the instrument user's manual. Perform necessary maintenance and make notations in the instrument maintenance logbook. It may be necessary to clean the ion source, or quadrupole, or take other necessary actions to achieve the acceptance criteria. An acceptable tune is required for sample results to be calculated and reported.

16.3 Initial Calibration Does Not Meet Calibration Criteria

Follow the initial calibration requirements detailed in Section 11.7 for information on re-analyzing or dropping points and the restriction of maintenance performed during the analysis of the initial calibration standards.

If the initial calibration results are outside the established acceptance criteria, corrective actions must be performed and all associated samples reanalyzed, if reanalysis of the samples is not possible, data associated with an unacceptable initial calibration shall be reported as estimated with the appropriate data qualifiers.

16.4 Initial Calibration Verification Does Not Meet Criteria

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If the initial calibration verification technical acceptance criteria are not met, reanalyze and if it fails again, prepare a new canister and analyze. If the criteria are still not met inspect the system for possible sources and perform any necessary maintenance and make a notation in the maintenance logbook of any steps taken. It may be necessary to clean the ion source or change the column. Perform a new initial calibration if any performed maintenance has altered instrument linearity and/or sensitivity. Perform another initial calibration or if reanalysis is not possible, data associated with an unacceptable ICAL/ICV shall be reported as estimated with the appropriate data qualifiers.

16.5 Continuing Calibration Verification Results Do Not Meet Calibration Criteria

If the continuing calibration verification technical acceptance criteria are not met, reanalyze and if it fails again, prepare a new canister and analyze. If the criteria are still not met inspect the system for possible sources of the problem and perform any necessary maintenance and make a notation in the maintenance logbook of any steps taken. It may be necessary to clean the ion source or change the column.

If any corrective action and/or reanalysis fails to produce continuing calibration verification within acceptance criteria (analyzed immediately following the initial failure), then either two consecutive successful verifications must be performed following corrective action or a new initial calibration must be performed; however, refer to 16.5.1 below.

16.5.1 Method Reporting Limit Check Standard

If the MRL check standard is unacceptable for any compound (sensitivity; ratio or %D), reanalyze at the same or higher level within the same batch and report data with the CCV flag and case narrative notes accordingly.

16.6 Internal Standards Do Not Meet Criteria

16.6.1 Internal Standard Responses If the problem is with the instrument, perform maintenance. If the problem is with a sample, check for interferences. If the response is high, it is likely that interference is present. In this case, lower the volume or aliquot of the sample and re-analyze. If the problem persists, report the results with the best quality and qualify the results. If the problem is corrected with the lower volume analysis, report those results.

16.6.2 Internal Standard Retention Times If the retention time for any internal standard within the sample changes by more than 20 sec from the latest daily calibration or initial calibration mid-point standard, the GC/MS system must be inspected for malfunctions, and maintenance performed as required. Repeat sample analysis where required.

16.7 Method Blank (MB) Results Do Not Meet Criteria

If the analyte concentration results in the blank do not meet the acceptance criteria repeat analysis with remaining QC canisters until results are acceptable or prepare a canister per Section 11.10. If the analyte results in the blank still do not meet the acceptance criteria the source of the problem must be investigated and measures taken to eliminate the source. Each method blank must be critically evaluated as to the nature of the interference and the effect on the analysis of each sample within the batch.

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Determine whether the contamination is from the instrument or due to contamination in the blank container (if results from the new can are not acceptable then the system is probably contaminated). In all cases, the corrective action (reprocessing or data qualifying codes) must be documented. However, the specific corrective action depends on the type of project the blank is utilized for; therefore, refer (below) to the reporting/reprocessing requirements.

DEPARTMENT OF DEFENSE (DoD) QSM PROJECT: Any sample associated with a blank that fails the criteria shall be reprocessed in the same or subsequent analytical batch, except when the sample analysis resulted in a non-detect. If reanalysis is not performed, the results shall be reported with appropriate data qualifier.

OTHER PROJECT TYPE: Appropriate corrective measures must be taken and documented before sample analysis proceeds. However, if this is not a possibility and the results must be reported follow the reporting requirements stated in Section 17.4.

16.8 Laboratory Control Sample (LCS) Results Do Not Meet Criteria

If the LCS criteria are not met, determine whether the cause is instrumentation or the result of a poor injection. If the problem is instrumentation, perform maintenance and if the problem is with the injection re-analyze the LCS. DoD considers the same analyte exceeding the LCS control limits two out of three consecutive LCS to be indicative of non-random behavior; therefore, this trend should be monitored and the appropriate corrective action taken when it occurs.

16.9 Laboratory Duplicate Results Do Not Meet Criteria

If the duplicate results do not meet the technical acceptance criteria, perform another duplicate analysis. If the results are still unacceptable and the associated samples are not reanalyzed then all of the sample results in the associated batch must be flagged accordingly.

16.10 Sample Results Do Not Meet Criteria

- If the retention time for any internal standard within the sample changes by more than 20 sec from the latest daily calibration or initial calibration mid-point standard, the GC/MS system must be inspected for malfunctions, and maintenance performed as required. Repeat sample analysis as needed.
- If the area for any internal standard changes by more than ±40 percent between the sample and the most recent calibration, check for possible matrix interferences and re-analyze at a greater dilution. If the requirement is still not met and matrix interference is not detected the GC/MS system must be inspected for malfunction and maintenance made where necessary.
- When corrective actions are made, samples analyzed while the instrument was not functioning properly must be re-analyzed or the appropriate data qualifiers must be attached to the results.

To the extent possible, samples shall be reported only if all of the quality control measures are acceptable. If a quality control measure is found to be out of control, and the data must be reported, all samples associated with the out of control quality control measure shall be reported with the appropriate data qualifier(s).

16.11 Sample Holding Time Expired

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The customer is to be notified that the sample's holding time was missed and the customer is to decide if the sample analysis is to continue. The documentation of missed holding time and the client's decision to proceed must be included in the corresponding job file. A statement dictating all holding time occurrences must accompany the sample results in the final report.

16.12 Surrogate Results Do Not Meet Criteria

Poor surrogate recovery should be followed by re-analyzing a smaller aliquot to mitigate any matrix interferences. Evaluate the out of control surrogate for the effect on individual sample results.

17) Contingencies for Handling Out-of-Control or Unacceptable Data

17.1 The following is specific information on how to report unacceptable data. If the data requires a data qualifier flag, as specified in this SOP, refer to Appendix D of the most recent version of the Quality Assurance Manual for the appropriate data qualifier.

17.2 Initial Calibration and/or Initial Calibration Verification

All results reported with an unacceptable ICAL must be reported as estimated and all data shall be reported using defined qualifiers or flags or explained in the case narrative accordingly.

17.3 Continuing Calibration Verification

All results associated with an unacceptable CCV (other than #1 below) must be reported with the appropriate data qualifier, flag and/or explained in the case narrative.

1. When the acceptance criteria for the continuing calibration verification are exceeded high, i.e., high bias, and there are associated samples that are non-detects, then those non-detects may be reported without a qualifier.
2. When the acceptance criteria for the continuing calibration verification are exceeded high, i.e., high bias, and there are associated samples with detects, then those detects must be reported with a qualifier, flag and/or explained in the case narrative.
3. If however, the acceptance criteria for the continuing calibration verification are exceeded low, i.e., low bias, and there are associated samples that are non-detects, then those non-detects must be reported with qualifiers, flags and/or explained in the case narrative as having less certainty. However, along with the data qualifiers, the case narrative may include information stating the fact that the results were not significantly affected if:
   a. *An MRL check standard was analyzed and found to be acceptable. The MRL must be the same as that analyzed in the MRL check standard for those analytes that were biased low in the CCV. Adjust MRLs (if required), flag data and state the certainty in the case narrative where the sensitivity of the instrument was demonstrated at the MRL; therefore, results were not significantly affected.*
   b. *With the reporting limit adjusted to the next level in the calibration curve (typically 5 times higher) to prove the nonexistence of a false negative and note procedure in case narrative.*
4. If the acceptance criteria was exceeded (biased high) for the CCV and there were detectable results in a sample, the results may be "qualified" if the results exceeded the regulatory/decision limit (this is to be stated in the case narrative along with the data qualifiers or flags).

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17.4 Method Blank

- If an analyte in the blank is found to be out of control and the analyte is also found in associated samples, those sample results shall be "flagged" in the report and the method blank results reported.
- If the analyte is found in the blank but not in the sample then the results for the sample may be reported without a qualifier.

17.5 Laboratory Control Sample

All results associated with an out of control laboratory control sample must be reported with the appropriate data qualifier. An indication of whether the LCS was out high or low should also be included.

17.6 Surrogate

Report sample results with the appropriate data qualifier.

17.7 Laboratory Duplicate

All batch sample results associated with an out of control laboratory duplicate must be flagged with the appropriate data qualifier.

17.8 Internal Standard

All target analytes associated with an out of control internal standard must be flagged with the appropriate data qualifier.

17.9 Estimated Sample Results

17.9.1 Sample Hold Time All occurrences of missed holding times must be included on the final report including those samples received and/or analyzed outside of the specified hold times detailed in this SOP.

17.9.2 Matrix Interference Sample data associated with matrix interference must be flagged with the appropriate data qualifier.

17.9.3 Results Outside Initial Calibration Range All sample results not bracketed by initial calibration standards (within calibration range) must be reported as having less certainty by reporting with the appropriate data qualifier.

18) Training

18.1 Demonstration of Capability

All analysts must be trained in accordance with the guidelines detailed in the *SOP for Training Policy*. Demonstrations shall also be performed in accordance with the 2009 TNI Standards (Volume 1 Module 4 Section 1.6) and DoD Quality Systems Manual 4.2 (Requirement Box 25). Attachment A shall be used to document the training plan for new analysts' initial demonstration. Additionally, these demonstrations are performed anytime there is a change in instrument type, personnel or method.

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Once performance is found to be acceptable, a required certification statement must be completed by the QA Manager and either the immediate supervisor or Laboratory Manager and retained on file as a demonstration of compliance.

18.1.1 Quarterly Demonstration A demonstration of method sensitivity must be performed *quarterly on each instrument* performing this method.

1) A spike at the current LOD must be analyzed.
2) Verification of precision and bias at the LOQ must be performed.

Refer to Section 11.7.4.2 (LOQ) and 11.17.1 (LOD) for additional information on how these demonstrations are to be performed as well as the acceptance criteria.

18.1.2 Annual Demonstration Each analyst must perform this demonstration both initially and annually. Analyze four LCS standards at 1-4x the MRL (LOQ) either concurrently or over a period of days as a verification of precision and bias of the quantitation range. The standard deviation (n-1) and average percent recovery of the four replicates are compared against the method requirement for precision (±25%) and current laboratory control limits for bias/LCS.

18.1.3 Change in Personnel, Instruments, Method and/or Matrix The requirements in Sections 18.1.1 and 18.1.2 must be performed per the schedule noted and when there is a change in personnel, instruments, method or matrix. "Change" refers to any change in personnel, instrument, test method, or sample matrix that potentially affects the precision and bias, sensitivity, or selectivity of the output (e.g., a change in the detector, column type, matrix, or other components of the sample analytical system, or a method revision).

All completed attempts at this demonstration must be completed and turned into the QA department for retention.

19) Method Modifications

19.1 Method modifications are not allowed under NELAC\TNI standards; therefore, a statement, however worded, must be included in the final report indicating that data reported does not fall under the laboratory's NELAC certificate of approval. In addition, the following items are considered to be method modifications and must be reported accordingly.

- Sample collection in gas collection bags
- The pressurization of canisters with nitrogen or helium (if EPA Method 3C is requested) refer to Section 11.11.

20) Summary of Changes

20.1 SOP updated using ALS SOP Template – New cover page, document tracking/signature page, and table of contents page. Header/footer and font revised throughout SOP to align with ALS SOP template specifications. Sections reorganized as outlined in the *SOP for Establishing Standard Operating Procedures* and section references updated as necessary. Section titled "Instrument Specific Addendum" removed (Section 21.0 in previous revision - "Not Applicable" listed under section).

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Table of Contents	Updated
Section 1	Renamed section heading "Scope and Applicability"
Section 2	Renamed section heading "Summary of Procedure"
Section 4	Was Section 10 in previous revision
Section 5	Was Section 4 in previous revision
Section 6	Was Section 5 in previous revision
Section 7	Was Section 6 in previous revision
Section 8	Was Section 7 in previous revision
Section 8.5	First Bullet – Updated Column
Section 8.6	Added Windows 7
Section 9	Was Section 8 in previous revision
Section 10	Was Section 9 in previous revision
Section 11.1	Updated SOP title
Section 11.5.1	Edited setpoints to reflect current use
Section 11.7	#13 – 4th bullet – Added parentheses in last sentence
	#13 – 5th bullet – Added for clarification
Section 11.10.1	Added second paragraph
Section 11.12.1	Updated "25cc" to "20cc"
Section 11.16.1	Updated both SOP titles
	Removed reference to SOP attachment
Section 13.9.4	Updated file path
Section 16.1	Updated SOP title
Section 18	Was Section 19 in previous revision
	Renamed section heading "Training"
Section 18.1	Updated SOP title
	Updated "QA Program Manager" to "QA Manager"
Section 19	Was Section 20 in previous revision
Section 20	Was Section 22 in previous revision
Table 2	Updated MDL values
	Corrected m-,p-Xylenes CAS Number
Table 2A	Updated MDL values
Table 3	Updated values
Table 3A	Updated values
Table 4	Updated values
Table 4A	Updated values
Attachment A	Removed information at top of checklist covered in SOP header
	#4 – Updated 2nd, 3rd, and 5th SOP titles
Attachment B	Removed MS10 from list of instruments and added MS21
	#13 – Changed "Point" to "calibration level"
Attachment C	Revised Note in Parentheses at top of checklist
	Changed "CAS Project #" to "Project #"
	Removed MS10 from list of instruments and added MS21

21) References

21.1 EPA Method TO-14A, Compendium of Methods for the Determination of Toxic Organic Compounds in Ambient Air, EPA/625/R-96/010b, U.S. Environmental Protection Agency, Research Triangle Park, NC, January 1997.

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21.2 EPA Method TO-15, Compendium of Methods for the Determination of Toxic Organic Compounds in Ambient Air, EPA/625/R-96/010b, U.S. Environmental Protection Agency, Research Triangle Park, NC, January 1997.
21.3 Compendium of Methods for the Determination of Toxic Organic Compounds in Ambient Air, Second Edition, January 1999.
21.4 Compendium of Methods for the Determination of Toxic Organic Compounds in Ambient Air, Second Edition, Addendum, January 17, 2002.
21.5 National Environmental Laboratory Accreditation Conference, *2003 NELAC Standard*, June 5, 2003, EPA 600/R-04/003 and 2009 TNI Standards.
21.6 *Preparation of Gas Phase Standards for Ambient Air Analysis,* Tekmar-DOHRMANN Application Note, Spring 96, Vol. 6.5.
21.7 *Department of Defense Quality Systems Manual for Environmental Laboratories*, Version 4.2, 10/25/2010.
21.8 Arizona Administrative Code, Title 9. Health Services, Chapter 14. Department of Health Services Laboratories, December 31, 2006.
21.9 Florida Department of Environmental Protection, Chapter 62-160.
21.10 Minnesota Department of Health, 4740.2065, *Standard Operating Procedures*, Statutory Authority: MS s 144.97; 144.98; History: 31 SR 446, Posted: October 09, 2006, Revised April 16, 2010.

22) Attachments

22.1 Tables

Table 1: Instrument Tune Check Ion Abundance Criteria (TO-15)
Table 1A: Instrument Tune Check Ion Abundance Criteria (TO-14A)
Table 2: Volatile Organic Compounds, EPA Compendium Method TO-15 (SCAN)
Table 2A: Volatile Organic Compounds, EPA Compendium Method TO-15 (SIM)
Table 3: Standard Concentrations (SCAN) (Primary Sources)
Table 3A: Standard Concentrations (SIM) (Primary Sources)
Table 4: Standard Concentrations (SCAN) (Secondary Sources)
Table 4A: Standard Concentrations (SIM) (Secondary Sources)

22.2 Attachments

Attachment A: Training Plan for Analysis of TO-15 by GC/MS
Attachment B: TO-15 by GC/MS Initial Calibration Checklist
Attachment C: TO-15 by GC/MS Analyses Data Review Checklist
Attachment D: State and Project Specific Requirement

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TABLE 1

Required BFB Key Ions and
Ion Abundance Criteria for Method TO-15

Mass	Ion Abundance Criteria[1]
50	8.0 to 40.0 percent of m/e 95
75	30.0 to 66.0 percent of m/e 95
95	Base Peak, 100 Percent Relative Abundance
96	5.0 to 9.0 Percent of m/e 95
173	Less than 2.0 Percent of m/e 174
174	50.0 to 120.0 Percent of m/e 95
175	4.0 to 9.0 Percent of m/e 174
176	93.0 to 101.0 Percent of m/e 174
177	5.0 to 9.0 Percent of m/e 176

[1] 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.

TABLE 1A

Required BFB Key Ions and
Ion Abundance Criteria for Method TO-14A

Mass	Ion Abundance Criteria
50	15 to 40 percent of m/e 95
75	30 to 60 percent of m/e 95
95	Base Peak, 100 Percent Relative Abundance
96	5 to 9 Percent of m/e 95
173	Less than 2 Percent of m/e 174
174	>50 Percent of m/e 95
175	5 to 9 Percent of m/e 174
176	>95 and <101 Percent of m/e 174
177	5 to 9 Percent of m/e 176

Note: The criteria listed in Tables 1 and 1A shall be met or exceeded in order for EPA Compendium Methods TO-15 or TO-14A to be referenced.

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TABLE 2 - VOLATILE ORGANIC COMPOUNDS, EPA COMPENDIUM METHOD TO-15 (SCAN)								
Compound[1]	**CAS Number**	**Molecular Weight**	**Density**	**Primary Ion[2]**	**Secondary Ion(s)[2]**	**MRL[3] (µg/m³)**	**MDL[3] (µg/m³)**	**IS[4]**
Bromochloromethane (IS1)	74-97-5	-	-	130	128, 132	-	-	-
Propene	115-07-1	42.08	NA	42	39,41	0.50	0.13	IS1
Dichlorodifluoromethane (CFC 12)[5]	75-71-8	120.9	1.329	85	87, 101, 103	0.50	0.14	IS1
Chloromethane	74-87-3	50.49	0.911	50	52	0.50	0.15	IS1
1,2-Dichloro-1,1,2,2-tetrafluoroethane (Freon 114)	76-14-2	170.9	1.455	135	137	0.50	0.14	IS1
Vinyl Chloride	75-01-4	62.50	0.9106	62	64	0.50	0.15	IS1
1,3-Butadiene	106-99-0	54.09	0.6149	54	39, 53	0.50	0.16	IS1
Bromomethane	74-83-9	94.94	1.6755	94	96	0.50	0.14	IS1
Chloroethane	75-00-3	64.52	0.8902	64	66	0.50	0.14	IS1
Ethanol	64-17-5	46.07	0.7893	45	46	5.0	0.92	IS1
Acetonitrile	75-05-8	41.05	0.7857	41	40	0.50	0.24	IS1
Acrolein	107-02-8	56.06	0.840	56	55	2.0	0.20	IS1
Acetone	67-64-1	58.08	0.7845	58	43	5.0	1.1	IS1
Trichlorofluoromethane	75-69-4	137.4	NA	101	103	0.50	0.13	IS1
Isopropyl Alcohol	67-63-0	60.10	0.7809	45	43	5.0	0.34	IS1
Acrylonitrile	107-13-1	53.06	0.8060	53	52	0.50	0.18	IS1
1,1-Dichloroethene	75-35-4	96.94	1.213	96	61	0.50	0.16	IS1
tert-Butanol	75-65-0	74.12	0.7887	59	57,41,43	1.0	0.29	IS1
Methylene Chloride	75-09-2	84.94	1.3266	84	49	0.50	0.15	IS1
Allyl Chloride	107-05-1	76.53	0.9376	41	76	0.50	0.14	IS1
Trichlorotrifluoroethane	76-13-1	187.38	1.5635	151	101	0.50	0.20	IS1

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TABLE 2 (Continued) - VOLATILE ORGANIC COMPOUNDS, EPA COMPENDIUM METHOD TO-15 (SCAN)								
Compound[1]	**CAS Number**	**Molecular Weight**	**Density**	**Primary Ion[2]**	**Secondary Ion(s)[2]**	**MRL[3] (µg/m³)**	**MDL[3] (µg/m³)**	**IS[4]**
Carbon Disulfide	75-15-0	76.14	1.2632	76	78	5.0	0.13	IS1
trans-1,2-Dichloroethene	156-60-5	96.94	1.2565	61	96	0.50	0.15	IS1
1,1-Dichloroethane	75-34-3	98.96	1.1757	63	65	0.50	0.14	IS1
Methyl tert-Butyl Ether	1634-04-4	88.15	0.7402	73	57	0.50	0.14	IS1
Vinyl Acetate	108-05-4	86.09	0.9317	86	43	5.0	0.60	IS1
2-Butanone (MEK)	78-93-3	72.11	0.7999	72	43	5.0	0.20	IS1
cis-1,2-Dichloroethene	156-59-2	96.94	1.2837	61	96	0.50	0.16	IS1
Diisopropyl Ether	108-20-3	102.18	0.7241	87	45,59,43	0.50	0.16	IS1
Ethyl Acetate	141-78-6	88.106	0.9003	61	70	1.0	0.32	IS1
n-Hexane	110-54-3	86.18	0.6548	57	86	0.50	0.16	IS1
Chloroform	67-66-3	119.4	1.4832	83	85	0.50	0.14	IS1
1,2-Dichloroethane-d4(S)	17060-07-0	-	-	65	67	-	-	IS1
Tetrahydrofuran	109-99-9	72.11	0.8892	72	71,42	0.50	0.26	IS1
Ethyl tert-Butyl Ether	637-92-3	102.176	0.7519	87	59,57	0.50	0.15	IS1
1,2-Dichloroethane	107-06-2	98.96	1.2351	62	64	0.50	0.14	IS1
1,4-Difluorobenzene(IS2)	540-36-3	-	-	114	88	-	-	-
1,1,1-Trichloroethane	71-55-6	133.4	1.3390	97	99, 61	0.50	0.14	IS2
Isopropyl acetate	108-21-4	102.13	0.8718	61	87,43	1.0	0.29	IS2
1-Butanol	71-36-3	74.1224	0.8098	56	41	1.0	0.43	IS2
Benzene	71-43-2	78.11	0.8765	78	77	0.50	0.14	IS2
Carbon Tetrachloride	56-23-5	153.8	1.5940	117	119	0.50	0.15	IS2

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TABLE 2 (Continued) - VOLATILE ORGANIC COMPOUNDS, EPA COMPENDIUM METHOD TO-15 (SCAN)								
Compound[1]	**CAS Number**	**Molecular Weight**	**Density**	**Primary Ion[2]**	**Secondary Ion(s)[2]**	**MRL[3] (µg/m³)**	**MDL[3] (µg/m³)**	**IS[4]**
Cyclohexane	110-82-7	84.16	0.7739	84	69,56	1.0	0.26	IS2
tert-Amyl Methyl Ether	994-05-8	102.176	0.7703	73	87,55,43	0.50	0.15	IS2
1,2-Dichloropropane	78-87-5	113	1.1560	63	62	0.50	0.14	IS2
Bromodichloromethane	75-27-4	163.8	1.980	83	85	0.50	0.15	IS2
Trichloroethene	79-01-6	131.4	1.4642	130	132	0.50	0.14	IS2
1,4-Dioxane	123-91-1	88.11	1.0337	88	58	0.50	0.14	IS2
Isooctane	540-84-1	114.23	0.6877	57	41	0.50	0.13	IS2
Methyl Methacrylate	80-62-6	100.12	0.944	100	69	1.0	0.26	IS2
n-Heptane	142-82-5	100.2	0.6837	71	57,100	0.50	0.14	IS2
cis-1,3-Dichloropropene	10061-01-5	111	1.224	75	77	0.50	0.13	IS2
4-Methyl-2-Pentanone	108-10-1	100.2	0.7965	58	85	0.50	0.16	IS2
trans-1,3-Dichloropropene	10061-02-6	111	1.217	75	77	0.50	0.13	IS2
1,1,2-Trichloroethane	79-00-5	133.4	1.4397	97	83	0.50	0.13	IS2
Chlorobenzene-d5(IS3)	3114-55-4	-	-	82	117	-	-	-
Toluene-d8(S)	2037-26-5	-	-	98	100	-	-	IS3
Toluene	108-88-3	92.14	0.8669	91	92	0.50	0.14	IS3
2-Hexanone	591-78-6	100.16	0.8113	43	58	0.50	0.19	IS3
Dibromochloromethane	124-48-1	208.3	2.451	129	127	0.50	0.15	IS3
1,2-Dibromoethane	106-93-4	187.9	2.1791	107	109	0.50	0.15	IS3
n-Butyl Acetate	123-86-4	116.16	0.8825	43	56, 73	0.50	0.18	IS3
n-Octane	111-65-9	114.23	0.6986	57	114	0.50	0.14	IS3

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TABLE 2 (Continued) - VOLATILE ORGANIC COMPOUNDS, EPA COMPENDIUM METHOD TO-15 (SCAN)								
Compound[1]	**CAS Number**	**Molecular Weight**	**Density**	**Primary Ion[2]**	**Secondary Ion(s)[2]**	**MRL[3] (µg/m³)**	**MDL[3] (µg/m³)**	**IS[4]**
Tetrachloroethene	127-18-4	165.8	1.6227	166	164	0.50	0.12	IS3
Chlorobenzene	108-90-7	112.6	1.1058	112	114	0.50	0.15	IS3
Ethylbenzene	100-41-4	106.2	0.8670	91	106	0.50	0.15	IS3
m-, p-Xylenes	179601-23-1	106.2	0.8642, 0.8611	91	106	1.0	0.29	IS3
Bromoform	75-25-2	252.8	2.899	173	175	0.50	0.16	IS3
Styrene	100-42-5	104.1	0.9060	104	78, 103	0.50	0.15	IS3
o-Xylene	95-47-6	106.2	0.8802	91	106	0.50	0.16	IS3
n-Nonane	111-84-2	128.26	0.7176	43	57, 85	0.50	0.14	IS3
1,1,2,2-Tetrachloroethane	79-34-5	167.9	1.5953	83	85	0.50	0.15	IS3
4-Bromofluorobenzene(S)	460-00-4	-	-	174	176	-	-	IS3
Cumene	98-82-8	120.2	0.8618	105	120	0.50	0.14	IS3
alpha-Pinene	80-56-8	136.24	0.8582	93	77	0.50	0.14	IS3
n-Propylbenzene	103-65-1	120.1938	0.8670	91	120,65	0.50	0.17	IS3
3-Ethyltoluene	620-14-4	120.2	0.8645	105	120	0.50	0.16	IS3
4-Ethyltoluene	622-96-8	120.2	0.8614	105	120	0.50	0.15	IS3
1,3,5-Trimethylbenzene	108-67-8	120.2	0.8652	105	120	0.50	0.15	IS3
alpha-Methylstyrene	98-83-9	118.19	0.9106	118	103,117	0.50	0.21	IS3
2-Ethyltoluene	611-14-3	120.2	0.8807	105	120	0.50	0.15	IS3
1,2,4-Trimethylbenzene	95-63-6	120.2	0.8758	105	120	0.50	0.15	IS3
n-Decane	124-18-5	142.28	0.7300	57	71,85	0.50	0.15	IS3
Benzyl Chloride	100-44-7	126.59	1.1004	91	126	0.50	0.15	IS3

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TABLE 2 (Continued) - VOLATILE ORGANIC COMPOUNDS, EPA COMPENDIUM METHOD TO-15 (SCAN)								
Compound[1]	CAS Number	Molecular Weight	Density	Primary Ion[2]	Secondary Ion(s)[2]	MRL[3] (µg/m³)	MDL[3] (µg/m³)	IS[4]
1,3-Dichlorobenzene	541-73-1	147	1.2884	146	148	0.50	0.14	IS3
1,4-Dichlorobenzene	106-46-7	147	1.2475	146	148	0.50	0.15	IS3
sec-Butylbenzene	135-98-8	134.2206	0.8601	105	134,91	0.50	0.16	IS3
p-Isopropyltoluene	99-87-6	134.2206	0.8573	119	134,91	0.50	0.16	IS3
1,2,3-Trimethylbenzene	526-73-8	120.1938	0.8944	105	120	0.50	0.15	IS3
1,2-Dichlorobenzene	95-50-1	147	1.3059	146	148	0.50	0.16	IS3
d-Limonene	5989-27-5	136.24	0.8402	68	93	0.50	0.17	IS3
1,2,Dibromo-3-Chloropropane	96-12-8	236.33	2.093	157	75, 39	0.50	0.14	IS3
n-Undecane	1120-21-4	156.31	0.7402	57	71, 85	0.50	0.15	IS3
1,2,4-Trichlorobenzene	120-82-1	181.5	1.459	180	182, 184	0.50	0.18	IS3
Naphthalene	91-20-3	128.17	1.0253	128	129	0.50	0.21	IS3
n-Dodecane	112-40-3	170.34	0.7487	57	71,85	0.50	0.21	IS3
Hexachlorobutadiene	87-68-3	260.8	1.556	225	227	0.50	0.14	IS3
Cyclohexanone	108-94-1	98.14	0.9478	55	42, 98	0.50	0.19	IS3
tert-Butylbenzene	98-06-6	134.22	0.867	119	134	0.50	0.16	IS3
n-Butylbenzene	104-51-8	134.22	0.867	91	134	0.50	0.16	IS3

(S) = Surrogate (IS1) = Internal Standard 1 (IS2) = Internal Standard 2 (IS3) = Internal Standard 3
NA = Not Available

Note 1: Additional compounds may be reported as long as the minimum requirements of this document are met. The compounds listed in this table are reported using TO-15 SCAN. The Selected Ion Monitoring (SIM) compounds are a subset of this list and are included in Table 2A.

Note 2: These are suggested primary and secondary ions. However, any ions in the analyte spectra that are sufficient enough in response to reach the desired reporting limit and having a limited amount of interference, is acceptable for both the primary and secondary ion selection. Analyst experience should be utilized in determining appropriate ions.

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Note 3: The laboratory performs three concentration level analyses (SIM, SCAN and Low Level SCAN). The method reporting limit listed is the standard SCAN limit (at or above lowest concentration in the initial calibration curve), but may change with each new initial calibration performed. Therefore, current reporting limits for the three analysis levels, MRLs in ppbv, and those from the Low Level SCAN should be reviewed in the electronic TO-15 Method Manual.

Note 4: The listing of the internal standard by which the compounds are quantitated is for TO-15 SCAN only. SIM compounds (SCAN subset) and their corresponding ions and internal standards are listed in Table 2A.

Note 5: m/e 101 is ~10% or less of m/e 85 (the base peak) and may not be present for low level results. Retention times must be carefully verified.

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Table 2A – Volatile Organic Compounds, EPA Compendium Method TO-15 (SIM)					
Compound	**Primary Ion[1]**	**Secondary Ion[1]**	**MRL[2] (ug/m3)**	**MDL[2] (ug/m3)**	**IS**
Dichlorodifluoromethane	85	87	0.025	0.0024	IS1
Chloromethane	52	50	0.025	0.0031	IS1
Vinyl Chloride	62	64	0.025	0.0025	IS1
Bromomethane	94	96	0.025	0.0052	IS1
Chloroethane	64	66	0.025	0.0021	IS1
Acetone*	58	43	0.50	NA	IS1
Freon 11	101	103	0.025	0.0025	IS1
1,1-Dichloroethene	96	98,61	0.025	0.0031	IS1
Methylene Chloride	84	49	0.10	0.0095	IS1
Trichlorotrifluoroethane	151	153	0.025	0.0030	IS1
trans-1,2-Dichloroethene	96	98,61	0.025	0.011	IS1
1,1-Dichloroethane	63	65	0.025	0.0023	IS1
Methyl tert-Butyl Ether*	73	57	0.025	0.0038	IS1
cis-1,2-Dichloroethene	96	98,61	0.025	0.0098	IS1
Chloroform	83	85	0.10	0.0031	IS1
1,2-Dichloroethane	62	64	0.025	0.0087	IS1
1,1,1-Trichloroethane	97	99	0.025	0.0032	IS1
Benzene	78	77	0.075	0.0046	IS1
Carbon Tetrachloride	117	119	0.025	0.0029	IS1
1,2-Dichloropropane	63	62,76	0.025	0.0021	IS2
Bromodichloromethane	83	85	0.025	0.0036	IS2
Trichloroethene	130	132	0.025	0.0058	IS2
1,4-Dioxane*	88	58	0.10	0.0048	IS2
cis-1,3-Dichloropropene	75	77,39	0.025	0.0095	IS2
trans-1,3-Dichloropropene	75	77,39	0.025	0.015	IS2
1,1,2-Trichloroethane	83	97,61	0.10	0.0034	IS2
Toluene	91	92	0.10	0.0031	IS2
1,2-Dibromoethane	107	109	0.025	0.013	IS2
Tetrachloroethene	166	164	0.025	0.0028	IS2
Chlorobenzene	112	114	0.10	0.0066	IS3
Ethylbenzene	91	106	0.10	0.0046	IS3
m-&-p-Xylene	91	106	0.10	0.0097	IS3
o-Xylene	91	106	0.10	0.0044	IS3
1,1,2,2-Tetrachloroethane	83	85	0.025	0.0047	IS3
1,3-Dichlorobenzene	146	148	0.025	0.011	IS3
1,4-Dichlorobenzene	146	148	0.025	0.012	IS3
1,2-Dichlorobenzene	146	148	0.025	0.0087	IS3
1,2,4-Trichlorobenzene	182	184	0.025	0.019	IS3
Naphthalene	128	129	0.10	0.018	IS3
Hexachlorobutadiene*	225	227	0.025	0.0092	IS3

* Reported upon request NA = Not Available

(IS1) = Internal Standard 1 (IS2) = Internal Standard 2 (IS3) = Internal Standard 3

Note 1: These are suggested primary and secondary ions. However, any ions in the analyte spectra that is sufficient enough in response to reach the desired reporting limit and having a limited amount of interference, is acceptable for both the primary and secondary ion selection. Analyst experience should be utilized in determining appropriate ions.

Note 2: The method reporting limit listed is the standard SIM limit (lowest concentration in the initial calibration curve; must be higher than MDL), but may change with each new initial calibration performed. Therefore, current reporting limits should be reviewed. MDLs in ppbV may be reviewed in the electronic TO-15 Method Manual.

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Table 3
Standard Concentrations (SCAN) (Primary Sources)[1]

Compound Name	0.1ng	0.2ng	0.5ng	1.0ng	5.0ng	25ng	50ng	100ng
Bromochloromethane (IS1)	**25.0**	**25.0**	**25.0**	**25.0**	**25.0**	**25.0**	**25.0**	**25.0**
Propene	0.104	0.208	0.520	1.04	5.20	26.00	52.0	104
Dichlorodifluoromethane (CFC 12)	0.102	0.204	0.510	1.02	5.10	25.50	51.0	102
Chloromethane	0.099	0.198	0.495	0.99	4.95	24.75	49.5	99
1,2-Dichloro-1,1,2,2-tetrafluoroethane (Freon 114)	0.105	0.210	0.525	1.05	5.25	26.25	52.5	105
Vinyl Chloride	0.100	0.200	0.500	1.00	5.00	25.00	50.0	100
1,3-Butadiene	0.122	0.244	0.610	1.22	6.10	30.50	61.0	122
Bromomethane	0.100	0.200	0.500	1.00	5.00	25.00	50.0	100
Chloroethane	0.100	0.200	0.500	1.00	5.00	25.00	50.0	100
Ethanol	0.471	0.942	2.355	4.71	23.55	117.75	235.5	471
Acetonitrile	0.112	0.224	0.560	1.12	5.60	28.00	56.0	112
Acrolein	0.104	0.208	0.520	1.04	5.20	26.00	52.0	104
Acetone	0.539	1.078	2.695	5.39	26.95	134.75	269.5	539
Trichlorofluoromethane	0.102	0.204	0.510	1.02	5.10	25.50	51.0	102
Isopropyl Alcohol	0.189	0.378	0.945	1.89	9.45	47.25	94.5	189
Acrylonitrile	0.112	0.224	0.560	1.12	5.60	28.00	56.0	112
1,1-Dichloroethene	0.109	0.218	0.545	1.09	5.45	27.25	54.5	109
tert-Butanol	0.209	0.418	1.045	2.09	10.45	52.25	104.5	209
Methylene Chloride	0.107	0.214	0.535	1.07	5.35	26.75	53.5	107
Allyl Chloride	0.108	0.216	0.540	1.08	5.40	27.00	54.0	108
Trichlorotrifluoroethane	0.107	0.214	0.535	1.07	5.35	26.75	53.5	107
Carbon Disulfide	0.104	0.208	0.520	1.04	5.20	26.00	52.0	104
trans-1,2-Dichloroethene	0.105	0.210	0.525	1.05	5.25	26.25	52.5	105
1,1-Dichloroethane	0.104	0.208	0.520	1.04	5.20	26.00	52.0	104
Methyl tert-Butyl Ether	0.106	0.212	0.530	1.06	5.30	26.50	53.0	106
Vinyl Acetate	0.492	0.984	2.460	4.92	24.60	123.00	246.0	492
2-Butanone (MEK)	0.110	0.220	0.550	1.10	5.50	27.50	55.0	110
cis-1,2-Dichloroethene	0.108	0.216	0.540	1.08	5.40	27.00	54.0	108
Diisopropyl Ether	0.109	0.218	0.545	1.09	5.45	27.25	54.5	109
Ethyl Acetate	0.214	0.428	1.070	2.14	10.70	53.50	107.0	214
n-Hexane	0.105	0.210	0.525	1.05	5.25	26.25	52.5	105
Chloroform	0.108	0.216	0.540	1.08	5.40	27.00	54.0	108
1,2-Dichloroethane-d4 (S)	**25.0**	**25.0**	**25.0**	**25.0**	**25.0**	**25.0**	**25.0**	**25.0**
Tetrahydrofuran	0.109	0.218	0.545	1.09	5.45	27.25	54.5	109
Ethyl tert-Butyl Ether	0.105	0.210	0.525	1.05	5.25	26.25	52.5	105
1,2-Dichloroethane	0.105	0.210	0.525	1.05	5.25	26.25	52.5	105
1,4-Difluorobenzene(IS2)	**25.0**	**25.0**	**25.0**	**25.0**	**25.0**	**25.0**	**25.0**	**25.0**
1,1,1-Trichloroethane	0.103	0.206	0.515	1.03	5.15	25.75	51.5	103
Isopropyl acetate	0.218	0.436	1.090	2.18	10.90	54.50	109.0	218
1-Butanol	0.205	0.410	1.025	2.05	10.25	51.25	102.5	205

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Table 3 - Continued
Standard Concentrations (SCAN) (Primary Sources)[1]

Compound Name	0.1ng	0.2ng	0.5ng	1.0ng	5.0ng	25ng	50ng	100ng
Benzene	0.110	0.220	0.550	1.10	5.50	27.50	55.0	110
Carbon Tetrachloride	0.110	0.220	0.550	1.10	5.50	27.50	55.0	110
Cyclohexane	0.208	0.416	1.040	2.08	10.40	52.00	104.0	208
tert-Amyl Methyl Ether	0.105	0.210	0.525	1.05	5.25	26.25	52.5	105
1,2-Dichloropropane	0.103	0.206	0.515	1.03	5.15	25.75	51.5	103
Bromodichloromethane	0.106	0.212	0.530	1.06	5.30	26.50	53.0	106
Trichloroethene	0.103	0.206	0.515	1.03	5.15	25.75	51.5	103
1,4-Dioxane	0.108	0.216	0.540	1.08	5.40	27.00	54.0	108
Isooctane	0.103	0.206	0.515	1.03	5.15	25.75	51.5	103
Methyl Methacrylate	0.210	0.420	1.050	2.10	10.50	52.50	105.0	210
n-Heptane	0.105	0.210	0.525	1.05	5.25	26.25	52.5	105
cis-1,3-Dichloropropene	0.099	0.198	0.495	0.99	4.95	24.75	49.5	99
4-Methyl-2-Pentanone	0.109	0.218	0.545	1.09	5.45	27.25	54.5	109
trans-1,3-Dichloropropene	0.111	0.222	0.555	1.11	5.55	27.75	55.5	111
1,1,2-Trichloroethane	0.102	0.204	0.510	1.02	5.10	25.50	51.0	102
Chlorobenzene-d5 (IS3)	**25.0**	**25.0**	**25.0**	**25.0**	**25.0**	**25.0**	**25.0**	**25.0**
Toluene-d8 (S)	**25.0**	**25.0**	**25.0**	**25.0**	**25.0**	**25.0**	**25.0**	**25.0**
Toluene	0.106	0.212	0.530	1.06	5.30	26.50	53.0	106
2-Hexanone	0.120	0.240	0.600	1.20	6.00	30.00	60.0	120
Dibromochloromethane	0.113	0.226	0.565	1.13	5.65	28.25	56.5	113
1,2-Dibromoethane	0.105	0.210	0.525	1.05	5.25	26.25	52.5	105
n-Butyl Acetate	0.119	0.238	0.595	1.19	5.95	29.75	59.5	119
n-Octane	0.105	0.210	0.525	1.05	5.25	26.25	52.5	105
Tetrachloroethene	0.096	0.192	0.480	0.96	4.80	24.00	48.0	96
Chlorobenzene	0.106	0.212	0.530	1.06	5.30	26.50	53.0	106
Ethylbenzene	0.104	0.208	0.520	1.04	5.20	26.00	52.0	104
m- & p-Xylene	0.204	0.408	1.020	2.04	10.20	51.00	102.0	204
Bromoform	0.106	0.212	0.530	1.06	5.30	26.50	53.0	106
Styrene	0.106	0.212	0.530	1.06	5.30	26.50	53.0	106
o-Xylene	0.101	0.202	0.505	1.01	5.05	25.25	50.5	101
n-Nonane	0.103	0.206	0.515	1.03	5.15	25.75	51.5	103
1,1,2,2-Tetrachloroethane	0.098	0.196	0.490	0.98	4.90	24.50	49.0	98
4-Bromofluorobenzene (S)	**25.0**	**25.0**	**25.0**	**25.0**	**25.0**	**25.0**	**25.0**	**25.0**
Cumene	0.101	0.202	0.505	1.01	5.05	25.25	50.5	101
alpha-Pinene	0.098	0.196	0.490	0.98	4.90	24.50	49.0	98
n-Propylbenzene	0.101	0.202	0.505	1.01	5.05	25.25	50.5	101
3-Ethyltoluene	0.106	0.212	0.530	1.06	5.30	26.50	53.0	106
4-Ethyltoluene	0.106	0.212	0.530	1.06	5.30	26.50	53.0	106
1,3,5-Trimethylbenzene	0.107	0.214	0.535	1.07	5.35	26.75	53.5	107
alpha-Methylstyrene	0.105	0.210	0.525	1.05	5.25	26.25	52.5	105
2-Ethyltoluene	0.103	0.206	0.515	1.03	5.15	25.75	51.5	103
1,2,4-Trimethylbenzene	0.105	0.210	0.525	1.05	5.25	26.25	52.5	105

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Table 3 - Continued
Standard Concentrations (SCAN) (Primary Sources)[1]

Compound Name	0.1ng	0.2ng	0.5ng	1.0ng	5.0ng	25ng	50ng	100ng
n-Decane	0.103	0.206	0.515	1.03	5.15	25.75	51.5	103
Benzyl Chloride	0.107	0.214	0.535	1.07	5.35	26.75	53.5	107
1,3-Dichlorobenzene	0.107	0.214	0.535	1.07	5.35	26.75	53.5	107
1,4-Dichlorobenzene	0.107	0.214	0.535	1.07	5.35	26.75	53.5	107
sec-Butylbenzene	0.106	0.212	0.530	1.06	5.30	26.50	53.0	106
p-Isopropyltoluene	0.101	0.202	0.505	1.01	5.05	25.25	50.5	101
1,2,3-Trimethylbenzene	0.105	0.210	0.525	1.05	5.25	26.25	52.5	105
1,2-Dichlorobenzene	0.104	0.208	0.520	1.04	5.20	26.00	52.0	104
d-Limonene	0.106	0.212	0.530	1.06	5.30	26.50	53.0	106
1,2-Dibromo-3-Chloropropane	0.105	0.210	0.525	1.05	5.25	26.25	52.5	105
n-Undecane	0.106	0.212	0.530	1.06	5.30	26.50	53.0	106
1,2,4-Trichlorobenzene	0.106	0.212	0.530	1.06	5.30	26.50	53.0	106
Naphthalene	0.095	0.190	0.475	0.95	4.75	23.75	47.5	95
n-Dodecane	0.102	0.204	0.510	1.02	5.10	25.50	51.0	102
Hexachlorobutadiene	0.106	0.212	0.530	1.06	5.30	26.50	53.0	106
Methacrylonitrile	0.103	0.206	0.515	1.03	5.15	25.75	51.5	103
Cyclohexanone	0.096	0.192	0.480	0.96	4.80	24.00	48.0	96
tert-Butylbenzene	0.106	0.212	0.530	1.06	5.30	26.50	53.0	106
n-Butylbenzene	0.110	0.220	0.550	1.10	5.50	27.50	55.0	110

Note 1: The concentrations detailed in this table may change with each standard purchased or internally prepared. Refer to the appropriate initial calibration file, where necessary for the corresponding concentrations.

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Table 3A - Standard Concentrations (SIM) (Primary Sources)[1]

Compound Name	10pg	25pg	50pg	100pg	500pg	1000pg	2500pg	10,000pg	20,000pg
Freon-12	10.20	25.50	51.00	102.0	510	1020	2550	10200	20400
Chloromethane	9.90	24.75	49.50	99.0	495	990	2475	9900	19800
Vinyl Chloride	10.00	25.00	50.00	100.0	500	1000	2500	10000	20000
Bromomethane	10.00	25.00	50.00	100.0	500	1000	2500	10000	20000
Chloroethane	10.00	25.00	50.00	100.0	500	1000	2500	10000	20000
Acetone*	53.90	134.75	269.50	539.0	2695	5390	13475	53900	107800
Freon-11	10.20	25.50	51.00	102.0	510	1020	2550	10200	20400
1,1-Dichloroethene	10.90	27.25	54.50	109.0	545	1090	2725	10900	21800
Methylene Chloride	10.70	26.75	53.50	107.0	535	1070	2675	10700	21400
Freon-113	10.70	26.75	53.50	107.0	535	1070	2675	10700	21400
trans-1,2-Dichloroethene	10.50	26.25	52.50	105.0	525	1050	2625	10500	21000
1,1-Dichloroethane	10.40	26.00	52.00	104.0	520	1040	2600	10400	20800
Methyl tert-Butyl Ether*	10.60	26.50	53.00	106.0	530	1060	2650	10600	21200
cis-1,2-Dichloroethene	10.80	27.00	54.00	108.0	540	1080	2700	10800	21600
Chloroform	10.80	27.00	54.00	108.0	540	1080	2700	10800	21600
1,2-Dichloroethane	10.50	26.25	52.50	105.0	525	1050	2625	10500	21000
1,1,1-Trichloroethane	10.30	25.75	51.50	103.0	515	1030	2575	10300	20600
Benzene	11.00	27.50	55.00	110.0	550	1100	2750	11000	22000
Carbon Tetrachloride	11.00	27.50	55.00	110.0	550	1100	2750	11000	22000
1,2-Dichloropropane	10.30	25.75	51.50	103.0	515	1030	2575	10300	20600
Trichloroethene	10.30	25.75	51.50	103.0	515	1030	2575	10300	20600
Bromodichloromethane	10.60	26.50	53.00	106.0	530	1060	2650	10600	21200
1,4-Dioxane*	10.80	27.00	54.00	108.0	540	1080	2700	10800	21600
cis-1,3-Dichloropropene	9.90	24.75	49.50	99.0	495	990	2475	9900	19800
trans-1,3-Dichloropropene	11.10	27.75	55.50	111.0	555	1110	2775	11100	22200
1,1,2-Trichloroethane	10.20	25.50	51.00	102.0	510	1020	2550	10200	20400
Toluene	10.60	26.50	53.00	106.0	530	1060	2650	10600	21200
1,2-Dibromoethane	10.50	26.25	52.50	105.0	525	1050	2625	10500	21000
Tetrachloroethene	9.60	24.00	48.00	96.0	480	960	2400	9600	19200
Chlorobenzene	10.60	26.50	53.00	106.0	530	1060	2650	10600	21200
Ethylbenzene	10.40	26.00	52.00	104.0	520	1040	2600	10400	20800
m,p-Xylenes	20.40	51.00	102.00	204.0	1020	2040	5100	20400	40800
o-Xylene	10.10	25.25	50.50	101.0	505	1010	2525	10100	20200
1,1,2,2-Tetrachloroethane	9.80	24.50	49.00	98.0	490	980	2450	9800	19600
1,3-Dichlorobenzene	10.70	26.75	53.50	107.0	535	1070	2675	10700	21400
1,4-Dichlorobenzene	10.70	26.75	53.50	107.0	535	1070	2675	10700	21400
1,2-Dichlorobenzene	10.40	26.00	52.00	104.0	520	1040	2600	10400	20800
1,2-Dibromo-3-chloropropane	10.50	26.25	52.50	105.0	525	1050	2625	10500	21000
1,2,4-Trichlorobenzene	10.60	26.50	53.00	106.0	530	1060	2650	10600	21200
Naphthalene	9.50	23.75	47.50	95.0	475	950	2375	9500	19000
Hexachloro-1,3-butadiene*	10.60	26.50	53.00	106.0	530	1060	2650	10600	21200

*Reported upon request.

Note 1: The concentrations detailed in this table may change with each standard purchased or internally prepared. Refer to the appropriate initial calibration file, where necessary for the corresponding concentrations.

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Table 4 - Standard Concentrations (SCAN) (Secondary Sources)[1]

Compound Name	25ng	Compound Name	25ng	Compound Name	25ng
Bromochloromethane (IS1)	**25.0**	1,1,1-Trichloroethane	25.50	alpha-Pinene	24.00
Propene	25.50	Isopropyl acetate	55.00	n-Propylbenzene	24.75
Dichlorodifluoromethane (CFC 12)	25.25	1-Butanol	52.25	3-Ethyltoluene	25.50
Chloromethane	24.50	Benzene	26.00	4-Ethyltoluene	25.50
1,2-Dichloro-1,1,2,2-tetrafluoroethane (Freon 114)	25.75	Carbon Tetrachloride	26.50	1,3,5-Trimethylbenzene	26.00
Vinyl Chloride	25.00	Cyclohexane	50.25	alpha-Methylstyrene	25.50
1,3-Butadiene	26.25	tert-Amyl Methyl Ether	25.75	2-Ethyltoluene	25.25
Bromomethane	25.00	1,2-Dichloropropane	25.50	1,2,4-Trimethylbenzene	25.00
Chloroethane	25.25	Bromodichloromethane	25.50	n-Decane	25.00
Ethanol	119.75	Trichloroethene	24.75	Benzyl Chloride	25.75
Acetonitrile	25.25	1,4-Dioxane	25.75	1,3-Dichlorobenzene	25.75
Acrolein	25.50	Isooctane	25.25	1,4-Dichlorobenzene	26.50
Acetone	129.50	Methyl Methacrylate	51.75	sec-Butylbenzene	26.00
Trichlorofluoromethane	26.25	n-Heptane	25.25	p-Isopropyltoluene	24.25
Isopropyl Alcohol	49.50	cis-1,3-Dichloropropene	24.50	1,2,3-Trimethylbenzene	26.00
Acrylonitrile	25.75	4-Methyl-2-Pentanone	26.25	1,2-Dichlorobenzene	25.50
1,1-Dichloroethene	27.25	trans-1,3-Dichloropropene	27.25	d-Limonene	25.75
tert-Butanol	51.75	1,1,2-Trichloroethane	25.25	1,2-Dibromo-3-Chloropropane	25.25
Methylene Chloride	26.50	**Chlorobenzene-d5 (IS3)**	25.0	n-Undecane	25.50
Allyl Chloride	26.75	**Toluene-d8 (S)**	**25.0**	1,2,4-Trichlorobenzene	25.00
Trichlorotrifluoroethane	26.50	Toluene	26.00	Naphthalene	22.25
Carbon Disulfide	26.00	2-Hexanone	28.50	n-Dodecane	22.75
trans-1,2-Dichloroethene	25.25	Dibromochloromethane	27.00	Hexachlorobutadiene	26.00
1,1-Dichloroethane	25.75	1,2-Dibromoethane	26.00	Methacrylonitrile	25.75
Methyl tert-Butyl Ether	25.50	Butyl Acetate	28.50	Cyclohexanone	23.75
Vinyl Acetate	123.50	n-Octane	25.75	tert-Butylbenzene	26.50
2-Butanone (MEK)	26.50	Tetrachloroethene	23.75	n-Butylbenzene	26.50
cis-1,2-Dichloroethene	26.75	Chlorobenzene	26.00		
Diisopropyl Ether	26.50	Ethylbenzene	25.75		
Ethyl Acetate	51.50	m- & p-Xylene	51.50		
n-Hexane	25.75	Bromoform	27.00		
Chloroform	27.75	Styrene	26.00		
1,2-Dichloroethane-d4 (S)	**25.0**	o-Xylene	25.00		
Tetrahydrofuran	26.00	n-Nonane	25.25		
Ethyl tert-Butyl Ether	25.50	1,1,2,2-Tetrachloroethane	24.75		
1,2-Dichloroethane	26.00	**4-Bromofluorobenzene (S)**	**25.0**		
1,4-Difluorobenzene(IS2)	**25.0**	Cumene	24.50		

Note 1: The concentrations detailed in this table may change with each standard purchased or internally prepared. Refer to the appropriate initial calibration file, where necessary for the corresponding concentrations.

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Table 4A – ICV/LCS Standard Concentrations (SIM) (Secondary Sources)[1]

Compound Name	500pg
Freon-12	505
Chloromethane	490
Vinyl Chloride	500
Bromomethane	500
Chloroethane	505
Acetone*	2590
Freon-11	525
1,1-Dichloroethene	545
Methylene Chloride	530
Freon-113	530
trans-1,2-Dichloroethene	505
1,1-Dichloroethane	515
Methyl tert-Butyl Ether*	510
cis-1,2-Dichloroethene	535
Chloroform	555
1,2-Dichloroethane	520
1,1,1-Trichloroethane	510
Benzene	520
Carbon Tetrachloride	530
1,2-Dichloropropane	510
Trichloroethene	495
Bromodichloromethane	510
1,4-Dioxane*	515
cis-1,3-Dichloropropene	490
trans-1,3-Dichloropropene	545
1,1,2-Trichloroethane	505
Toluene	520
1,2-Dibromoethane	520
Tetrachloroethene	475
Chlorobenzene	520
Ethylbenzene	515
m,p-Xylenes	1030
o-Xylene	500
1,1,2,2-Tetrachloroethane	495
1,3-Dichlorobenzene	515
1,4-Dichlorobenzene	530
1,2-Dichlorobenzene	510
1,2-Dibromo-3-chloropropane	505
1,2,4-Trichlorobenzene	500
Naphthalene	445
Hexachloro-1,3-butadiene*	520

*Report upon request

Note 1: The concentrations detailed in this table may change with each standard purchased or internally prepared. Refer to the appropriate initial calibration file, where necessary for the corresponding concentrations.

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ATTACHMENT A

Training Plan for Analysis of VOCs by GC/MS

Training Plan for Analysis of VOCs by GC/MS

Trainee _____ Trainer _____ Instrument _____ Training Completion Date _____

1. Read SOP *Training Duration* _____ Trainer ____ Trainee ____ Date _____

2. Read Methods TO-14A & TO-15A *Training Duration* _____ Trainer ____ Trainee ____ Date _____

3. Demonstrated understanding of the scientific basis of the analysis Trainer ____ Trainee ____ Date _____
   Whole air sample preconcentration techniques
   Gas chromatography *Training Duration* _____
   Mass spectrometry

4. Demonstrated familiarity with related SOPs Trainer ____ Trainee ____ Date _____
   SOP for Batches and Sequences; Rev. ___
   SOP for Making Entries onto Analytical Records; Rev. ___ *Training Duration* _____
   SOP for Manual Integration Policy; Rev. ____
   SOP for Significant Figures; Rev. ____
   SOP for Nonconformance and Corrective Action; Rev. ____
   SOP for Performing MDL Studies and Establishing Limits of Detection and Quantitation; Rev. ____
   SOP for Cleaning and Certification of Summa Canisters; Rev. ____

5. Observe performance of SOP *Training Duration* _____ Trainer ____ Trainee ____ Date _____
   ___sample preparation/dilution and sample loading and analysis
   ___analytical sequence setup
   ___standard preparation
   ___BFB tuning evaluation
   ___initial calibration (model, calculations, manual integrations)/initial calibration verification
   ___manual integrations
   ___continuing calibration verification
   ___EnviroQuant introduction (recognizing saturation and sensitivity issues)
   ___data reduction and reporting including reporting req. for various agencies, autotexts, documentation
   ___canister and bag handling (including leakers)

6. Perform SOP with supervision *Training Duration* _____ Trainer ____ Trainee ____ Date _____
   ___sample preparation/dilution and sample loading and analysis
   ___analytical sequence setup
   ___standard preparation
   ___BFB tuning evaluation
   ___initial calibration (model, calculations, manual integrations)/initial calibration verification
   ___manual integrations
   ___continuing calibration verification
   ___EnviroQuant use (recognizing saturation and sensitivity issues)
   ___data reduction and reporting including reporting req. for various agencies, autotexts, documentation
   ___canister and bag handling (including leakers)

7. Independent performance of the SOP *Training Duration* _____ Trainer ____ Trainee ____ Date _____
   ___sample preparation/dilution and sample loading and analysis
   ___analytical sequence setup
   ___standard preparation
   ___BFB tuning evaluation
   ___initial calibration (model, calculations, manual integrations)/initial calibration verification
   ___manual integrations
   ___continuing calibration verification
   ___EnviroQuant proficiency (recognizing saturation and sensitivity issues)
   ___data reduction and reporting including reporting req. for various agencies, autotexts, documentation
   ___canister and bag handling (including leakers)
   ___initial demonstration of competency (4 Laboratory Control Samples)

8. Instrument operation and maintenance Trainer ____ Trainee ____ Date _____
   ___autosampler *Training Duration* _____
   ___GC and capillary column installation *Training Duration* _____
   ___mass spectrometer *Training Duration* _____
   ___data system *Training Duration* _____

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ATTACHMENT B

Initial Calibration Review Checklist

Initial Calibration Review Checklist - EPA Compendium Method TO-15

ICAL Date:_____ ICAL ID:_____ LIMS ICAL ID:_____

Instrument: ☐ MS3 ☐ MS7 ☐ MS8 ☐ MS9 ☐ MS11 ☐ MS13 ☐ MS16 ☐ MS19 ☐ MS21

Mode: ☐ SIM ☐ Scan Scan Low Level (0.1ng): ☐ Yes ☐ No

Analyst **Reviewer**

☐ 1. Is the required documentation in the ICAL file? ..... ☐
- ☐ BFB Tune analysis Report ..... ☐
- ☐ Calibration Status Report (aka Calibration History) ..... ☐
- ☐ Response Factor Report/Percent RSD ..... ☐
- ☐ Quantitation Report for each calibration standard (including manual integration documentation) ..... ☐
- ☐ ICV Quantitation Report ..... ☐
- ☐ TO-15 Standard Calculation Spreadsheet ..... ☐

☐ 2. Was the ICAL performed continuously (not interrupted for maintenance or sample analysis)? ..... ☐

☐ 3. Have all the calibration standards been analyzed within 24 hours of each other? ..... ☐

☐ 4. Does the BFB tune check standard analysis at the start meet the tune criteria? ..... ☐

☐ 5. Are all the analytes in the blank analysis <MRL? ..... ☐

☐ 6. Does each analyte's ICAL include a minimum of 5 concentrations at 5 consecutive levels? ..... ☐

☐ 7. Were the standards analyzed from low concentration to high concentration? ..... ☐

☐ 8. For each analyte, are there no levels skipped? ..... ☐

☐ 9. For each analyte, is there only one value used for each calibration level? ..... ☐

☐ 10. For each analyte, is the lowest standard's concentration at or below the analyte's MRL? ..... ☐

☐ 11. For each analyte, is the corresponding signal to noise ratio at least 3:1 at the lowest point on the curve? ..... ☐

☐ 12. For each analyte, are the corresponding upper levels free from saturation? ..... ☐

☐ 13. If a calibration level is dropped, are all the responses for each target analyte dropped and is the information noted in the ICAL explaining the reason? ..... ☐

☐ 14. Is the average RSD ≤30% for all analytes, with no more than two exceptions ≤40%? ..... ☐

☐ 15. Is the response Y at each calibration level within 40% of the mean area response over the initial calibration range for each internal standard? ..... ☐

☐ 16. Percent recovery for each analyte in the ICV 70%-130% (50-150% for VA, unless AFCEE or DoD)? ..... ☐

☐ 17. Was the RRT for each target compound at each calibration level within 0.06RRT units of the mean RRT for the compound? ..... ☐

☐ 18. Is the retention time shift for each of the internal standards at each calibration level within 20s of the mean retention time over the initial calibration range for each standard? ..... ☐

☐ 19. If there are any manual integrations, are they performed correctly according to the corresponding SOP? If so, initial and date the appropriate pages. ..... ☐

☐ 20. Is the ICAL good at 0.5ng (or 0.1ng) – 100ng (Scan) or 10-20000pg (SIM) for all compounds? ☐ Yes ☐ No
If not, note exceptions and the corresponding MRLs below – *Specify applicable range...* ..... ☐

☐ 21. Are ALL of the peak selections for each analyte correct according to retention time (all RTs must be checked by both the initial and peer reviewer)? ..... ☐

COMMENTS:

Analyst:_____ Date: _____

Secondary Reviewer: _____ Date: _____

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ATTACHMENT C

Data Review Checklist

EPA Compendium Method TO-15 - Data Review Checklist

(Note exceptions in Comments Section and attach Analysis Observations / Case Narrative Summary Form as appropriate)

Method: ☐ EPA TO-15 ☐ EPA TO-14A **Analysis Date:** _____ **Project #:** _____

Instrument: ☐ MS3 ☐ MS7 ☐ MS8 ☐ MS9 ☐ MS11 ☐ MS13 ☐ MS16 ☐ MS19 ☐ MS21

Mode: ☐ SIM ☐ Scan **Scan Low Level (0.1ng):** ☐ Yes ☐ No **DOD:** ☐ Yes ☐ No

Analyst **Reviewer**

☐ 1. Is the required documentation present? ..... ☐
- ☐ CORRECT BFB Tune analysis Report
- ☐ CCV analysis Quantitation Report & %D Report
- ☐ Duplicate analysis Quantitation Report
- ☐ Quantitation Report for each sample included
- ☐ Manual Integration & Q Deletion Documentation
- ☐ LCS analysis Quantitation Report
- ☐ MB analysis Quantitation Report
- ☐ Spectral match details for each hit, where necessary

☐ 2. Does the BFB **tune** check standard analysis meet the tune criteria for the method indicated above? ..... ☐

☐ 3. Are all analyses within the tune's **24-hr window or** ☐ **Client's 12hr window requirement**? ..... ☐

☐ 4. Does the **CCV** have a difference ≤30% for all analytes? ..... ☐
[Note all outliers biased high and/or low]

☐ 5. Are all the **IS** retention times within 20 seconds of the CCV RT or the RT from the midpoint (ICAL)? ..... ☐

☐ 6. Are all the **IS** responses within ±40% of CCV or the midpoint in the ICAL? ..... ☐

☐ 7. Are all the **surrogate** recoveries (in CCVs, MB, LCSs, etc.) within acceptance limits (70%-130%) ..... ☐

☐ 8. Are all the analytes in the **MB** <MRL? (DoD <1/2MRL, except Acetone, MeCl2, EtOH, Carbon Disulfide)? .... ☐

☐ 9. Is the **LCS** %R within the lab control limits for all analytes except AZ samples (70%-130%, VA 50%-150%)? . ☐

☐ 10. Are all the analytes in the **Lab Duplicate / DLCS** within ±25% or the client specified limits? ..... ☐

☐ 11. Are all the analyte hits in the samples within the **calibration range** and/or noted? ..... ☐

☐ 12. Are all **peak integrations** acceptable? ..... ☐

☐ 13. Are all **manual integrations** flagged and documented? ..... ☐

☐ 14. Are ALL of the peak selections for each analyte correct according to **retention time**? ..... ☐

☐ 15. Are all the Q Deletions correct? ..... ☐

☐ 16. Are all **calculations** correct? ..... ☐

☐ 17. Has the analyst initialed and dated each **quantitation report**? ..... ☐

☐ 18. For **TICs** are the relative intensity and other requirements met? ..... ☐

☐ 19. **Auto report** correct? ..... ☐

☐ 20. **MRL** = _____ ☐ ug/m3 ☐ pg (ethanol, acetone, vinyl acetate = 5.0ug/m3) ..... ☐

☐ 21. Pressurized with **Helium**? Is the worksheet completed for all samples? ..... ☐

☐ 22. Report to **MDL**? ☐ Yes ☐ No ..... ☐

☐ 23. **Global Minimum Detection Limit** = _____ ☐ ug/m3 ☐ pg ..... ☐

☐ 24. **DOD:** Are **manual integrations** notated in the **case narrative?** ..... ☐

COMMENTS:

☐ LIMS Run Approval ☐ LIMS Supervisor Approval

Analyst: _____ Secondary Reviewer: _____

Date: _____ Date: _____

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ATTACHMENT D

State and Project Specific Requirements

Minnesota Requirements	
Item	**Criteria**
Holding Time (HT)	14 days
Tedlar bags	Not allowed for sampling or sample dilution
Canisters and flow controllers	Individually certified Individually leak checked before shipment
	Samples with concentrations outside of the calibration curve will have a zero canister analysis performed to check for carryover. If carryover is detected, system bake out shall be performed and documented. Additionally, in instances where the laboratory has evidence on file that a particular compound when present at a high concentration does not exhibit carry-over, the samples will not be reanalyzed. When samples are analyzed that have a higher concentration than the evidence on file, the above requirements must be followed. Also, samples that have hits below the MRL will not be reanalyzed when analyzed after a sample with concentrations over the calibration range.
Method Reporting Verification Check	Analyze a Method Reporting Verification at the beginning of the sequence prior to analyzing samples. Acceptance criteria ±40%.
Duplicates	10 percent laboratory duplicates
Record retention	MN/NELAC 5 years MPCA (Minnesota Pollution Control Agency) compliant samples 10 years
Tier level	TIII

Arizona Requirements	
Item	**Criteria**
LCS	70-130% (vinyl acetate 50-150%)

Department of Toxic Substances Control (DTSC) Requirements	
Item	**Criteria**
Holding Time (HT)	72 hour hold time for canisters

EPA Region 9 Requirements	
Item	**Criteria**
Holding Time (HT)	14 days

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APPENDIX C

OCCUPIED DWELLING QUESTIONNAIRE

OCCUPIED DWELLING QUESTIONNAIRE

Indoor Air Assessment Survey

Date: _____

1. Name:_____

   Address:_____

   _____

   Home Phone:_____ Work Phone:_____

2. What is the best time to call to speak with you?_____ At: Work ❑ or Home ❑?

3. Are you the Owner ❑, Renter ❑, Other ❑ (please specify)_____ of this Home/Structure?

4. Total number of occupants/persons at this location?_____
   Number of children? _____ Ages?_____

5. How long have you lived at this location? _____

General Home Description

6. Type of Home/Structure (check only one): Single Family Home ❑, Duplex ❑, Condominium❑, Townhouse ❑, Other ❑_____

7. Home/Structure Description: number of floors _____
   Basement? Yes ❑ No ❑
   Crawl Space? Yes ❑ No ❑
   If Yes, under how much of the house's area? ____%

8. Age of Home/Structure: _____ years, Not sure/Unknown ❑

9. General Above-Ground Home/Structure construction (check all that apply):
   Wood ❑, Brick ❑, Concrete ❑, Cement block ❑, Other ❑_____

10. Foundation Construction (check all that apply):
    Concrete slab ❑
    Fieldstone ❑
    Concrete block ❑

Elevated above ground/grade ❑
Other_____

11. What is the source of your drinking water (check all that apply)?
Public water supply ❑
Private well ❑
Bottled water ❑
Other, please specify _____

12. Do you have a private well for purposes other than drinking?
Yes ❑ No ❑
If yes, please describe what you use the well for:_____
_____

13. Do you have a septic system? Yes ❑ No ❑ Not used ❑ Unknown ❑

14. Do you have standing water outside your home (pond, ditch, swale)? Yes ❑ No ❑

Basement Description, please check appropriate boxes.
<u>If you do not have a basement</u> go to question 23.

15. Is the basement finished ❑ or unfinished ❑?
16. If finished, how many rooms are in the basement?_____
How many are used for more than 2 hours/day?_____
17. Is the basement floor (check all that apply) concrete ❑, tile ❑, carpeted ❑, dirt ❑, other❑(describe)_____?
18. Are the basement walls poured concrete ❑, cement block ❑, stone ❑, wood ❑, brick ❑, other❑_____?
19. Does the basement have a moisture problem (check one only)?
Yes, frequently (3 or more times/yr) ❑
Yes, occasionally (1-2 times/yr) ❑
Yes, rarely (less than 1 time/yr) ❑
No ❑

20. Does the basement ever flood (check one only)?
Yes, frequently (3 or more times/yr) ❑
Yes, occasionally (1-2 times/yr) ❑
Yes, rarely (less than 1 time/yr) ❑
No ❑

21. Does the basement have any of the following? (check all that apply) Floor cracks ❑, Wall cracks ❑, Sump ❑, Floor drain ❑, Other hole/opening in floor ❑ (describe)_____

22. Are any of the following used or stored in the basement (check all that apply)
Paint ❑ Paint stripper/remover ❑ Paint thinner ❑
Metal degreaser/cleaner ❑ Gasoline ❑ Diesel fuel ❑ Solvents ❑ Glue ❑
Laundry spot removers ❑ Drain cleaners ❑ Pesticides ❑

23. Have you recently (within the last six months) done any painting or remodeling in your home? Yes ❑ No ❑
If yes, please specify what was done, where in the home, and what month:
_____
_____

24. Have you installed new carpeting in your home within the last year? Yes ❑ No ❑
If yes, when and where?_____

25. Do you regularly use or work in a dry cleaning service (check only one box)?
Yes, use dry-cleaning regularly (at least weekly)❑
Yes, use dry-cleaning infrequently (monthly or less)❑
Yes, work at a dry cleaning service ❑
No ❑

26. Does anyone in your home use solvents at work?
Yes ❑ If yes, how many persons_____
No ❑ If no, go to question 28

27. If yes for question 26 above, are the work clothes washed at home? Yes ❑ No ❑

28. Where is the washer/dryer located?
Basement ❑
Upstairs utility room ❑
Kitchen ❑
Garage ❑
Use a Laundromat ❑
Other, please specify ❑_____

29. If you have a dryer, is it vented to the outdoors? Yes ❑ No ❑

30. What type(s) of home heating do you have (check all that apply)
Fuel type: Gas ❑, Oil ❑, Electric ❑, Wood ❑, Coal ❑, Other_____
Heat conveyance system: Forced hot air ❑
Forced hot water ❑
Steam ❑
Radiant floor heat ❑
Wood stove ❑
Coal furnace ❑
Fireplace ❑
Other_____

31. Do you have air conditioning? Yes ❑ No ❑. If yes, please check the appropriate type(s)
Central air conditioning ❑
Window air conditioning unit(s)❑
Other ❑, please specify_____

32. Do you use any of the following? Room fans ❑, Ceiling fans ❑, Attic fan ❑
Do you ventilate using the fan-only mode of your central air conditioning or forced air heating system? Yes ❑ No ❑

33. Has your home had termite or other pesticide treatment: Yes ❑ No ❑ Unknown ❑
If yes, please specify type of pest controlled, _____
and approximate date of service _____

34. Water Heater Type: Gas ❑, Electric ❑, By furnace ❑, Other ❑_____
Water heater location: Basement ❑, Upstairs utility room ❑, Garage ❑, Other ❑ (please describe) _____

35. What type of cooking appliance do you have? Electric ❑, Gas ❑, Other ❑_____

36. Is there a stove exhaust hood present? Yes ❑ No ❑
Does it vent to the outdoors? Yes ❑ No ❑

37. Smoking in Home:
None ❑, Rare (only guests)❑, Moderate (residents light smokers)❑,
Heavy (at least one heavy smoker in household)❑

38. If yes to above, what do they smoke?
Cigarettes ❑ Cigars ❑
Pipe ❑ Other ❑

39. Do you regularly use air fresheners? Yes ❑ No ❑

40. Does anyone in the home have indoor home hobbies of crafts involving: None ❑
Heating ❑, soldering ❑, welding ❑, model glues ❑, paint ❑, spray paint, wood finishing ❑, Other ❑ Please specify what type of hobby: _____
_____

41. General family/home use of consumer products (please circle appropriate): Assume that **Never** = never used, **Hardly ever** = less than once/month, **Occasionally** = about once/month, **Regularly** = about once/week, and **Often** = more than once/week.

Product	Frequency of Use				
Spray-on deodorant	Never	Hardly ever	Occasionally	Regularly	Often

Aerosol deodorizers	Never	Hardly ever	Occasionally	Regularly	Often
Insecticides	Never	Hardly ever	Occasionally	Regularly	Often
Disinfectants	Never	Hardly ever	Occasionally	Regularly	Often

(Question 41, continued)

Product	Frequency of Use				
Window cleaners	Never	Hardly ever	Occasionally	Regularly	Often
Spray-on oven cleaners	Never	Hardly ever	Occasionally	Regularly	Often
Nail polish remover	Never	Hardly ever	Occasionally	Regularly	Often
Hair sprays	Never	Hardly ever	Occasionally	Regularly	Often

42. Please check weekly household cleaning practices:
    Dusting ❑
    Dry sweeping ❑
    Vacuuming ❑
    Polishing (furniture, etc) ❑
    Washing/waxing floors ❑
    Other ❑_____

43. Other comments: _____
_____
_____
_____
_____
_____
_____
_____
_____
_____