BASIC REMEDIATION COMPANY
STANDARD OPERATING PROCEDURES
BMI COMMON AREAS
CLARK COUNTY, NEVADA

SOP-16

FLUX CHAMBER SOURCE TESTING

CE Schmidt Revision Dated 02/19/2008

STANDARD OPERATING PROCEDURES

SOP-16
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DISCLAIMER

THE FOLLOWING STANDARD OPERATING PROCEDURE PROVIDES GENERAL GUIDANCE FOR BRC CONTRACTORS FOR TECHNICAL ISSUES ADDRESSED DURING ENVIRONMENTAL SITE INVESTIGATION AND REMEDIATION ACTIVITIES. IT IS NOTED, HOWEVER, THAT EACH SITE IS UNIQUE AND THESE GUIDELINES ARE NOT A SUBSTITUTE FOR COMMON SENSE AND GOOD MANAGEMENT PRACTICES BASED ON PROFESSIONAL TRAINING AND EXPERIENCE. IN ADDITION, INDIVIDUAL CONTRACT TERMS MAY AFFECT THE IMPLEMENTATION OF THIS STANDARD OPERATING PROCEDURE. BRC CONTRACTORS RESERVE THE UNRESTRICTED RIGHT TO CHANGE, MODIFY OR NOT APPLY THESE GUIDELINES IN THEIR SOLE, COMPLETE, AND UNRESTRICTED DISCRETION TO MEET CERTAIN CIRCUMSTANCES, CONTRACTUAL REQUIREMENTS, SITE CONDITIONS, OR JOB REQUIREMENTS.

1.0 INTRODUCTION

This Standard Operating Procedure (SOP) is a guidance document that describes the sampling and analytical methodology prescribed for performing an Air Pathway Analysis (APA). The APA includes using the U.S. Environmental Protection Agency (USEPA) surface emission isolation flux chamber (flux chamber) technology in order to perform an APA at BMI Common Areas located in Henderson, Nevada. This SOP describes the quality control (QC) and quality assurance (QA) procedures developed to meet the project data quality objectives (DQOs) which are intended to generate a data set that meets the specific project goals and objectives.

Volatile organic compounds (VOCs), specifically chlorinated compounds, have been detected in the groundwater on site. In addition, imported ore that potentially contained radioactive compounds are known to have been used on site. This SOP was prepared for Basic Remediation Company (BRC) with the intent to collect emission data representing potential exposure to VOCs (related to the groundwater contamination) and radon gas (related to the imported ore) via the subsurface air pathway. BRC plans to use the results of the dynamic flux chamber study for a risk assessment for the current and future land use scenarios. A description of the history, background, and operation of the USEPA-recommended flux chamber flux chamber is provided, along with sampling and analytical protocol, sampling strategy, QC requirements, and sample management protocol.

This SOP document is intended to serve as the fundamental technical document under which all sampling and analytical activities as part of the APA are governed. There is no site or parcel specific sample collection information such as parcel number or location, sample count, sample frequency, sample collection location, rational for sample count, frequency or location information provided in the SOP. Rather, the SOP provides the methodology and QA procedures that will be followed for all APA or site assessment work conducted on site. Project, area, or parcel information as described above will be provided in separate Field Sampling Plans (FSPs) as appropriate, and the FSP documents will include: parcel number or location, sample count, sample frequency, sample collection location, rational for sample count, frequency or location information. Thus, the combined use of this SOP, plus site or parcel specific assessments described in the FSPs, will in concert fully describe as site investigation work.

2.0 PROJECT DESCRIPTION

VOCs, specifically chlorinated compounds, have been detected in the groundwater on site. In addition, imported ore that potentially contained radioactive compounds are known to have been part of the effluent disposal. The goal of the APA work for this site is to assess the surface flux of study compounds meeting data collection needs of the multiple site parcels on a project schedule meeting overall program needs.

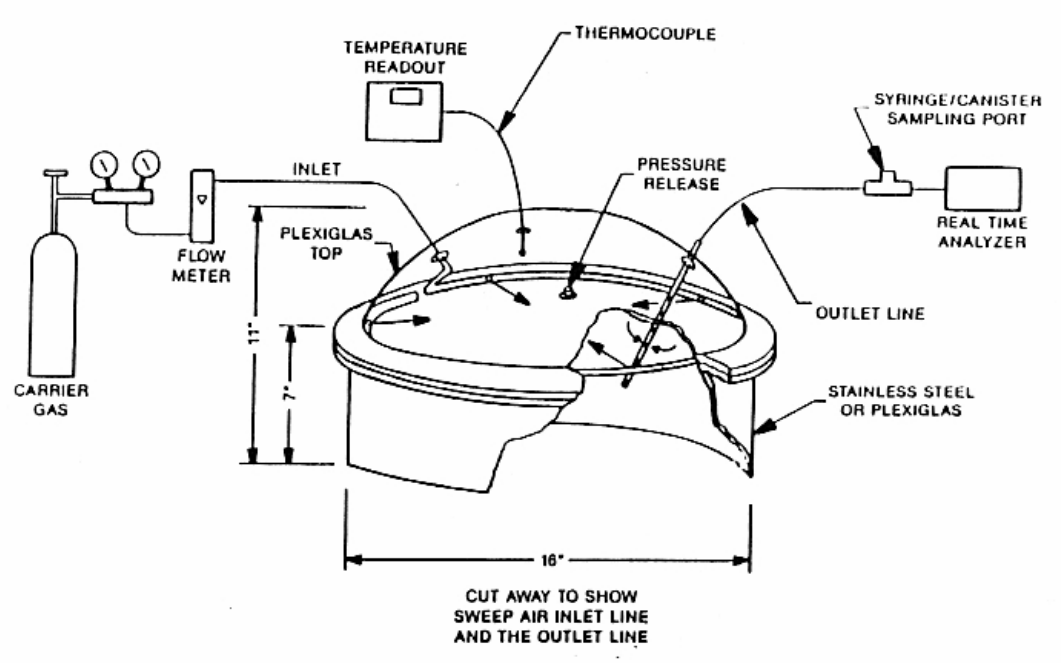
This test protocol is intended to provide area source flux data representative of air emissions of selected VOCs and radon on any properties on or off site. Testing for VOCs will be focused on the 10 parcels or areas on the site that range in size from 49 acres to 285 acres. Testing of specific parcels will be described in FSPs. Individual test locations will be selected based to meet a variety of parcel-specific needs and may be: 1) co-located with existing or future groundwater wells, 2) selected to serve as a transect array across groundwater flow patterns, 3) geographically distributed to represent specific parcels or areas, or 4) may be selected based on historic land use (effluent disposal patterns in particular). Additional testing may be needed depending on the results of each round of testing, depending on the results of the testing efforts described in parcel-specific FSPs, and locations tentatively selected for testing may be relocated using more current and relevant groundwater characterization data. If additional test data are needed, a revised FSP will be prepared, submitted for review, and a second round of testing will be conducted after the first round of flux samples are collected and evaluated. Testing off site will include any area or interest in support of the APA, and individual test locations will likewise be selected to meet area-specific project needs.

Compounds found in groundwater are the primary source for potential surface flux of VOCs and it is this source that is the focus of the sample collection strategy. As such, the site groundwater database will be used, as is possible, to select locations for flux chamber testing. In the absence of groundwater data, locations for VOC/flux chamber testing may be selected to spatially represent a given test area.

The potential source for radon emissions is imported ores that were historically part of the effluent disposal. The test locations for radon (dynamic flux chamber technology and real-time radon detection) will be based on the soil matrix data and/or groundwater data and these test locations will be identified in the FSP.

2.1 USEPA Emission Measurement Technology

For this study, assessment of VOCs and radon gas will be performed using the USEPA recommended surface emission isolation flux chamber (emission flux chamber) and appropriate sample collection/analytical technologies. A schematic diagram of the USEPA emission flux chamber is provided below.



The emission flux chamber is a dynamic environmental chamber designed for operation on land surfaces, but is applicable to sludge and liquid surfaces as well. The chamber qualifies as a mixed tank reactor or a 'continuously stirred tank reactor' (CSTR) and has been designed to assess flux without a significant bias, even for diffusion-limited sources. The chamber is pressure vented and operated at 5.0 liters per minute (L/min; sweep air flow rate) and sample gas is withdrawn from the chamber at equilibrium (5 residence times or 30 minutes; chamber volume is 30 liters and the flow rate of sweep air gas is 5.0 L/min). Because the emission chamber equipment is identical to the USEPA/Environmental Monitoring Systems Laboratory (EMSL) design, and because the protocol for use is followed to exact specifications, the data generated by this technology can be used to quantify area source emission fluxes to within the specifications of accuracy, precision, and repeatability of the published method. All data collected for this research effort, since all data will be collected following the USEPA protocol for dynamic flux chamber testing, will be directly comparable to previously collected flux data by this technology, and will be within the method QA/QC specifications. The comparison to flux data collected using the USEPA flux chamber technology may be useful for project purposes.

Samples are collected from the USEPA flux chamber at equilibrium using appropriate and valid sample collection and analytical methodology. The analytical methods, combined with the field QC testing (minimum amount of field system blank tests and replicate sample tests) will be adequate to demonstrate compliance with method performance.

Several studies have been conducted using the USEPA emission flux chamber at sites with subsurface soil gas sources, and the technology is highly applicable to this type of land application (e.g., diffusive flow subsurface VOC source). The strengths of the flux chamber technology for this application are:

1. The air emissions from a given parcel measured using flux chambers are unaffected by upwind sources including other parcels or upwind sources;

2. The sensitivity (<0.02 $\mu g/m^2\text{-}min^{-1}$ for TO-15 VOC full scan analysis (0.1 parts per billion by volume [ppbv] method detection limit [MDL}), <0.002 $\mu g/m^2\text{-}min^{-1}$ VOC for TO-15 selective ion mode (SIM) analysis for a short list of compounds (0.01 ppbv MDL), and selectivity using the appropriate sample collection techniques and analytical methods is superior to other assessment approaches (e.g. downwind ambient approaches);

3. Other flux chamber studies have been conducted for sites with groundwater sources, and all flux data collected from a given parcels by this technology will be directly comparable to the data collected on other parcels and other sites;

4. All data collected will be of known accuracy and precision;

5. The technology is a direct measurement approach and no modeling is required to obtain emission factor information since data are reported in engineering units, for example: mass per time/surface area, which can be used to estimate process specific emissions if the overall surface area is known.

6. The USEPA technology, using the USEPA/EMSL User's Guide (available on *ceschmidt.com* website) and specified equipment, is a verified approach producing data that can be used to estimate emissions from a process by knowing the surface area and measured flux of the test source.

Thus, the strength of this approach is that it provides accurate and specific flux data from a given test location that can meet the project objectives. Unfortunately, this strength can also be a weakness, since the exact areas tested must be carefully selected so that they represent the parcel-specific fugitive emissions. Therefore, proper planning is needed prior to testing including the selection of representative test locations. Test locations will be recommended and rationale for selection will be provided in the parcel-specific FSPs.

Radon gas may be detected in the dynamic flux chamber using either the activated charcoal canister method and/or real-time monitoring using an instrumental method. Once the radon detection method of choice is established, SOP-16 will be revised to reflect the selected method.

2.2 Analysis of Flux Samples

Several types of compounds have been identified for analysis, and sample analysis will be completed for both full scan species analysis for VOCs, low level detection for specific VOCs by SIM analysis, and radon gas.

The QA program will include strict adherence to sampling and analytical procedure, QC procedures, and project plan specifications. Blank, replicate field, and laboratory QC samples will be collected and analyzed at a frequency of at least five percent as described in the FSPs. All QA procedures will focus on insuring and assessing the quality of program data.

2.3 Calculation of Flux Data

The compound-specific flux rate for VOCs in the dynamic flux chamber will be calculated using the laboratory data for compound concentration in the flux chamber Summa canister ($\mu g/m^3$), the sweep air inflow rate (0.005 m^3/min), and the surface area of the chamber (0.13 m^2). The calculation result gives a direct measure of the flux rate of compounds ($\mu g/m^2$-min) from a given surface.

$$(\mu g/m^3)(0.005\ m^3/min)/(0.13\ m^2) = \text{VOC Flux } (\mu g/m^2\text{-min}) \qquad \text{Eqn. 2-1}$$

The radon flux rate in the dynamic flux chamber using the real-time radon detector will be calculated using the instrument sensitivity with 1-hour of integration of 33 pCi/m^3, the sweep air inflow rate (0.005 m^3/min), and the surface area of the chamber (0.13 m^2). The calculation result gives a direct measure of the flux rate of radon (pCi/m^2-min) from a given surface.

$$(pCi/m^3)(0.005\ m^3/min)/(0.13\ m^2) = \text{Radon Flux } (pCi/m^2\text{-min}) \qquad \text{Eqn. 2-2}$$

2.4 Calculation of Emission Rate Data

The emission rate ($\mu g/m^3$ or pCi/m^3) of compounds from a land surface area will be calculated using the flux data ($\mu g/m^2$-min; pCi/m^2-min) and the surface area tested (m^2). The emission rate of compounds from a given parcel can be estimated by summing the emission rate (per compound) from each specific area of the parcel.

3.0 PROJECT ORGANIZATION AND RESPONSIBILITY

The organization for the program includes overall project management by BRC and subcontract by Dr. CE Schmidt as contractor to the BRC. Environmental Analytical Service in San Luis Obispo, CA (for TO-15 VOC SIM and full scan analyses), and Radon Testing Laboratory of America in Elmsford, NY (for radon analyses) will provide laboratory services. The subcontractor to CE Schmidt for field testing is Harold Litwiler.

The field sampling and analytical activities are the responsibility of Dr. CE Schmidt. Dr. Schmidt will: arrange for the field sampling, coordinate with key personnel at BRC, coordinate with the subcontract laboratories for analytical services, conduct the field sampling, ship samples for analysis, receive and review laboratory data, and report (Technical Memorandum) the results of the field sampling to BRC. Contact information is provided below:

Contact	Address	Phone/Fax	Email
Ranajit Sahu	Basic Remediation Co. 875 W. Warm Springs Henderson, NV 89011	P (702) 567-0465 F (702) 567-0475	sahuron@earthlink.net
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CE Schmidt	19200 Live Oak Road Red Bluff, CA 96080	P (530) 529-4256 F (530) 529-4878	schmidtce@aol.com
Steve Hoyt	EAS 173 Cross Street San Luis Obispo, CA 93401	P (805) 781-3585 F (805) 541-4550	stevehoyt@easlab.com
Nancy Bredhoff	Radon Testing Corp of America 2 Hayes Street Elmsford, NY 10523	P (914) 345-3380 F (914) 345-8546	rtca.com

4.0 QUALITY ASSURANCE OBJECTIVES

The purpose of a QA/QC program is to produce data of known quality that satisfy the project objectives set forth in this document. The QA/QC program shall:

- Provide a mechanism for ongoing control and evaluation of measurement data quality
- Provide an estimate of data quality in terms of accuracy, precision, completeness, representativeness, and comparability for use in data interpretation

Two VOC methods have been selected for this study; USEPA Method TO-15 for 22 study compounds with ultra low method detection limits (gas chromatography with mass spectroscopy detection operated in SIM), and USEPA Method TO-15 for a full list of compounds with low method detection limits (gas chromatography with mass spectroscopy detection operated in the full scan mode).

The DQO level and laboratory contact information is provide in Table 1, and the QA objectives for accuracy and precision are presented by sample matrix for all sampling and analytical parameters in Table 2. These values are estimates of the degree of uncertainty that is considered acceptable in order for the data to fulfill the needs of the program area source testing. The QA/QC program focuses on controlling and quantifying measurement error within these limits, and provides a basis for understanding the uncertainty associated with these data. In the first step of data validation, measurement data are compared to the QA objectives to determine whether gross performance problems occurred.

Table 1. Sample Matrix and Parameters

Parameter	Method	Instrument or Laboratory	DQO Level
22 target compounds	USEPA Method TO-15 Selective Ion Mode (SIM) Analysis	EAS 173 Cross Street San Luis Obispo, Ca 93401 (805) 781-3585	4
70 target compounds	USEPA Method TO-15 (Full Scan Mode-edited list)	EAS 173 Cross Street San Luis Obispo, Ca 93401 (805) 781-3585	4
Radon gas	Methods for measuring radon in air	Radon Testing Corp of America 2 Hayes Street Elmsford, NY 10523 (914) 345-3380	4
Radon gas	PTG-7RN Portable Radon Detector	Direct Scientific 124 San Tropez Ct. Laguna Beach, CA 92651 (310) 589-0601	4

Table 2. Accuracy, Precision, and Sensitivity of Analysis

Parameter	Method	Accuracy	Precision	Sensitivity
TO-15 VOCs SIM	GC/MS	+30% Multi-Component Standard	±30%	0.01 ppbv
TO-15 VOCs Full Scan	GC/MS	+30% Multi-Component Standard	+30%	0.1 ppbv
Radon	Gamma Count	+30% -Standard	+30%	100 pCi/m3
Radon	Real Time Detection by Ion Chamber	+30% -Standard	+30%	33 pCi/m3

The basis for assessing precision, accuracy, completeness, representativeness, and comparability is discussed in the following subsections. Specific calculations for data quality measurements are presented in Section 13. VOC method QC specifications are provided in Table 3.

Table 3. QC Specifications for USEPA TO-15 SIM and TO-15 Full Scan

Parameter	EAS	Comments
Instrumentation	GC with FPD and all Teflon Concentrator	USEPA 15 GC/FPD ASTM D-5504M Sievers Detector
Initial Calibration	3 points minimum **Single injections** Span range of 0.5 to 10.0 ppmv based on 1 ml inj.	Method calls for 3 replicate injections with RSD < 5%
Calibration Check Sample (CCS)	After Initial Calibration **< 30% RSD**	The CCV is run as an LCS
Continuing Calibration Verification (CCV)	Daily (24 hours) **< 30% RSD** Ending Calibration Std to check for drift **< 30% RSD** (Carb 15 only)	The method does not specify a CCV Method specifies a triplicate ending standard <5% of beginning triplicate
Method Blank	No target analytes above 3xMDL	
Laboratory Control Spike	1 per Daily Batch 70-130% recovery	No QC specified in method other than replicate standard analysis
Matrix Spike(5)	1 per Daily Batch if Requested 70-130% recovery	There is an extra charge for matrix spike

Table 3. QC Specifications for USEPA TO-15 SIM and TO-15 Full Scan

Parameter	EAS	Comments
Duplicate (One of below) Lab Control Dup Sample Matrix Spike Dup	1 duplicate with each 20 samples <30% RSD	Only one duplicate is done in each DAB. This is usually an LCD
Holding Times	72 hours in Tedlar bag, and 7 days in Silio canister, from sampling date	
Silico can Certification	Certification by TO-14	Certification can be done by USEPA 15 if requested
Field Duplicates	50% concentrations over 1 ppbv	

DQOs are qualitative and quantitative statements, which specify the quality of the data to satisfy the end uses of the data to be collected. As such, different data uses may require different levels of data quality. There are five analytical levels, which address various data uses, and the methods required to achieve the desired level of quality. These levels are:

- Screening (DQO Level 1): This provides the lowest data quality but the most rapid results. It is often used for health and safety monitoring at the site, preliminary comparison to local regulations or criteria, initial site characterization to locate areas for subsequent and more accurate analyses, and for engineering screening of alternatives. These types of data include those generated on-site (field analysis) through the use of real-time monitoring equipment at the site like the Organic Vapor Analyzer (OVA).

- Field Analyses (DQO Level 2): This provides rapid results and better quality than in Level 1. This level may include mobile lab generated data depending on the level of QC exercised.

- Engineering (DQO Level 3): This provides an intermediate level of data quality and is used for site characterization. Engineering analyses may include mobile lab generated data and some analytical lab methods (e.g., laboratory data with quick turnaround used for screening but without full QC documentation).

- Conformational (DQO Level 4): This provides the highest level of data quality and is used for purposes of risk assessment, and evaluation of remedial alternatives. These analyses require full Contract Laboratory Program (CLP) analytical and data validation procedures in accordance with USEPA recognized protocol.

- Non-Standard (DQO Level 5): This refers to analyses by non-standard protocols, for example, when exacting detection limits or analysis of an unusual chemical compound is required. These analyses often require method development or adaptation. The level of QC is usually similar to DQO Level 4 data.

The data collected for the APA includes Level 3 engineering data using the static chamber technique and radon charcoal sample collection, and Level 4 detailed data for USEPA flux chamber and VOC/radon off-site (fixed laboratory) analysis. The laboratory will perform under DQO Level 4 analysis, however, will not be asked to prepare or submit a CLP type data package. These back-up data will be archived and available upon request.

4.1 Precision

Precision measures the reproducibility of repetitive measurements. It is strictly defined as the degree of mutual agreement among independent measurements as the result of repeated application of the same process under similar conditions. Analytical precision (estimated at 30 percent) is a measurement of the variability associated with duplicate (two) or replicate (more than two) analyses of the same sample in the laboratory. Total precision is a measurement of the variability associated with the entire sampling and analysis process. It is determined by analysis of duplicate or replicate field samples, and incorporates the variability caused by matrix variability, field sampling procedures, and analytical variability. The results of total and analytical precision must be interpreted by taking into consideration all possible sources of variability. Duplicate samples will be analyzed to assess field and laboratory precision, and the results will be reported as the relative percent difference (RPD) between duplicate measurements. In all cases, field precision objectives for RPD will be less than 50 percent. Analytical precision objectives are presented for each method and matrix in Table 2.

4.2 Accuracy

Accuracy is a statistical measurement of correctness, and includes components of random error (variability due to imprecision) and systematic error (bias). As such, it reflects the total error associated with a measurement. A measurement is accurate when the value reported does not differ from the true value, or known concentration, of the spike or standard. Analytical accuracy is typically measured by determining the percent recovery of known target analytes that are spiked at known concentrations into a field sample (a matrix spike) or reagent water (a method spike) before extraction at known concentrations. The stated accuracy limits apply to spiking levels at five times the MDL or higher. The individual methods provide equations for acceptance criteria at lower spiking levels.

Surrogate compound recovery is also reported and is used to assess method performance for each sample analyzed for volatile compounds. Sampling accuracy is assessed by evaluating results for field and trip blanks.

Both accuracy and precision are calculated for specific sampling or analytical batches, and the associated sample results must be interpreted considering these specific measures. Application of calculated precision and accuracy to measurement sample results will be discussed in Section 13. An additional consideration in applying accuracy and precision is the concentration level of the samples; a procedure capable of producing the same value within 50 percent would be considered precise for low-level (near the detection limit) analyses of minor constituents, but would be unacceptable, and possibly useless, for major constituents at high concentrations.

4.3 Completeness

Completeness, also referred to as percent data capture, is defined as the percentage of valid data reported compared to the total number of samples collected for analysis. Valid data are determined during the data assessment process and satisfy the QA objectives. Completeness is determined after precision and accuracy are calculated. The objective for completeness for all measurement parameters and all sample matrices is 90 percent.

4.4 Representativeness

Objectives for representativeness will be defined for each sampling and analysis task and will be a function of the investigative objectives. Representativeness will be achieved through use of the standard sampling and analytical procedures described in this SOP and the frequency of testing as described in Section 5. Adequate representativeness will be evaluated and documented and will consider source and exposure information, area-specific results, data distribution, analyte toxicity, and human receptor locations.

4.5 Comparability

Comparability is the confidence with which one data set can be compared to another. The objectives for this QA/QC program are to produce data with the greatest degree of comparability possible. The number of matrices sampled and the range of field conditions encountered must be considered in ultimately determining comparability. Comparability will be achieved by using the same (standard) methods for sampling and analysis, reporting data in standard units, and using standard and comprehensive reporting formats. Analysis of reference samples may also be used

to provide additional information that can be used to assess comparability of analytical data produced within the program.

5.0 AIR SAMPLING PROCEDURES

This section contains detailed descriptions of the sample collection protocols to be used for field sampling. All field personnel will be familiar with the procedures they will be using and will have a copy of the SOP available for reference.

The following subsections describe two distinct types of air sampling:

5.1 Surface Emission Isolation Flux Chamber Sampling
5.2 Sampling for VOCs and Radon in the Flux Chamber

5.1 Surface Emission Isolation Flux Chamber Sampling

Isolation emission flux chamber sampling is a dynamic direct measurement of emission rates of air contaminants. Flux chambers can be used for measuring source emissions from:

- Liquid surface, quiescent or agitated;
- Solid land surfaces or sludge surfaces;
- Open ports in processes; and
- Cracks or vents in a process or landfill cover.

Flux measurements will be conducted on solid (soil). All flux chamber measurements will be conducted as per the USEPA guidance document, *Measurement of Gaseous Emission Rates from Land Surfaces Using an Emission Isolation Flux Chamber*, February 1986. All solid surface testing will be conducted by placing the chamber directly on the solid surfaces. The method is briefly described below.

The enclosure device, referred to as the flux chamber, is used to sample gaseous emissions from a defined surface area. Clean, dry sweep air is added to the chamber at a fixed, controlled rate. The chamber temperature and volumetric flow rate of air through the chamber is recorded and the concentration of the species of interest is measured at the exhaust of the chamber. The emission rate is calculated as:

$$ER_i = \frac{(C_i)(Q)}{A} \qquad \text{Eqn. 5-1}$$

where:

ER_i = Emission rate of species i ($\mu g/m^2\text{-}min^{-1}$, $pCi/m^2\text{-}min^{-1}$)
C_i = Measured concentration of species i ($\mu g/L$, pCi/L)
Q = Air flow rate (L/min)
A = Exposed Surface Area (m^2)

The response time of the flux chamber is characterized by the residence time. The chamber reside time (t) is a function of chamber volume (V) and air flow rate (Q). The quotient of volume and flow rate (V/Q = t) is the theoretical residence time. Four to five residence times are normally needed to establish steady-state conditions in the chamber at which time representative sampling can occur. This will require 24 to 30 minutes per sampling point.

The chamber is an acrylic topped, stainless steel cylinder designed to penetrate the sampling surface. The chamber has a 0.13 m^2 exposed surface area and a volume of approximately 0.03 m^3. One 1/4 inch port will be used to withdraw sample gas.

Dry, hydrocarbon-free sweep air (zero grade air) will be provided from compressed gas cylinders. The sweep air will pass through a calibrated rotometer with a needle-valve flow control. Inlet and outlet lines are made of Teflon and all fittings in contact with the gas will be Teflon or stainless steel. The outlet line will include a sampling manifold for monitoring and/or collection of the gaseous specie of interest. This manifold will consist of ports for gas-canister sampling and gas-syringe collection. A thermocouple and readout will be used (when possible) to measure the surface and air temperatures at the sample point.

The flux chamber will be wiped clean and dried before each use and then placed over the sampling area. The sweep air is added at a flow rate of 5.0 L/min and the time noted when the chamber is placed on the test surface. The outlet gas concentration can be monitored using a real time instrument such as a TVA-1000 until steady-state conditions are reached (typically four to five residence times); gas concentrations are recorded every residence time. Monitoring with the TVA-1000 is not required. (Note: the TVA-1000 is a real time hydrocarbon analyzer that simultaneously measures total hydrocarbon response by flame ionization detection and ionizable hydrocarbon response by photoionization. It is likely that given the high MDLs of the instrument and the anticipated low flux rate, that monitoring with a TVA-1000 or similar instrument will not be productive.) The TVA-1000 will be used, along with other relevant site-specific information,

to identify flux chamber sample locations for VOCs. Air temperatures inside and outside the chamber are also taken and recorded. Once steady state is reached (about 30 minutes), gas samples are collected. Samples will be collected using evacuated stainless steel canisters.

Data will be recorded on the data form shown in Attachment 1. The following data collection steps will be taken:

- Locate equipment at the sampling location;
- Document location of measurement, date, time, and operator;
- Initiate sampling by starting sweep air, checking the flow rate, and placing the chamber on the testing location;
- Document the gas flow rate and the initial and final temperatures of ambient air, of air inside the chamber, and of the bulk soil/waste;
- Document any other data such as waste characteristics, meteorological conditions, etc., for possible correlations with emission rate measurements;
- Monitor the outlet gas concentrations using a TVA-1000 (if necessary) and radon detector (if necessary) and record data every residence time;
- Collect gas samples at steady state indicated by time readings;
- Fill out appropriate chain-of-custody forms and master sample log entries for sample collected.

5.2 Sampling for VOCs/Radon in the Flux Chamber

Samples will be collected as grab samples in evacuated canisters for VOCs and using a real time instrument for radon. Evacuated, summa-polished canisters will be used to collect exhaust gas grab samples for VOC analyses under ambient or near-ambient conditions from the flux chamber. Canisters will be used in the vacuum mode; that is, the vacuum in the clean canisters will be used to pull a sample of gas into the container. The sampling rate will be maintained by opening the valve to the canister, listening to the filling of the canister and filling the canister over a time period to provide a sample collection rate that does not exceed 2 L/min. Note that locations for sample analysis by TO-15 full scan and TO-15 SIM will be identified in the FSP, as will the criteria for selection for those locations analyzed by TO-15 full scan and TO-15 SIM.

Monitoring is required for the collection of radon gas. Two methods may be used for radon sample collection: activated charcoal canister collection (24-to-48 hour exposure), or up to 1-

hour real-time instrument detection (ion chamber integration). Prior to sample collection, all sampling media (VOC canisters, activated charcoal [AC] radon canister sorbents, blank check on radon monitor) will be cleaned according to the specific analytical methods selected.

Grab Sampling

Grab samples will be collected in evacuated, summa-polished 6-liter canisters. The canister will be used to collect exhaust gas samples for VOC analyses using USEPA Method TO-15 full scan and TO-15 SIM. Both TO-15 full scan and TO-15 SIM analyses will be conducted from the same canister; not all canister will be analyzed for both full scan and SIM. Methods of analysis will be identified in each specific FSP. Canisters will be used in the vacuum mode; that is, the vacuum in the clean canisters will be used to pull a sample of gas into the container.

Prior to sample collection, each canister will be cleaned and evacuated in the laboratory, and the absolute pressure will be recorded.

To collect canister samples from the flux chamber:

- The canister pressure (vacuum) is checked prior to sampling and recorded. The initial pressure should be between -30 and -27 inches of mercury. However, the canister will be considered acceptable (useable) if the value is >-24 inches of mercury;
- Attach sampling line from the to flux chamber to the canister using a clean, 1/4 inch Teflon or stainless steel tube with 1/4 inch Swagelok fittings;
- Record start time on data sheet and open canister inlet valve slowly. A slight hissing sound can be heard during sampling by placing an ear against the canister;
- The canister grab samples will be collected over a 3 minute period. Sample time is controlled by slowly opening the inlet valve so that the hissing sound is barely audible or the vacuum gauge begins to drop. A stopwatch or watch with a second hand should be used;
- After sample collection is completed, the canister inlet valves are closed and the sample line is disconnected from the canister;
- The absolute canister pressure is again measured and recorded on the data sheet and the canister chain-of-custody form;
- Prior to transporting to the laboratory, all canister valves are tightened and stem nuts sealed with Swagelok plugs;
- Complete appropriate chain-of-custody forms, master sample log entries, and canister tags for samples collected, and ship canisters.

Integrated Sampling

Integrated samples will be collected on solid sorbent media or monitored with an ion chamber (ion chamber count) for radon gas. Since it is expected that monitoring with the analyzer will be used for the radon assessment, the monitoring protocol with the real time instrument is described below.

Prior to sample collection, the radon detector will be prepared and calibrated as recommended by the manufacturer. To measure radon from the dynamic flux chamber using a real-time instrument:

- Interface the PTG-7RN instrument to the exhaust port of the dynamic chamber and initialize the instrument;
- Record start time on data sheet and initiate flux chamber test;
- The integrated sample will be collected at the over a period of time that will allow for the collection of ions in the ion chamber, assumed to be one hour;
- After sample monitoring, the radon concentration is read off the instrument read-out and recorded;
- The data capture is documented on the Flux Form, and the test is concluded.

6.0 SAMPLE CUSTODY FORMS

Sample possession during all testing efforts must be traceable from the time of collection until the results are verified and reported. Sample custody procedures provide a mechanism for documentation of all information related to sample collection and handling to achieve this objective.

Dr. Schmidt will be responsible for seeing that the field team adheres to proper custody and documentation procedures for all sampling operations. Preformatted field data and Chain-of-Custody forms will be used as the primary documentation mechanism to ensure that information pertaining to each sample is properly recorded. In addition, a master sample logbook will be maintained for all samples collected. Examples of these data documentation forms are presented in this section. Copies of the Chain-of-Custody forms and the field logs will be retained in the project file.

6.1 Documentation Procedures

6.1.1 Field Records

Field personnel will be required to keep accurate written records of their daily activities in a bound logbook. All entries will be legible, written in waterproof ink, and contain accurate and inclusive documentation of an individual's field activities, including field data and observations, any problems encountered, and actions taken to solve the problem. The type of data recorded in the field logbook includes field measurements, ambient conditions, and any other information pertinent to sample collection. Entry errors or changes will be crossed out with a single line, dated, and initialed by the person making the correction. Entries made by individuals other than the person to whom the logbook was assigned will be dated and signed by the individual making the entry. Field logbooks will be available for review by interested parties.

6.1.2 Sample Labels

Each sample collected will receive a sample label that identifies the sample by a unique sample identification number. These labels are affixed to the sample container prior to sample collection.

6.1.3 Sample Log Book

A sample master log will be maintained for all samples collected. Each sample will be assigned a unique identification number; a full description of the sample, its origin, and disposition will be included in the log entry.

6.2 Chain-of-Custody Procedures

After the samples are collected and documented in the master logbook, a Chain-of-Custody form will be completed and will accompany the samples to the laboratory. A Chain-of-Custody form is used for sample types. Team members collecting the samples are responsible for the care and custody of the samples until they are transferred or dispatched to the appropriate laboratory. When transferring samples, the individuals relinquishing and receiving the samples will sign, date, and note the time on the record.

This record documents sample possession from collection to the laboratory sample control center. When the samples are received by the laboratory, the sample control officer will verify the Chain-of-Custody form against the samples received. If any discrepancies are observed, they

will be recorded on the Chain-of-Custody form and Dr. Schmidt will be notified to correct the problem.

6.3 Shipment

All sample shipments will be accompanied by the Chain-of-Custody record, which identifies the contents of each crate. The person relinquishing the samples to the laboratory will request the signature of a laboratory representative to acknowledge receipt of the samples. Sample collection and shipment will be coordinated to ensure that the receiving laboratory has staff available to process the samples according to method specifications.

All shipping containers will be secured for safe transportation to the laboratory. The method of shipment, courier name(s), and other pertinent information is entered in the "Remarks" section when the samples are to be shipped (i.e., FedEx, Express Mail, etc.) instead of hand-delivered.

6.4 Sample Handling Procedures

The objective of sample handling procedures is to ensure that samples arrive at the laboratory intact, at the proper temperature, and free of external contamination. VOC canister samples will be shipped via Federal Express to EAS in San Luis Opisbo, CA.

Sample packaging requirements for hazardous materials requiring interstate transport are defined in the Code of Federal Regulations 40 (CFR) 49, Chapter 1, Part 171. These requirements outline in detail the proper classification and transportation procedures for hazardous materials that will be used in the transporting of samples.

6.5 Sample Preservation

Once the samples have been collected, the methods specify preservation, storage requirements and holding time limitations. Table 4 summarizes the preservation requirements for the type of samples collected during this program.

Table 4. Parameters for Sample Preservation

Parameter	Preservation and Storage Requirements	Maximum Holding Time (Days)
6-Liter Summa Polished Stainless Steel Canisters for VOCs	Cool storage area; avoid direct sunlight when/if possible. Wrap Valves, Ship in Cardboard	14 Days

7.0 CALIBRATION PROCEDURES AND FREQUENCY

Information is presented in this section pertaining to the laboratory calibration of sampling equipment. Included are descriptions of each procedure or references to applicable SOPs, the frequency of calibrations, and the calibration standards to be used.

7.1 Laboratory Instrument Calibration

Laboratory instruments are calibrated according to manufacturer's specifications and are in compliance with the analytical method requirements. Detailed calibration procedures and recommended frequencies are included along with the analytical SOPs, which can be found in the appendix to this volume. A brief description of the analytical methodologies, and their associated calibration procedures, is included in Section 9.

7.2 Sampling Equipment Calibration Procedures

An important function in maintaining data quality is the check-out and calibration of all sampling equipment. Using referenced procedures, the equipment will be calibrated prior to field sampling. These results will be properly documented and retained. If a referenced calibration technique for a particular piece of apparatus is not available, then state-of-the-art techniques are used. A discussion of the procedures used to calibrate equipment is presented below. Calibration frequency of the field sampling equipment is presented in Table 5.

Table 5. Calibration Frequency of Field Sampling Equipment

Sampling Equipment	Calibration Before Sampling	Frequency	
		Annual	Project Needs
Rotometer	X	X	X
PTG-7RN Radon Detector	X	As Specified by Manufacturer	As Specified by Manufacturer

Rotometers used be to control the flow rate of sweep air gas into the flux chamber. Rotometers will be calibrated using a primary gas flow standard (DC Lite—frictionless piston device) generating a multipoint (minimum of three points up to 5 L/min) calibration curve. The calibration will be performed prior to the testing event and the calibration curve data will be available on request. Note that only pure, dry air is delivered through the rotometers.

The PTG-7RN radon detector will be maintained and operated as per the instrument manufacturer. Pre-use performance checks and calibration procedures will be followed in order

to insure proper operation of the detector. Specific operating procedures will be included in subsequent revisions of SOP-16.

8.0 DATA REDUCTION, VALIDATION AND REPORTING

The data reduction, validation, and reporting procedures described in this section will ensure that complete documentation is maintained throughout the program, that transcription and data reduction errors are minimized, that the quality of the data is reviewed and documented, and that the reported results are properly qualified and in a conventional format.

8.1 Data Reduction

The reduction of raw data generated at the laboratory bench is the responsibility of the analyst producing it. The data interpretation that is required to calculate sample concentrations follows the methodology described in the specific analytical SOP. After all analyses have been completed, a preliminary laboratory report is generated for review by the laboratory supervisors who verify that the analyses were properly performed and interpreted. After the final review by the laboratory supervisor, the raw data is transferred to sample control and presented for review by the QA coordinator. Raw data, together with all supporting documentation, are stored permanently in confidential files by sample control.

The QA coordinator reviews the data for adherence to the QC method limits. In addition, the data are reviewed for the presence of outliers. An outlier is an unusually large (or small) value in a set of observations. There are many possible reasons for outliers, among which are:

- Faulty instruments or component parts
- Inaccurate reading of a record, dialing error, etc.
- Errors in transcribing data
- Calculation errors

Sometimes analysts or operators can identify outliers by noting the above types of occurrences when they record observations. In these instances, the errors are corrected, or if correction is not possible the suspect observations may be removed from the data before calculations are performed. If no such information exists, the Dixon Criteria are used to test suspected outliers at the five percent significance level if there are three or more points in the data set containing the outlier. Outliers identified by this method may be removed from the data before further

processing (see W.J. Dixon, "Processing Data for Outliers," Biometrics, 1953, Vol. 9, No. 1, pp. 74-89).

8.2 Data Transfer and Verification

A laboratory database is used to store and transfer analytical data from the laboratory. Sample control staff is responsible for entering into the system and verifying sample and result information and generating hard copies of the analytical results.

8.3 Data Validation

Dr. Schmidt will review field documentation and all measurement data for acceptable sample collection and analysis procedures, consistency with expected results or other results, adherence to prescribed QA procedures, and agreement with the acceptance criteria described in Section 4.

Initially, the reviewer will determine whether hold times were met and that all required analytical QC checks were reported with the data. Then, all QC sample results will be reviewed to evaluate the sampling and analytical performance. Reagent blank results will be evaluated to identify any systematic contamination; spike and duplicate results will be compared to the QA objectives presented in Section 4, and the results will be used to calculate precision and accuracy for the data set. This process will identify any analytical methods and compounds for which the QA objectives are not satisfied, and corresponding sample data will be qualified with a "flag" indicating the problem. Samples collected on the same day, analyzed in the same run or batch, or individual samples may be flagged, depending on the type of problem that has been identified. Reanalysis or re-sampling may be recommended at this time if data are determined to be unacceptable for the intended application.

The qualifier codes, or "flags," will be stored with the data and printed with the data when reported or transferred for any purpose. The specific statistical procedures and qualifier codes used in the validation process are described in detail in Section 13. After data are received from the laboratory, entered, checked, and qualified, they are a permanent part of the database and cannot be deleted or altered.

The data validation process is one where the USEPA National Functional Guidelines will be followed as applied systematically to the APA data set. The following data qualifiers may be used, depending on the results of the QC data for the data set:

B- Compound found in the laboratory or method blank data

U- Compound reported as less than the method detection limit

J- Compound reported at above the method detection limit but below the reporting limit

E- Compound exceed the instrument calibration range

8.4 Reporting

Data reporting for this project will consist of QA reporting, investigative data reporting, and QC data reporting.

General reporting practices for measurement data will include:

- Heading information identifying the sample batch and the analytical method
- Unique sample identification number or code
- Consistent units of measure
- Consistent number of significant figures
- No blank or dashed places reported; all spaces will contain a designation (i.e., not analyzed, not sampled, etc.)
- Explanation of outlier values or the cause for deviation from historical data
- Comparison with regulatory threshold values if applicable
- QA flags
- Quantification of accuracy and precision for analytical data
- Non-detect results will be recorded as "< [reporting limit]"

8.4.1 Investigative Data Reporting

Measurement data generated during the course of an investigation will be reported in tabular form from the computerized database. The formats of the reports will vary, depending on the objectives of the investigation. In general, data will be presented according to sampling location, analytical method, parameter, and/or matrix. All data will be reported with the qualifiers discussed above, and units will be specified. Commonly used reporting formats will be catalogued and used repeatedly, while specialized formats will be developed as needed.

8.4.2 General Reporting Procedures

The procedures employed to ensure report quality involve the following:

- All calculations and measurements will be verified by recalculation by the person initially providing data. The calculations and measurements are then checked by another individual who signs and dates the calculation sheets. Any calculations and measurements that differ from the initial totals are resolved by both individuals. Once the calculations and measurements are included in an internal working copy of a document, the figures are rechecked during peer review. If there are many such calculations within a report, a certain percentage (10 to 50 percent) are checked again during peer review.

- Numerical values presented in reports and comparisons of numbers appearing in text, tables, and appendixes will be addressed in the manner discussed above.

8.4.3 QC Data Reporting

QC results will be reported by sample matrix and method in tabular form. How these QC results influence the measurement data will be delineated. For example, matrix spike interference will influence specific samples, while laboratory blank contamination will influence all samples extracted or analyzed on a specific day or during a specific analytical run. Two levels of tables may be constructed for each type of QC check. The first level table will contain all QC data, and will present one line per parameter or analysis. First level table formats will be used in presenting duplicate samples and analyses, matrix and method spikes, and system blank results. First level QC data tables will be generated for all investigations.

Specially developed table formats will be used occasionally as an aid to interpretation of the investigative data. The particular format will depend on how the QC results are expected to influence the investigative data. This type of table might be used to identify corresponding investigative results (samples analyzed on corresponding dates), which may be inaccurate. Specialty tables will be generated automatically or manually, depending on the volume of data to be processed and the complexity of the calculations.

9.0 ANALTYICAL PROCEDURES AND CALIBRATION

This section contains brief descriptions of calibration procedures and analytical methodology for the analysis of air samples that will be collected during the testing. Reproductions of the methods used during this program are included as an appendix to this volume. Each method is briefly described in the following sections.

9.1 Method Detection Limits

The MDL is defined as the minimum concentration of a substance that can be measured and reported with 99 percent confidence that the analyte concentration is greater than zero. Laboratories perform MDL studies on an annual or quarterly basis (depending on the method) to demonstrate that they can meet or exceed the method-recommended MDLs. The USEPA procedure used for establishing MDLs is described in Appendix B of Part 136 Definition and Procedure for the Determination of the Method Detection Limit -Revision 1.1, 40 CFR 136, 1984. This procedure consists of analyzing seven aliquots of a standard spiked at three-to-five times the expected MDL, which is taken through all the sample processing steps of the analytical method. The MDL is defined as three times the standard deviation of the mean value for the seven analyses. In the few cases where the individual laboratory has experimentally determined MDLs higher than recommended MDLs, the method-recommended MDL is shown in parentheses in the appropriate table or text.

9.2 Laboratory Standards and Reagents

Laboratory standards and reagents are obtained from the following suppliers:

For organic analysis, analytical standards are obtained from USEPA sources, SUPELCO, Radian laboratory in Austin, and MSD isotopes. Spectral grade and reagent grade solvents and reagents are obtained from chemical suppliers such as Aldrich, Sigma, Burdick and Jackson, EM Science, and Baxter.

All standards and laboratory reagents, with the exception of common laboratory solvents, are dated upon receipt. The preparation and use of all standards are recorded in bound laboratory notebooks that document standard traceability to USEPA or National Institute of Standards and Technology (NIST) standards. Additional information recorded includes date of preparation, concentration, name of the preparer, and expiration date, if applicable.

9.3 Methods of Canister Analysis

The following describes the analysis procedure to be used for the determination of VOCs. When a sample is received from the field, it is first assigned a sample delivery group (SDG) number and a laboratory number. The sample information is logged into a master log notebook and the computer Laboratory Information System (LIMS). The canister pressure is measured and logged into the computer and then the canister is pressurized with Ultra High Purity (UHP) nitrogen or

helium to 5 pounds per square inch gauge (psig). The final pressure is then measured and recorded. The canisters are pressurized to provide positive pressure for removing the sample.

9.3.1 USEPA Method TO-15 Full Scan Analysis, Canister Sampling Gas Chromatography/Mass Spectroscopy (GC/MS) for VOCs

USEPA Method TO-15 GC/MS full scan method uses a sorbent trapping system with a high-resolution capillary column to analyze for VOCs using full scan GC/MS. This method can be used for many hazardous air pollutants (HAPs) compounds including polar organics, since no dryer is used. The method can measure most compounds down to 0.1 ppbv. The list of compounds selected for the program APA is provided in Attachment 2, and a copy of the EAS protocol is provided in Attachment 3. Note that quantitation of up to 86 compounds can be achieved by this method, and 70 compounds have been selected for the APA based on compounds known to have been used on site, and compounds known or suspected to be found in the subsurface. That is, only compounds listed on BRC's site-related chemical (SRC) list are included in the full scan mode analyte list. The 16 compounds not included are: 2-propanol, 4-ethyltoluene, acrolein, acrylonitrile, allyl chloride, cyclohexane, Freon 114, hexane, meth-acrylonitrile, methyl methacrylate, methylstyrene, octane, propionitrile, t-1,4-dichloro-2-butene, tetraethyl lead, and tetrahydrofuran. Limiting the compound list to project related or potentially project related compounds reduces unnecessary data as well as improves the analytical sensitivity of the method.

Samples are analyzed on an HP 5890 Series II gas chromatograph and HP 5971 MSD quadrapole mass spectrometer detector. A 1.0 milliliter (mL) to 200 mL gas sample is loaded from the air sampling canister or bag onto the sorbent trap.. A gas phase internal standard mixture is injected with each sample prior to sample loading. The sample is concentrated on a solid sorbent which affords large sample volume and low detection limits for target analytes. After the sample is trapped, it is thermally desorbed and focused onto the beginning of a 0.32 mm ID deactivated fused silica capillary column; 60 meter, DB-5, 0.25 mm ID fused silica capillary column in the GC. The GC is temperature programmed from -10° C to 220° C at a ramp rate of 13° C/min. As the column is heated, the compounds elute off the column and enter the mass spectrometer. The MS is scanned from 45 to 300 atomic mass units (amu) with a scan rate of 1 to 2 seconds for the full scan mode of operation. The GC/MS is tuned and operated according to the specifications outlined in the USEPA draft scope of work for VOC compounds in ambient air, and USEPA Method 8260 in the SW-846 Test Methods. Compounds are calibrated by the external standard procedure using NIST traceable air standards as described below. The RPD of a duplicate pair is

less then 30% at 10 ppbv and the average MDL is approximately 0.1 ppbv for most compounds at a 500 mL load volume.

The standards used for the routine analytical tests are commercial NIST traceable gas standards normally ordered at a concentration of 2 to 10 parts per million by volume (ppmv). Commercial standards are available for the TO-14 GC/MS list and special in-house standards are prepared for those compounds where commercial standards are not readily available.

The GC/MS compounds are calibrated by using a dilution of the NIST traceable standard. The initial calibration curve consists of three to five calibration points. The continuing calibration consists of one point for the GC/MS full scan. The response factors for the initial calibration curve are to be within 30% RSD. If the response factor for the daily standard is more than 30% RPD from the average response factor of the initial calibration a new calibration curve is prepared. Standards are prepared by using a gas dilution system on the gas chromatograph or by making static dilutions to atmospheric levels. The gas dilution system is constructed from an eight-port gas sampling valve with various size sample loops. The loops are filled with the standard and flushed with "zero air." The gas dilution system is used for the daily instrument calibration. The concentration of the individual target compounds is determined using the initial calibration curve response factors.

Laboratory data for TO-15 full scan will be reported in concentration units of ppbv and $\mu g/m^3$ and flux units of $\mu g/m^2$-min.

9.3.2 USEPA Method TO-15 SIM, Canister Sampling Gas Chromatography/Mass Spectroscopy (GC/MS) for VOCs

USEPA Method TO-15 GC/MS SIM operation method uses a sorbent trapping system with a high-resolution capillary column to analyze for VOCs for a selected list of VOCs. This method can be used for many VOCs, except that the number of compounds is limited in order to achieve the lowest detection limits possible. SIM operation is similar to the description provided above for TO-15 full scan, except that the instrument if focused to a handful of 'ion windows' as opposed to a full spectra of ion counts. The advantage to this is that lower MDLs can be achieved. The method can measure most compounds on the SIM list down to less than 0.01 ppbv.

Compound selection was based on Method TO-15 full scan not being able to meet risk-based target levels. Attachment 4 presents the Method TO-15 full scan reporting limits, the reporting

limits needed to meet risk-based target levels (based on calculated indoor air concentration using the equations and parameters in BRC's *Closure Plan* [BRC, ERM, and DBS&A 2007] and comparison to USEPA Region 9 preliminary remediation goals [PRGs] for air), and whether the Method TO-15 full scan mode reporting limits achieves these risk-based target levels. There are 20 compounds for which Method TO-15 full scan mode does not meet the required report limits. In addition to these 20 compounds, 1,2-dichlorobenzene and 1,3-dichlorobenzene are also included because of their relative frequency of detection in soil and groundwater. The list of the 22 compounds selected for the program APA SIM analysis is provided in Table 6.

Table 6. Compounds Included in the TO-15 SIM Analysis and Detection Limits

Compound	CAS Number	MDL ppbv	Reporting Limit ppbv	MDL µg/m^3	Reporting Limit µg/m^3
1,1,1,2-Tetrachloroethane	630-20-6	0.0005	0.026	0.0035	0.18
1,1,2,2-Tetrachloroethane	79-34-5	0.0005	0.026	0.0035	0.18
1,1,2-Trichloroethane	79-00-5	0.0005	0.026	0.0028	0.14
1,2,3-Trichloropropane	96-18-4	0.0005	0.026	0.0031	0.16
1,2-Dibromo-3-chloropropane	96-12-8	0.0005	0.026	0.0049	0.26
1,2-Dibromoethane	106-93-4	0.0005	0.026	0.0039	0.20
1,2-Dichlorobenzene	95-50-1	0.0005	0.026	0.0031	0.16
1,2-Dichloroethane	107-06-2	0.0005	0.026	0.0021	0.11
1,2-Dichloropropane	78-87-5	0.0005	0.026	0.0024	0.12
1,3-Dichlorobenzene	541-73-1	0.0005	0.026	0.0031	0.16
1,4-Dichlorobenzene	106-46-7	0.0005	0.026	0.0031	0.16
Benzene	71-43-2	0.0005	0.026	0.0016	0.085
Benzyl chloride	100-44-7	0.0005	0.026	0.0026	0.14
Bromodichloromethane	75-27-4	0.0005	0.026	0.0034	0.18
Carbon tetrachloride	56-23-5	0.0005	0.026	0.0032	0.17
Chloroform	67-66-3	0.0005	0.026	0.0025	0.13
Dibromochloromethane	124-48-1	0.0005	0.026	0.0043	0.23
Hexachlorobutadiene	87-68-3	0.0005	0.026	0.0054	0.28
Naphthalene	91-20-3	0.0005	0.026	0.0267	0.14
Tetrachloroethene	127-18-4	0.0005	0.026	0.0035	0.18
Trichloroethene	79-01-6	0.0005	0.026	0.0027	0.14
Vinyl chloride	75-01-4	0.0005	0.026	0.0013	0.068

1) The MDL = 3.14 * standard deviation of seven replicate measurements
2) The actual reported MDL may vary based on Canister dilution or matrix interferences

9.4 USEPA Recommended Method for Measuring Radon Gas in Air with Charcoal Canisters

AC canisters, collected from static chambers and the USEPA dynamic flux chamber will be sampled and analyzed following the USEPA Office of Air and Radiation (6604J)guidance document titled "*Indoor Radon and Radon Decay Product Measurement Device Protocols'* dated August, EPA 402-R-92-004, July 1992 revised, (www.epa.gov/radon/pubs/devprot1.html). This protocol describes the use of passive dosimetry for radon adsorbed onto AC with detection by gamma scintillation or gamma spectroscopy. The commercial laboratory Quality Manual used for conducting the analysis is included in Attachment 3.

The AC canisters are sampled by removing the cylindrical canister from the shipping container, opening the access port on the canister, suspending the canister in the flux chamber for the duration of the sample collection interval, removing the canister from the flux chamber, fixing the lid on the canister, repackaging the canister for shipping, and then shipping the AC canister to the laboratory for analysis. The canister is analyzed in the laboratory for radon decay products by placing the canister in a gamma detector. The gamma count is used, along with the exposure time period, to calculate a radon concentration expressed as pCi/L. Calibration is performed by exposing the detector to known radon standards. Water content corrections (water may be adsorbed on the AC which reduces the adsorption of the charcoal) may be conducted by the laboratory depending on the canister configuration and the weight of the canister as it is received by the laboratory. The MDL for this technique is about 0.1 pCi/L. Laboratory data for the radon method will be reported in concentration units of pCi/L. The data analysis will report the flux of radon in the units of pCi/m^2-min.

10.0 INTERNAL QUALITY CONTROL

Internal QC consists of collecting and/or analyzing a series of duplicate, replicate, blank, and matrix spike samples to ensure that the analytical results are within QC limits specified for the program. Laboratory QC samples are documented at the bench and reported with the analytical results. The QC sample results are used to quantify precision and accuracy, and identify any problems or limitations in the associated sample results. Field QC samples will be documented in field logbooks and submitted "blind" to the laboratory. These components of the sampling program will help produce data of known quality throughout the sampling and analysis component of the program.

The USEPA methods selected for use on this program (USEPA TO-14, USEPA TO-15, and activated charcoal dosimetry for radon), will meet the USEPA QA/QC specifications in the respective methods.

10.1 Analytical Laboratory Quality Control Samples

Laboratory QC is necessary to control the analytical process, to assess the accuracy and precision of analytical results, and to identify assignable causes for atypical analytical results. The QC checks in the laboratory protocol are specific to the analytical method and generally include the use of one or more of the following QC samples.

10.1.1 Calibration Standards

Initial calibration is performed as required for each analytical method, usually using a range of calibration standards with the low standard near the detection limit for the compound. These standards are used to determine the linear dynamic range for the initial instrument calibration.

10.1.2 Quality Control Check Samples

QC check samples are standard samples containing the analytes of interest at a specified concentration, usually in the mid-calibration range. These samples are prepared independent of the calibration standard, and are used to demonstrate that the instrument is operating within acceptable accuracy and precision limits. QC check samples are required for GC/MS analyses. They are usually analyzed at the beginning and after every 10 samples are analyzed.

10.1.3 Reagent Blanks

A reagent blank is a sample composed of all the reagents (in the same quantities) used in preparing a real sample for analysis. It is carried through the same sample preparation procedure as a real sample. Reagent blanks are used to ensure that interferences from the analytical system, reagents, and glassware are under control. The required frequency for analyzing VOC method reagent blanks is specified in the analytical SOP for each method, and generally consists of one per day for each method/instrument and/or one per extraction batch.

10.1.4 Method Spike/Method Spike Duplicates

A method spike is a sample of target analytes at known concentrations that is spiked into a field sample before sample preparation and analysis. Two aliquots of the sample may be spiked and used for the duplicate analysis. The results of the analysis of the duplicate spiked samples are

used to measure the percent recovery of each spiked compound and to compare the recovery between samples, which provides an estimate of the accuracy and precision of the method. The calculations for accuracy and precision are outlined in Section 13, and the QA objectives for accuracy are given in Section 4.

10.1.5 Laboratory Duplicates (Duplicate Analyses)

Laboratory duplicates are repeated but independent determinations of the same sample by the same analyst, at essentially the same time and under the same conditions. The sample is split in the laboratory and each fraction is carried through all stages of sample preparation and analysis. Duplicate analyses measure the precision of each analytical method. The method of calculation for precision is outlined in Section 4, and QC objectives for precision are listed in Table 1. Laboratory duplicate analyses are performed for 10 percent of samples analyzed, or at least one per day, for analytical methods that do not require matrix spike-matrix spike duplicates.

Attachment 3 summarizes the specific internal QC checks performed as required for the analytical methods. This attachment also includes information relating to the initial calibration and ongoing calibration checks.

10.2 Field Quality Control Samples

Field QC includes QC for the TVA-1000 instrument and replicate and blank sample collection and analysis. Field QC is summarized in Attachment 5.

10.2.1 Field Duplicate Samples

A field duplicate sample is a second sample collected at the same location with the original sample. Duplicate sample results are used to assess precision, including variability associated with both the laboratory analysis and the sample collection process. Duplicate samples will be collected simultaneously or in immediate succession using identical recovery techniques, and treated in an identical manner during storage, transportation, and analysis.

Recovery and analysis of five percent or at least one duplicate sample per day for each method will be performed. The sample containers will be assigned a control number such that they cannot be identified (blind duplicate) as duplicate samples by laboratory personnel performing the analysis.

10.2.2 Field Blanks

Field blanks are samples of purified air that are collected and processed in the field using the same sampling and handling procedures as other samples. Field blanks are used to assess the potential introduction of contaminants to the samples during sample collection in the flux chamber and analysis in the laboratory. The frequency requirements for preparing field blanks will be five percent of the samples collected over the course of the sampling program.

11.0 PERFORMANCE AND SYSTEM AUDITS

Systems audits, performance audits, and data quality audits are independent assessments of sample collection and analysis procedures. Audit results are used to evaluate the ability of the system to produce data that fulfill the objectives established for the program, satisfy the QC criteria, and identify any areas requiring corrective action. A systems audit is a qualitative review of the overall sampling or measurement system, while a performance and data quality audit is a quantitative assessment of a measurement system.

11.1 Technical Systems Audit

A technical systems audit is an on-site, qualitative review of the sampling or analytical system. Sampling systems will be inspected at the beginning of the sampling task by Dr. Schmidt. Due to the limited activity and adherence to standard protocols, no formal systems auditing will be performed. Based on the results of Phase 1, performance auditing may be included in Phase 2 testing activities. It is assumed that the Nevada Division of Environmental Protection (NDEP) will conduct systems auditing during the field and data reduction activities.

The internal laboratory systems reviews routinely include:

- Calibration procedures and documentation
- Completeness of data forms, notebooks, and other reporting requirements
- Data review and validation procedures
- Data storage, filing, and record-keeping procedures
- Sample custody procedures
- QC procedures, control limits, and documentation
- Operating conditions of facilities and equipment

- Documentation of maintenance activities
- Systems and operations overview

The field systems review will include:

- Calibration procedures and documentation for field meters and other measurement devices
- Complete documentation of field logbooks and sampling data sheets
- Organization and minimization of potential contamination sources while in the field
- Proper sample collection, storage, and transportation procedures
- Compliance with the established Chain-of-Custody procedures for sample documentation and transfer to the laboratory

11.2 Performance Audits and Data Quality Audits

Performance audits and data quality audits quantitatively assess the data produced by a measurement system. A performance audit involves submitting accepted reference standards for analysis for each analytical method and/or analytical instrument. The standards for each matrix are selected to reflect the range of concentrations expected for the sampling program. The performance audit answers questions about whether the measurement system is within control limits and whether the data produced meet the analytical QA specifications. The data quality audit evaluates data quality indicators, and identifies limitations that may be encountered in data applications.

Because selected laboratories are licensed by the State of Nevada as certified testing laboratories and participate in an approved Performance Evaluation Program, no laboratory audits will be performed.

12.0 PREVENTATIVE MAINTENANCE PROCEDURES

12.1 Field Equipment/Instruments

The field equipment for this project includes the calibrated rotometers used to control sweep air flowrate into the flux chamber. Specific preventative maintenance procedures to be followed for field equipment are those recommended by the manufacturer.

Critical spare parts and disposable or expendable items will be kept on-site to minimize instrument down time. Backup rotometers and primary standard instrumentation should be available on-site or within one-day shipment to avoid delays in the field schedule.

12.2 Laboratory Instruments

As part of their QA/QC Program, a routine preventative maintenance program will be conducted by the selected laboratory to minimize the occurrence of instrument failure and other system malfunctions. Designated laboratory personnel will be responsible for performing routine scheduled maintenance, and coordinate with the vendor for the repair of all instruments. All laboratory instruments are maintained in accordance with manufacturer's specifications and the requirements of the specific method employed. This maintenance will be carried out on a regular, scheduled basis, and will be documented in the laboratory instrument service logbook for each instrument. Emergency repair or scheduled manufacture's maintenance is provided under a repair and maintenance contract with factory representatives. Routine preventative maintenance schedules will be provided with the selected laboratories SOPs.

13.0 SPECIFIC ROUTINE PROCEDURES USED TO ACCESS DATA PRECISION

The assessment of measurement data is required to ensure that the QA objectives for the project are met, and that quantitative measures of data quality are provided. A distinction must be made between routine QC and data assessment that is conducted as a part of laboratory operations, and the project-related data assessment process conducted after the data have been reported. It must be assumed that the planning and monitoring that have gone into the sampling and analysis process have served to control the process as much as possible to produce data of sufficient quality for project needs. After the data have been reported, it is necessary to identify any part of the process that could not be controlled, and to what extent that may affect the quality of the reported data.

The routine QC procedures conducted in the laboratory are established in the analytical SOPs. The laboratory is responsible for following those procedures and operating the analytical systems within statistical control limits. These procedures include proper instrument maintenance, calibration checks, and internal QC sample analyses at the required frequencies (i.e., reagent blanks, matrix spike/matrix spike duplicates [MS/MSD], laboratory duplicates). One of the additional ongoing data assessment processes is to maintain control charts for representative QC sample analyses in order to monitor system performance. This provides verification that the system is in statistical control and indicates when performance problems occur, so the problems

can be corrected as soon as possible. When reporting the sample data, the laboratory is required to provide the results of associated QC sample analyses.

Problems occur in spite of all precautions taken in planning and execution of the sampling and analysis task. In these cases, the data assessment conducted by Dr. Schmidt after the data have been reported must identify the problem, determine which data are affected, and state how these data may be limited for use in the intended applications.

The discussion of data assessment presented in this section pertains to the project-related assessment of data that have been reported after laboratory analyses have been completed. Data assessment procedures established for the testing include:

- Evaluation of blank results to identify systematic contamination
- Statistical calculations for accuracy and precision using the appropriate QC sample results
- Estimation of completeness in terms of the percent of valid data
- Recommendations for corrective actions such as reanalysis or resampling if data are critically affected
- Assignment of data qualifier flags to the data as necessary to reflect limitations identified by the process

Some basic statistical calculations used in the data assessment process are presented along with a discussion of specific applications to environmental sample results.

13.1 Blank Data Assessment

Reagent blank results indicate whether any of the contaminants reported in sample results may be attributed to laboratory sources and, therefore, would not likely present in the sampled medium. The most common laboratory contaminants are methylene chloride, phthalates, acetone, and toluene. These are recognized as being potentially ubiquitous in the laboratory environment and controlling them to within acceptable low levels is part of standard laboratory procedure.

If contamination from these compounds is reported in blank samples, the samples associated with the blank--either the same analytical or extraction batch--may be qualified using a data qualifier (B) to indicate that some or all of these compounds may be from laboratory sources. If the concentrations reported in the samples are similar to the blank concentrations, it is likely that

all of the contamination was introduced; this assessment is then made in the report for the sampling task.

13.2 Accuracy

As previously defined, accuracy is associated with correctness and is a comparison between a measured value and a known, or 'true,' value. Accuracy is calculated from method spike (spikes of the pure matrix) or matrix spike results.

Spike results are reported by the laboratory as percent recovery and are compared to the accuracy objectives stated in Section 4. Results that do not satisfy the objectives are assigned a data qualifier flag (A) to indicate uncertainty associated with inaccuracy.

Method spikes are spikes of a reference material into a sample matrix (e.g., canister or cryotrap) in the lab. If recovery is outside the established limits, samples from the same batch may be qualified. If any results appear atypical and could be related, those results may also be qualified. The flagged data will be discussed in the report for the sampling task, and specific limitations such as poor or enhanced recovery for specific compounds will be stated.

The percent recovery of matrix spike samples will be calculated using Equation 13-1.

$$\%R = \frac{A - B}{C} \times 100 \qquad \text{Eqn. 13-1}$$

Where:

A = The analyte concentration determined experimentally from the spiked sample;
B = The background level determined by a separate analysis of the unspiked sample and;
C = The amount of the spike added.

13.3 Precision

Precision is a measure of variability between duplicate or replicate analyses and is calculated for field and laboratory replicates. By definition, field precision incorporates laboratory precision. Precision is calculated as the RPD between duplicate analyses or MS or MSD as appropriate. The calculated RPDs are compared to the objectives stated in Section 4. Results that do not satisfy the objectives are assigned a data qualifier flag indicating uncertainty associated with imprecision (P).

An average RPD may be calculated and reported as a measure of overall analytical precision for compounds with multiple measurements. The specific samples collected or analyzed in duplicate are flagged if they do not satisfy the QA objectives. In addition, associated samples may be flagged to indicate variability due to poor precision. For poor field duplicate precision, samples collected by the same sampling team, from the same equipment, or on the same day may be affected; close evaluation of those results should indicate the most likely source of variability and the corresponding samples will be qualified as warranted. For poor laboratory precision, samples processed and analyzed in the same batch will be more closely evaluated, and any anomalous results will be qualified.

Dr. Schmidt is responsible for ensuring that these codes are assigned to the data as required by the established QC criteria, and that they are reported and understood by project staff using the data for specific applications. He is also responsible for initiating corrective actions for analytical problems identified during the QC data assessment process. These corrective actions range from verifying that the method was in statistical control during the analytical runs, to reanalysis of the sample, to resampling.

The RPD will be calculated for each pair of duplicate analysis using the Equation 13-2.

$$RPD\,(\%) = \left[\frac{S - D}{\left(\frac{S + D}{2}\right)}\right] \times 100 \qquad \text{Eqn. 13-2}$$

Where:

S = First sample value (original or MS value)
D = Second sample value (duplicate or MSD value)

13.4 Completeness

Completeness is determined after the QC data have been evaluated and the results applied to the measurement data. In addition to results identified as being outside of the QC limits established for the method, the occurrence of matrix effects, and lost samples, samples that could not be analyzed for any other reason are included in the assessment of completeness. The percentage of valid results is reported as completeness.

Data completeness will be calculated using Equation 13-3.

$$Completeness = \frac{Valid\ Data\ Obtained}{Total\ Data\ Planned} \times 100 \qquad \text{Eqn. 13-3}$$

13.5 Corrective Action

Review of sampling logs and/or analytical results may indicate problems that invalidate the results or critically influence their use. In these cases, corrective action may be required to ensure that valid data are provided. Corrective actions include: recalibration and reanalysis if the analytical system is shown to be out of statistical control; reanalysis if systematic contamination has occurred; resampling if sampling procedures or sample handling have been improper or caused contamination. The severity of the problem and the importance of affected samples will dictate when one of these actions will be required.

General recommendations in any case are to follow good laboratory practice and good management practice for all aspects of the sampling and analysis program. These include development and strict adherence to SOPs for all areas, and the establishment of clear responsibilities and lines of communication within the sampling and analytical staff, as well as between project and laboratory staff.

14.0 CORRECTIVE ACTION

During the course of the testing program, it is the responsibility of the Dr. Schmidt to see that all measurement procedures are followed as specified and that measurement data meet the prescribed acceptance criteria. In the event a problem arises, it is imperative that prompt action be taken to correct it.

14.1 Reporting Malfunctions

Problems that require corrective action will be documented by Dr. Schmidt as presented in the field log book. He will initiate the corrective action request in the event that QC results exceed acceptability limits or upon identification of some other problem or potential problem. Corrective action may also be initiated by the laboratory coordinators or a representative of BRC based upon QC data. Depending upon the severity of the problem, corrective actions range from use of data qualifier flags, to reanalysis of the sample or samples affected.

14.2 Quality Assurance Reports to Management

Effective management of a field sampling and analytical effort requires timely assessment and review of field activities. This will require effective interaction between the field team members, the laboratory, and the client.

Dr. Schmidt will be responsible for informing team members on the status of their respective tasks and results of the QC activities. This will ensure that quick and effective solutions can be implemented should any data quality problems arise. The use of frequent, oral reporting provides an effective mechanism for ensuring ongoing evaluation of measurement efforts. These discussions will address some of all of the following:

- Summary of activities and general program status
- Summary of calibration and QC data
- Summary of unscheduled maintenance activities
- Summary of corrective action activities
- Status of any unresolved problems
- Assessment and summary of data completeness

Summary of any significant QA/QC problems, corrective action, and recommended and/or implemented solutions not included above

15.0 HEALTH AND SAFETY

All project personnel working on site are required to:

1. Contact facility site health and safety personnel and obtain information on facility health and safety requirements, and

2. Adhere to the health and safety plan for the site.

Common sense will help to keep field personally out of harms way, which may include but is not limited to the usual slip-trip hazards, lifting injuries, and awareness of working with compressed gases, but also extends to working on rough terrain.

16.0 REFERENCES

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ATTACHMENT 1

SURFACE FLUX MEASUREMENT DATA FORM

SURFACE FLUX MEASUREMENT DATA FORM

DATE _____ **SAMPLERS** _____

LOCATION _____

SURFACE DESCRIPTION _____

CURRENT ACTIVITY _____

INSTRUMENT TYPE _____ **I.D. NO.** _____ **TYPE** _____ **ID NO.** _____

INSTRUMENT BASELINE _____

PROJECT QC: BACKGROUND MEASUREMENTS BLANK MEASUREMENTS REPLICATE MEASUREMENTS
AMBIENT CONCENTRATIONS _____
CHAMBER I.D. _____ **PHOTO TAKEN:** Yes No _____
CHAMBER SEAL _____ **CONDENSATION:** Yes No B. PRESS _____
AMBIENT CONDITIONS: Sun P.Sun Cloudy Wind at 5', __ mph Wind at Seal,__ mph
TEMP_____ **RAIN:** Yes No Comment _____
PRIOR CHAMBER CLEANING: Full Wash Wet Wipe Dry Wipe None _____
SAMPLE LINE: Back Flushed Prior to Start Purged Prior to Sampling New Line Used Line
SWEEP AIR _____ **CC**_____ **SUPPLIER** _____ **PSIG START**_____ **PSIG STOP** _____

Time	Sweep Air (L/min)	Residence Number	Temperature				Real-Time		Sample Number	Comments
			Chamber		Ambient					
			Surface	Air	Surface	Air	(µg/m^3)	(ppmv)		
		0								
		1								
		2								
		3								
		4								
		5								

COMMENTS:

SITE DIAGRAM:

ATTACHMENT 2

LIST OF COMPOUNDS FOR TO-15 FULL SCAN MODE OPERATION AND MDLs

ATTACHMENT 2
LIST OF COMPOUNDS FOR USEPA METHOD TO-15 FULL SCAN MODE OPERATION AND MDLs

Compound	CAS Number	MDL ppbv	RL ppbv	MDL µg/m^3	RL µg/m^3
1,1,1,2-Tetrachloroethane	630-20-6	0.1	0.51	0.72	3.62
1,1,1-Trichloroethane	71-55-6	0.1	0.52	0.58	2.89
1,1,2,2-Tetrachloroethane	79-34-5	0.1	0.52	0.73	3.65
1,1,2-Trichloroethane	79-00-5	0.1	0.51	0.57	2.86
1,1-Dichloroethane	75-34-3	0.1	0.52	0.43	2.15
1,1-Dichloroethene	75-35-4	0.1	0.52	0.42	2.13
1,1-Dichloropropene	563-58-6	0.1	0.49	0.46	2.3
1,2,3-Trichloropropane	96-18-4	0.11	0.55	0.68	3.39
1,2,4-Trichlorobenzene	120-82-1	0.1	0.52	0.79	3.94
1,2,4-Trimethylbenzene	95-63-6	0.1	0.52	0.52	2.61
1,2-Dibromo-3-chloropropane	96-12-8	0.22	1.1	2.2	10.98
1,2-Dibromoethane	106-93-4	0.1	0.52	0.82	4.09
1,2-Dichlorobenzene	95-50-1	0.1	0.52	0.64	3.2
1,2-Dichloroethane	107-06-2	0.1	0.52	0.43	2.15
1,2-Dichloropropane	78-87-5	0.1	0.52	0.49	2.46
1,3,5-Trimethylbenzene	108-67-8	0.1	0.52	0.53	2.64
1,3-Dichlorobenzene	541-73-1	0.1	0.52	0.64	3.2
1,3-Dichloropropane	142-28-9	0.11	0.54	0.52	2.58
1,4-Dichlorobenzene	106-46-7	0.1	0.52	0.64	3.2
1,4-Dioxane	123-91-1	0.09	0.44	0.33	1.64
2,2-Dichloropropane	594-20-7	0.11	0.53	0.5	2.53
2-Butanone	78-93-3	0.09	0.43	0.26	1.31
2-Hexanone	591-78-6	0.09	0.44	0.37	1.86
Acetone	67-64-1	0.09	0.45	0.22	1.1
Acetonitrile	75-05-8	0.22	1.12	0.48	2.39
Benzene	71-43-2	0.1	0.52	0.34	1.7
Benzyl chloride	100-44-7	0.09	0.45	0.48	2.41
Bromochloromethane	74-97-5	0.1	0.51	0.55	2.76
Bromodichloromethane	75-27-4	0.08	0.4	0.55	2.77
Bromoform	75-25-2	0.09	0.47	0.99	4.96
Bromomethane	74-83-9	0.1	0.51	0.41	2.04
Carbon disulfide	75-15-0	0.09	0.45	0.29	1.45
Carbon tetrachloride	56-23-5	0.1	0.52	0.67	3.38
Chlorobenzene	108-90-7	0.1	0.52	0.5	2.48
Chloroethane	75-00-3	0.1	0.51	0.28	1.39
Chloroform	67-66-3	0.1	0.52	0.52	2.59
Chloromethane	74-87-3	0.1	0.51	0.22	1.09
cis-1,2-Dichloroethene	156-59-2	0.1	0.52	0.42	2.11
cis-1,3-Dichloropropene	10061-01-5	0.1	0.52	0.48	2.41
Dibromochloromethane	124-48-1	0.09	0.44	0.77	3.87
Dibromomethane	74-95-3	0.11	0.55	0.97	4.84
Dichlorodifluoromethane	75-71-8	0.1	0.51	0.52	2.61

ATTACHMENT 2
LIST OF COMPOUNDS FOR USEPA METHOD TO-15 FULL SCAN MODE OPERATION AND MDLs

Compound	CAS Number	MDL ppbv	RL ppbv	MDL µg/m^3	RL µg/m^3
Dichloromethane	75-09-2	0.1	0.52	0.37	1.86
Ethanol	64-17-5	0.22	1.12	0.44	2.18
Ethylbenzene	100-41-4	0.1	0.52	0.46	2.33
Freon 113	76-13-1	0.1	0.52	0.81	4.07
Hexachlorobutadiene	87-68-3	0.1	0.52	1.14	5.68
Isobutyl alcohol	78-83-1	0.23	1.13	0.84	4.21
Isopropylbenzene	98-82-8	0.11	0.57	0.58	2.89
Isopropyltoluene	99-87-6	0.11	0.55	0.62	3.12
m & p-Xylene	108-38-3	0.21	1.03	0.92	4.61
Methyl iodide	4227-95-6	0.19	0.94	1.13	5.67
Methyl Isobutyl Ketone	108-10-1	0.09	0.46	0.38	1.95
Methyl tert butyl ether	1634-04-4	0.08	0.39	0.29	1.45
Naphthalene	91-20-3	0.22	1.09	1.19	5.9
n-Butylbenzene	104-51-8	0.1	0.52	0.59	2.95
n-Heptane	142-82-5	0.08	0.42	0.35	1.78
n-Propylbenzene	103-65-1	0.11	0.54	0.55	2.74
o-Xylene	95-47-6	0.1	0.52	0.46	2.31
sec-Butylbenzene	135-98-8	0.11	0.52	0.59	2.95
Styrene	100-42-5	0.1	0.52	0.45	2.26
tert-Butylbenzene	98-06-6	0.11	0.52	0.59	2.85
Tetrachloroethene	127-18-4	0.1	0.52	0.72	3.61
Toluene	108-88-3	0.1	0.52	0.4	2
trans-1,2-Dichloroethene	156-60-5	0.09	0.44	0.36	1.8
trans-1,3-Dichloropropene	10061-02-6	0.1	0.52	0.48	2.41
Trichloroethene	79-01-6	0.1	0.52	0.57	2.85
Trichlorofluoromethane	75-69-4	0.1	0.51	0.59	2.95
Vinyl acetate	108-05-4	0.09	0.43	0.31	1.56
Vinyl chloride	75-01-4	0.1	0.51	0.27	1.35

Note:
The actual reported MDL may vary based on Canister dilution or matrix interferences.
CAS - Chemical abstract system
MDL - Method detection limit
RL - Reporting limit
ppbv - Parts per billion by volume
µg/m^3 - microgram per cubic meter

ATTACHMENT 3

SUMMARY OF LABORATORY QUALITY CONTROL

TO-15 FULL SCAN AND SIM LABORATORY PROTOCOLS

Environmental Analytical Service
STANDARD OPERATING PROCEDURE

SOP Number: 11.TO15.01

EAS Method: EPA TO15 Volatile Organics by GC/MS Full Scan (MSD1)

EPA Method: TO15 The Determination of Volatile Organic Compounds (VOCs) in Ambient Air & Source Using SUMMA Passivated Canisters and Gas Chromatographic Analysis.

Written By: Steve Hoyt

Approval: _____
Lab Director

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1. MATRIX, INTERERENCES, SCOPE:

This method can be used to analyze for volatile organic compounds (VOCs) in ambient air samples collected in SUMMA canisters or Tedlar bags. This method can be used for TO-14, TO-14A and TO-15. The EAS QC criteria is different for each of these methods and must be checked prior to analysis.

2. METHOD SUMMARY:

The ambient sample is loaded through a Carbo trap to remove water vapor, carbon dioxide, and concentrated on a stainless steel trap packed with glass beads. The VOCs are separated using a 60 meter fused capillary column directly connected to the source of an HP 5970 MSD. Depending on the compounds and volume of the sample analyzed, the Method Detection Limit (MDL) can be as low as 0.01 parts-per-billion by volume (ppbv) and as high as 20 ppbv (depending on linearity) for an ambient air sample. For soil gas or landfill gas, the MDL can be as low as 0.2 ppmv and as high as 5000 ppmv**.**

3. HOLDING TIMES, PRESERVATION, CONTAINERS:

The standard holding time as specified in EPA TO15 is 30 days. However some clients have a project specific holding time of 14 days.

4. SAFETY:

4.1. The method uses Liquid Oxygen which can cause frostbite.

4.2. Safety goggles and gloves are recommended.

4.3. Secure all pressurized gas cylinders with chains.

4.4. Do not wear open-toed shoes.

5. EQUIPMENT, SUPPLIES:

5.1. Hewlett Packard 5890 Gas Chromatograph
5.2. Hewlett Packard 5970 Mass Selective Detector
5.3. Hewlett Packard Chemstation Software
5.4. NUTECH 8533 Universal Sample Concentrator
5.5. NUTECH Trap Temperature Controller
5.6. Zero Air, Helium, Compressed Nitrogen, and Liquid N_2 tanks
5.7. NIST traceable Standard cylinder(s)
5.8. Electrically heated Cryotrap
5.9. Liquid O_2
5.10. 2 metal-cased, cylindrical, glass Dewar flasks; 665 ml and 350 ml
5.11. NIST traceable flow controller for determining the sample volume
5.12. NIST traceable timer for determining the sample volume

6. REAGENTS, STANDARDS:

6.1. Internal Standard Mix: Prepared Gas Standard (5-10 ppmV) containing Pentafluorobenzene, 1,4-Difluorobenzene, Toluene-d8, Chlorobenzene-d5, and Bromofluorobenzene. For the EAS IS compound list the Internal Standard Mix is made from neet materials and is NIST traceable by weight.

6.2. Primary Calibration Standard: TO-14 Compound List. Commercial gas standard from Scott Marrin, Spectra, or Scott Specialty NIST traceable 1 ppmv gas standards for TO-14 target compounds.

6.3. Primary Calibration Standard: TO-15 Compound List. For the EAS TO-15 compound list the Primary Calibration Standard is made from neet materials and is NIST traceable by weight. The standard is prepared from three mixes. The TO-15 list is made at 1 ppmv, the 8260 List is made at 1 ppmv, and the 8260 Special List is made at 2 ppmv. The standard is prepared in an AL150 cylinder according to the Standard Preparation SOP 1.05.

6.4. A lab spike with an approximate concentration of laboratory spike compounds of 0.100 ppbv is prepared in a AL150 cylinder. The cylinder is spiked and pressurized to 1500 psig and the final pressure is measured using a NIST traceable pressure gauge. The actual concentration of the lab spike is calculated from the final dilution information and certification against a Primary standard.

7. STANDARDIZATION, CALIBRATION:

See Section 10 for initial and continuing calibration criteria.

8. OPERATING CONDITIONS:

Nutech Clamshell Heater to 280 C
Nutech Oven Temperature 120-150C
Nutech Heater to trap 195 C

GC/MS Conditions

Capillary Column: 60 meter, 0.25 DB-5 column
Carrier Gas: Helium, 20 psig column pressure; 150 ml/min (NUTECH)
Sample Flow: 100 ml/min

Summary of Method Parameters (Full Scan Regular List)

EM volts: based on tune
Mass scan: 35 to 260 amu
Scan/sec: 1.6
Threshold: 500

Temperature Program: -10°C, 3 min
Ramp Rate: 6°C/min
Final Temp: 203°C for 0.5 min
Run Time: 38 min

9. PROCEDURE:

9.1. Daily Startup

9.1.1. Turn on the monitor and computer for MSD1.

9.1.2. Turn on the NUTECH Controller.

9.1.3. Make sure that the Nutech Clamshell control box is turned on and the temperature is at 280°C. A temperature sensor may need to be plugged into the yellow connector if it has been previously removed. Make sure the clamshell is not on the Carbo trap. Check Nutech oven temperature, it should be about 122C (look in back of concentrator). Run the small fan to cool the carbo trap for about 5 minutes or until it is cool to touch.

9.1.4. Turn on the liquid N_2, Zero Air, and Compressed Nitrogen tanks. Make sure that the pressures on the primary pressure gauge for the Zero Air, Compressed Nitrogen, and UHP Helium tanks are above 200 psi. Replace the tank if the primary pressure is less than 200 psi. Set the pressure on the secondary pressure gauge to 75 psi for the Zero Air tank, 25 psi for the Compressed Nitrogen, and 60 psi for the UHP Helium. These pressures are approximate and should be verified in the run log or maintenance log.

9.1.5. Reset the carrier flow through the Carbo trap with the Digital Flow Check™ to 350 ml/min by attaching the flow check to the right side of the desorption apparatus.

9.1.6. Check Zero Air line and ambient line flow with the Digital Flow Check™. Set the flow from the Zero Air line to the same rate the initial 5 pt calibration

curve was run. (The default flow rate is 100 ml/min.) Make certain that the flow controller is connected to the vent line. Set the intake flow rate through the ambient line to 100 ml/min unless otherwise specified by your supervisor. The flow meter (black box) should read 99 - 103 if the flow has been properly set. Record the flow in the run log.

9.1.7. Create a new directory for the day on the hard drive of the instrument computer as described in SOP 2.03.

9.1.8. Click on START and scroll to PROGRAMS, then to "EnviroQuant #1" and scroll to "ENVTop" and click to launch Chemstation. Under the "Methods" menu click on "Load and Run Method". Load the most current Full Scan method and set up the data collection file for the run. Under data file name enter the directory and name of the file to which you want to save.

See SOP 2.03 Analytical File Naming and Storage and 2.02 Sample Analysis and Processing for more details.

9.1.9. Daily Analytical Protocol

See SOP 2.03 Analytical File Naming and Storage and 2.02 Sample Analysis and Processing for more details.

9.1.10. Check the PFTBA Tune. Select Tune MS menu, Manual tune, File -> load tune file. Select the most current tune from the menu. Open the calibration valve, File -> Generate report. Close calibration valve and exit Manual tune. Refer to SOP 2.06 for more information on tuning the GC/MS for BFB.

9.2 Daily Standardization

9.2.1. Under the "Methods" menu in Chemstation, click on "Load and Run Method". Load the most current Full Scan method and set up the data collection file for the run. Once all the necessary information has been properly entered click on "Run Method".

9.2.2. Make certain that switches for valves 1,2, and 6 on the NUTECH are in the "Load" position and that the 8 port valve lever is positioned to load the 5.0 ml loop (normal CCV standard volume to be loaded). (RL Position)

9.2.3. Make sure that the heat box is off the Carbo trap. The Carbo trap must be at room temperature before proceeding (use the small fan as needed to aid in cooling).

9.2.4. Place the cryotrap in liquid O_2.

9.2.5. Connect the source line to the standard cylinder. Load the standard (section 6.2.) for 10 seconds onto a 5.0 ml loop (normal CCV standard volume) on the 8 port valve.

9.2.6. Rotate the 8 port valve lever and allow Zero Air to flow through for 2 minutes.

9.2.7. Connect the source line to the internal standard (IS) cylinder. Load the IS (section 6.1.) for 2 minutes onto the 2.0 ml loop (LL Loop) on the 8 port valve.

9.2.8. Rotate the 8 port valve lever and allow Zero Air to flow through for 2 minutes.

9.2.9. Repeat Steps 9.2.5 to 9.2.8 to load the second standard (TO-15 compound standard) if needed. If more then two standards are to be loaded, repeat Steps 9.2.5 to 9.2.8 for each standard.

9.2.10. Flip the valve 2 switch into the "Inject" position. Place the heat box on (around) the Carbo trap and run the timer for 10 minutes.

9.2.11. After the 10 minutes, flip valve 6 to the "inject position", dip cryofocus in liquid O_2, take the liquid O_2 off the cryotrap, and turn on the heater and heat the trap for 10 minutes.

9.2.12. After the 10 minutes, flip valve 6 back to the "Load" position, and turn on the cryo (liquid N_2), press [CLEAR] [**.**] [ENTER] [ON] on the GC keypad.

9.2.13. When the oven temp on the GC panel reaches -10°C, press [START] on the GC keypad and quickly (gently) pull the cryofocus out of the liquid O_2. Set the timer for 7 minutes.

9.2.14. After the timer goes off, turn off the cryo, press [CLEAR] [**.**] [ENTER] [OFF] on the GC keypad. Turn off the trap heater and take the heat box off of the Carbo trap (use the small fan as needed to aid in cooling).

9.2.15. After the standard run is over and the data analysis is complete, go to "2nd Data Analysis" and load the method and data file for the standard run. Check the BFB and make sure that it passes the PFTBA tune criteria (See SOP 2.06).

9.2.16. In "2nd Data Analysis" under "Quant" click on "Qedit Quant Result" and make sure that all the compounds detected have been integrated correctly. Manually integrate if necessary (and instructed to do so) according to the guidelines described in Appendix 1.0 in the SOP 3.01 Processing Samples in the DAB folder. Exit and save changes (if any) otherwise exit and abort changes. Under "Quant" click on "Generate Report", "Detailed, Text Only" to the printer. When manually integrating, integrate the sample to match the way the initial calibration standards were integrated. For Data Deliverable packages print the integration, explain why the manual integration was done, and sign.

9.2.17. Under "ConCal" click on "Evaluate Data File as Con Cal to Screen". Check the continuing calibration against the QC criteria for the project or the method. If it still does not pass run the standard again. If it passes go on to the next section. If it fails you may have to run a new 5 pt curve or take other corrective actions; consult your supervisor before doing so.

9.3. Laboratory Control Spikes

9.3.1. Set up Laboratory control spike file name and method in the computer as described in Section 9.2.1 using the file naming system detailed in SOP 2.03.

9.3.2. Repeat steps 9.2.2 through 9.2.14 substituting the appropriate Laboratory Control Spike Cylinder for the "standard" in the loading instructions.

9.3.3. After the run is over and the data analysis is complete, go to "2nd Data Analysis" and load the method and data file for the spike run. Manually integrate if necessary (and instructed to do so) according to the guidelines described in Appendix 1.0 in the SOP 3.01 Processing Samples in the DAB folder.

9.3.4. Run the spike again, this will be the duplicate control spike. Refer to the QC Criteria for the method and project for information on acceptable laboratory control duplicates.

9.3.5. Check the report against the QC criteria for the Method or Project. If the criterion is not met, reanalyze the spike(s). If no reproducibility is obtained,

notify your supervisor immediately. The spike may have to be re-certified or a new spike needs to be prepared. .

9.4. Zero Air Blank / Method Blank / System Blanking

9.4.1. Set up Method Blank file name and method in the computer as described in Section 9.2.1 using the file naming system detailed in SOP 2.03.

9.4.2. Make certain that switches for valves 1,2, and 6 on the NUTECH are in the "Load" position and that the 8 port valve lever is positioned to load the internal standard (IS).

9.4.3. Repeat Steps 9.2.3 through 9.2.4

9.4.4. Allow Zero Air to flow through for a total of 5 minutes (500 ml).

9.4.5. During the Zero Air loading, connect the source line to the IS cylinder. Load the IS (see section 6.1.) for 10 seconds onto the 2.0 ml loop on the 8 port valve. (LL position).

9.4.6. After the timer goes off, proceed with steps 9.2.10 through 9.2.14.

9.4.7. After the run is over and the data analysis is complete, go to "2nd Data Analysis" and load the method and data file for the spike run. Manually integrate if necessary (and instructed to do so) according to the guidelines described in Appendix 1.0 in the SOP 3.01 Processing Samples in the DAB folder.

9.4.8. Check the report against the QC criteria for the Method or Project. If the criterion is not met, reanalyze the blank. If the second blank does not pass, notify your supervisor. Your supervisor may have you run a third blank, fill out a corrective action form, or perform troubleshooting/maintenance on the GC/MS. If you have a blank that passes the criterion then go on to the next step. (Note that "can checks" may be used as method blanks; consult your supervisor before doing so.)

9.5. Loading Ambient/Low Level Samples

9.5.1. Set up analysis file name and method in the computer as described in Section 9.2.1 using the file naming system detailed in SOP 2.03.

9.5.2. Make certain that switches for valves 1,2, and 6 on the NUTECH are in the "Load" position and that the 8 port valve lever is positioned to load the internal standard (IS).

9.5.3. Connect sample canister to the ambient intake line. Make sure that the canister is closed.

9.5.4. Repeat Steps 9.2.3 through 9.2.4.

9.5.5. The flow meter should read approximately 1.0 -2.0 to indicate that there are no system leaks.

9.5.6. Open the sample canister and run the timer for desired load volume based on flow (usually 100 ml/min).

9.5.7. During the sample loading, connect the source line to the IS cylinder. Load the IS (see section 6.1.) for 10 seconds onto the 2.0 ml loop on the 8 port valve. (LL position).

9.5.8. Close the sample canister when the timer goes off. Proceed with Steps 9.2.10 through 9.2.14.

9.5.9. After the run is over and the data analysis is complete, go to "2nd Data Analysis" and load the method and data file for the spike run. Manually integrate if necessary (and instructed to do so) according to the guidelines described in Appendix 1.0 in the SOP 3.01 Processing Samples in the DAB folder.

9.5.10. Check the report against the QC criteria for the Method or Project. If the criterion is not met, reanalyze the sample to meet QC Criteria. If the second sample does not meet QC requirements, notify your supervisor. Your supervisor may have you run a 3rd analysis, fill out a corrective action form, or perform troubleshooting/maintenance on the GC/MS.

9.6 Loading Source Samples

9.6.1. Set up analysis file name and method in the computer as described in Section 9.2.1 using the file naming system detailed in SOP 2.03

9.6.2. Repeat steps 9.2.2 through 9.2.14 substituting the appropriate Laboratory Control Spike Cylinder for the "standard" in the loading instructions.

9.6.3. After the run is over and the data analysis is complete, go to "2nd Data Analysis" and load the method and data file for the spike run. Manually integrate if necessary (and instructed to do so) according to the guidelines described in Appendix 1.0 in the SOP 3.01 Processing Samples in the DAB folder.

9.6.4. Check the report against the QC criteria for the Method or Project. If the criterion is not met, reanalyze the sample to meet QC Criteria. If the second sample does not meet QC requirements, notify your supervisor. Your supervisor may have you run a 3rd analysis, fill out a corrective action form, or perform troubleshooting/maintenance on the GC/MS.

10. QUALITY CONTROL CRITERIA:

TO-15

Parameter	EAS	Comments
BFB Tune	Daily	
Tuning Criteria with BFB	TO-15	
Initial Calibration	5pt points minimum Relative Standard Deviation (RSD) < 30% for TO-14 Compounds < 40% for other compounds 1,2,4-Trichlorobenzene, naphthalene, and hexachlorobutadiene can be up to 80% 4 Compounds can exceed criteria by 10%	2 to 50 ppbv 1 to 25 ppbv Project Specific Criteria can be specified in advance
Calibration Check Sample (CCS)	Every 12 months Same Percent RSD as Initial Calibration	
Continuing Calibration Verification (CCV)	Daily (24 hours) 5 ppbv Std Same Percent RSD as Initial Calibration	TO-15 uses 10 ppbv std
Internal Standard (IS)RT	Pentafluorobenzene 1,4-Difluorobenzne	Pentafluorobenzene is used instead of

	RT < 0.5 min daily std. Response 60% to 140%	Bromochlorobenzene which is a target compound
Surrogate	Toluene-d8 70-130% recovery	Matrix interferences can cause out of limits recoveries.
Method Blank	No target analytes above 3xMDL	
Laboratory Control Spike	1 per Daily Batch 70-130% for LCS list	LCS does not contain complete target list unless specified.
Matrix Spike	1 per Daily Batch if Requested 70-130% special list	There is an extra charge for matrix spike
Duplicate (One of below) Lab Control Dup Sample Matrix Spike Dup	1 duplicate with each 20 samples <30% for special LCS spike list Same Percent RSD as Initial Calibration for other compounds.	Only one duplicate is done in each DAB. This is usually an LCD
Canister Holding Times	30 days from sampling date	
Canister Certification	Certification <0.2 ppbv or less then 2x MDL of target compound by full scan GC/MS	
Field Duplicates	50% concentrations over 1 ppbv	

11. CALCULATIONS, DATA PROCESSING:

11.1. Automated Data Processing

11.1.1. The data is automatically processed on the Chemstation Software, but must be checked manually. The results are exported to an EXCEL spreadsheet for generation of the final report.

11.1.2. At the end of the sample run go into “2nd Data Analysis” and load the data file. Make sure that the correct method is loaded.

11.1.3. Once the file has been manually checked for proper integration, click on "Create/Modify Template/Database" under "CustRpt". This will prompt you to the linked EXCEL spreadsheet.

11.1.4. When the system links with EXCEL, click on the report you want to generate, e.g., to14fs.xls.

11.1.5. See SOP 2.07 – The Excel Analytical Report Template for information on processing reports.

12. METHOD PERFORMANCE, DETECTION LIMITS:

12.1. Method Detection Limits and Reporting Limits

12.1.1. The Reporting Limit (RL) for each compound is set to be the concentration of the lowest standard (0.5 ml standard). The RL values are obtained from the standard worksheet used for the calibration values in the initial calibration table.

12.1.2. The Method Detection Limit (MDL) values for each compound are obtained from the latest MDL study which is stored on the EAS_Server_MDL Studies\[instrument ID].

12.1.2.1. The MDL studies are updated each year or when there has been a major change instrument performance or operating procedures.

12.1.2.2. All results between the RL and the MDL are "J" flagged indicating that they are approximate concentrations.

13. DATA ASSESSMENT:

13.1. Determine whether the results of the DAB meet the QC requirements for the project.

13.2. Do a sample calculation to verify the accuracy of the calculations, conversions to $\mu g/m^3$, and the transfers of electronic files and spreadsheets.

13.3. Check for Transcription errors, omissions, and mistakes

13.4. Check to verify that the appropriate method, SOP, and target list have been used.

13.5. Enter any exceptions to the QC requirements or other quality comments in the case narrative.

13.6. There should be three-tier review process on all reports

13.1.1. The data should be 100% reviewed by the data entry person

13.1.2. The data should be 100% reviewed by a project manager other then a project manager that participated in data entry (second person review).

13.1.3. Final review by the Lab Director to verify reviews and do a sanity check. Quality Manager will review 10% of the packages for QC requirements.

14. HANDLING UNACCEPTABLE DATA:

14.1. Unacceptable Data is Data that does not meet all of the QC requirements either for the method or project specific QC.

14.2. When the analyst, data entry person, or project manager identifies any data that appears to be unacceptable, they will contact the Lab Director.

14.3. The Lab Director will determine if the data is unacceptable, and then either schedule a rerun of the sample, contact the client to determine if the data is acceptable, or will determine from prior experience with the project that the data is acceptable with a note in the case narrative.

15. REFERENCES:

15.1. S.D. Hoyt, Oregon Graduate Center, Beaverton, Oregon Ph.D., 1982. Environmental Science and Engineering Dissertation: Air-Sea Exchange of Atmospheric Trace Gases.

15.2. S.D. Hoyt, & R.A. Rasmussen, "Determining Trace Gases in Air and Seawater", Mapping Strategies in Chemical Oceanography, ACS Advances in Chemistry Series 209, American Chemical Society, Washington, D.C., 1985

15.3. Quality Assurance Division, EMSL, U.S. EPA, Compendium of Methods for the Determination of Toxic Organic Compounds in Ambient Air, May, 1998

15.4. Center for Environmental Research, U.S. EPA, Compendium of Methods for the Determination of Toxic Organic Compounds in Ambient Air, Second Edition, EPA/625/R-96/01b, January 1999.

15.5. S.D. Hoyt, V. Longacre, M. Straupe, "Measurement of Oxygenated Hydrocarbons and Reduced Sulfur Gases by Full-Scan Gas Chromatography/Mass Spectrometry (GC/MS): EPA Method TO-14", Sampling and Analysis of Airborne Pollutants, p. 133, Edited by Eric Wineger, L.H. Keith, Lewis Publications, 1993.

15.6. Office of Emergency and Remedial Response, U.S. EPA, U.S. Environmental Protection Agency Contract Laboratory Program, February, 1990.

15.7. T.J. Kelly, M.W. Holdren, Atmospheric Environment, 1995, 19,2595-2608

16. CORRECTIVE ACTIONS:

16.1. The following table lists the corrective actions for this method.

Corrective Actions

QC Parameter Out of Control	Corrective Action
Holding Times	1. Prepare to extract / analyze the samples immediately. 2. Inform the EAS project chemist immediately so that impact to data usability can be assessed. 3. Depending on impact to data usability, the sample will either be extracted / analyzed outside the holding time or a new sample will be collected.
Initial Calibration	1. Evaluate system. 2. Recalibrate as necessary. 3. Analyze samples only after initial calibration is acceptable.
Continuing Calibration	1. Evaluate system. 2. Reanalyze standard. 3. Recalibrate as necessary. 4. Reanalyze affected samples
Method Blank	1. Evaluate system. 2. Re-extract and reanalyze method blank and associated samples. 3. Analyze samples only after method blank is acceptable.
LCS Recovery	1. Evaluate system. 2. Re-extract and reanalyze LCS and associated samples within the holding time. 3. Report sample data only after LCS is acceptable
Surrogate Recovery	1. Evaluate system. 2. Reanalyze sample within the holding time. If acceptable, report acceptable data only. 3. If unacceptable, attempt to re-extract and reanalyze the sample within the holding time (expiration of holding time does not remove the need to re-extract and reanalyze the sample). 4. If no control exceedance is observed and the reanalysis is within the holding time, report acceptable data for sample and surrogate. 5. If a control exceedance is observed, or if reanalysis not within the holding time, report both sets of sample and surrogate data.
Internal Standard Recovery	1. Evaluate system. 2. Reanalyze sample within the holding time. If acceptable, report acceptable data only. 3. If unacceptable, attempt to re-extract and

Corrective Actions

QC Parameter Out of Control	Corrective Action
	reanalyze the sample within the holding time (expiration of holding time does not remove the need to re-extract and reanalyze the sample). 4. If no control exceedance is observed and the reanalysis is within the holding time, report acceptable data for sample and internal standards. 5. If a control exceedance is observed, or if reanalysis is not within the holding time, report both sets of sample and internal standard data.
MS/MSD Recovery and RPD	1. Evaluate system. 2. Reanalyze MS/MSD. IF acceptable, report acceptable data only. 3. If unacceptable, re-extract and reanalyze MS/MSD and report both sets of MS/MSD data.
Matrix Duplicate RPD	1. Evaluate system. 2. Reanalyze matrix duplicate. If acceptable, report acceptable data only. 3. If unacceptable, re-extract and reanalyze matrix duplicate and report both sets of matrix duplicate data.
Raised PQLs	1. Notify the EAS project chemist and document in the laboratory case narrative any raised PQLs due to matrix interference's and/or large sample dilutions.
Field-generated Blanks (includes trip blanks, equipment blanks, and field water blanks)	1. Evaluate method blank. 2. Evaluate field sampling and decontamination procedures. 3. Evaluate field water source. 4. Modify sampling and decontamination procedures, as appropriate.

17. TROUBLESHOOTING:

17.1. For Mechanical problems with the GC or MS consult with the troubleshooting guides in the manufacturers instrument manuals.

17.1.1. For GC: HP 5890A Gas Chromatograph Reference Manual, Volume II

17.1.1.1. Chromatographic Troubleshooting: Section 15

17.1.1.2. Electronic Troubleshooting: Section 19

17.1.2. For MS: There are two types of MS units. First consult the manual for the MS model being checked, then consult the manual for the other type of MS for additional ideas.
17.1.2.1. HP 5970B Mass Selective Detector – Hardware Manual. See Section 5.0.
17.1.2.2. HP 5971A MSD – Hardware Manual. Chapter 5

17.2. Routine Maintenance: Consult the manufacturers instrument manuals for routing maintenance information.

17.1.1. For GC: HP 5890A Gas Chromatograph Reference Manual, Volume I
17.2.1.1. Chromatographic Troubleshooting: Section 12
17.1.2. For MS: There are two types of MS units. First consult the manual for the MS model being checked, then consult the manual for the other type of MS for additional ideas.
17.2.2.1. HP 5970B Mass Selective Detector – Hardware Manual. See Section 4.0.
17.2.2.2. HP 5971A MSD – Hardware Manual. Chapter 6

18. SAMPLE COLLECTION, SHIPPING, STORAGE:

18.1. The sample collection procedures are described in a separate SOP.
18.2. The Holding Time for samples is given in the QC Criteria. Generally the holding time for Canisters is 30 days, although there are project specific holding times of 14 days or less depending on the project.
18.3. SUMMA canisters have not special storage or shipping requirements. Canisters are stored on shelves in the laboratory.

19. WASTE MANAGEMENT, POLLUTION PREVENTION:

19.1. There are no wastes generated by this method.

20. DEFINITIONS:

20.1. Analyte: The specific chemicals or components for which a sample is analyzed.
20.2. Calibration Standard: A substance or reference material used to calibrate an instrument.
20.3. Component: A single chemical entity, such as an element or compound.
20.4. Compromised Samples: Samples which are improperly samples, insufficiently documented, improperly collected, or exceeded holding times then delivered to lab.

20.5. Corrective Action: The action taken to eliminate the cause of a nonconformity, defect, or undesirable situation to prevent recurrence.
20.6. Detection Limit: The lowest concentration or amount of a target analyte that can be identified, measured, and reported with comfidence that the concentration is not a false positive value.
20.7. Holding Time: The time elapsed from the time of sampling to the time of extraction or analysis.
20.8. Internal Standard: A known amount of standard added to a test portion of a sample as a reference for evaluating and controlling precision.
20.9. Laboratory Control Sample: A sample matrix free of analytes of interest, spiked with verified known amounts of analytes. Used to establish intra-laboratory bias or assess the performance of all or a portion of the measurement system.
20.10. Matrix: The component or substrate that contains the analyte of interest.
20.11. Method Detection Limit: The minimum concentration of a substance that can be measured and reported with 99% confidence that the analyte concentration is greater then zero.
20.12. Quality Control: The overall system of technical activities whose purpose is to measure and control the quality of a product or service.
20.13. Quantitation Limits: Levels of target analytes that can be reported at a specific degree co confidence.
20.14. Spike: A known mass of target analysts added to a blank sample to determine the recovery efficiency for QC purposes.
20.15. Validation: The process of validating specified performance criteria.

21. TABLES, DIAGRAMS, FORMS:

21.1. See the following pages.

ENVIRONMENTAL ANALYTICAL SERVICE INC. **MSD #1**

INSTRUMENT LOGBOOK

EPA METHOD: _____ RUN METHOD: _____ BATCH DATE: _____

ANALYST: _____ TUNE: _____ EMV:_____ IS: _____

IS VOL: _____ INITIAL CAL STD / IS 1 : _____ CON CAL STD / IS 1: _____

INITIAL CAL STD / IS 2: _____ CON CAL STD / IS 2: _____

COMMENTS:

FILE NAME	CAN NO.	DESCRIPTION	VOLUME	COMMENTS

MSD #1 DAILY INSTRUMENT PERFORMANCE

ANALYST: _____

DATE: _____

☐ SETUP ☐ GASES ON

☐ VALVES IN START POSITION

☐ HEAT AREAS IN START POSITION

☐ LINES CONNECTED

GC OVEN TEMP

☐ TANK LEVEL AIR: N2:

(includes Liquid Nitrogen) HE: N2(l):

☐ GAS FLOW CHECK AIR:

FLOW:

☐ TUNE PEAK ☐ MS TEMP ☐

AIR LEAK ☐ VACUUM ☐

ISO RATIO ☐ GC OVEN TEMP ☐

☐ LOAD METHOD

☐ STANDARD RUN VERIFY CORRECT LOOPS and STANDARD

CORRECTIVE ACTION OR REPAIR FOR QC FAILURE

☐ Issued Repair Order Date: _____ Analyst: _____

Environmental Analytical Service
STANDARD OPERATING PROCEDURE

SOP Number: 11.TO15.02

EAS Method: EPA TO15 Volatile Organics by GC/MS SIM (MSD1)

EPA Method: TO15 The Determination of Volatile Organic Compounds (VOCs) in Ambient Air & Source Using SUMMA Passivated Canisters and Gas Chromatographic Analysis.

Written By: Steve Hoyt

Approval: _____
Lab Director

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1. MATRIX, INTERERENCES, SCOPE:

This method can be used to analyze for volatile organic compounds (VOCs) in ambient air samples collected in SUMMA canisters or Tedlar bags. This method can be used for TO-14, TO-14A and TO-15. The EAS QC criteria is different for each of these methods and must be checked prior to analysis.

2. METHOD SUMMARY:

The ambient sample is loaded through a Carbo trap to remove water vapor, carbon dioxide, and concentrated on a stainless steel trap packed with glass beads. The VOCs are separated using a 60 meter fused capillary column directly connected to the source of an HP 5970 MSD. Depending on the compounds and volume of the sample analyzed, the Method Detection Limit (MDL) can be as low as 0.01 parts-per-billion by volume (ppbv) and as high as 20 ppbv (depending on linearity) for an ambient air sample. The use of selected ion monitoring (SIM) greatly improves the method detection limit (MDL) for VOC's. It is preferable to use a short list

of compounds for the SIM to get better MDL values, but this method can be used for the full TO-14 list.

3. HOLDING TIMES, PRESERVATION, CONTAINERS:

The standard holding time as specified in EPA TO15 is 30 days. However some clients have a project specific holding time of 14 days.

4. SAFETY:

4.5. The method uses Liquid Oxygen which can cause frostbite.

4.6. Safety goggles and gloves are recommended.

4.7. Secure all pressurized gas cylinders with chains.

4.8. Do not wear open-toed shoes.

5. EQUIPMENT, SUPPLIES:

5.13. Hewlett Packard 5890 Gas Chromatograph
5.14. Hewlett Packard 5970 Mass Selective Detector
5.15. Hewlett Packard Chemstation Software
5.16. NUTECH 8533 Universal Sample Concentrator
5.17. NUTECH Trap Temperature Controller
5.18. Zero Air, Helium, Compressed Nitrogen, and Liquid N_2 tanks
5.19. NIST traceable Standard cylinder(s)
5.20. Electrically heated Cryotrap
5.21. Liquid O_2
5.22. 2 metal-cased, cylindrical, glass Dewar flasks; 665 ml and 350 ml
5.23. NIST traceable flow controller for determining the sample volume
5.24. NIST traceable timer for determining the sample volume

6. REAGENTS, STANDARDS:

6.5. Internal Standard Mix. A 0.1 ppmv mixture of d3-vinyl chloride, aaa-trifluorotoluene, toluene-d8, and d3-methyl bromide is used for the internal standard and surrogate. A 2.0 ml volume is used for the internal standard.

6.6. Primary Calibration Standards: Commercial Gas Standard Mix: Scott Marrin and Scott Specialty NIST traceable 100 ppbv gas standard for TO-14 Target compounds which includes the SIM Target Compounds. A 1.0 ml volume is used for dilution.

6.7. Primary Calibration Standard: TO-15 Compound List. For the EAS TO-15 compound list the Primary Calibration Standard is made from neet materials and is NIST traceable by weight. The standard is prepared from three mixes. The TO-15 list is made at 1 ppmv, the 8260 List is made at 1 ppmv, and the 8260 Special List is made at 2 ppmv. The standard is prepared in an AL150 cylinder according to the Standard Preparation SOP 1.05.

6.8. Secondary Calibration Standard for TO-14: A 1 ppmv commercial TO-14 gas standard is used for a secondary calibration standard. A 0.1 ml volume is used for dilution.

6.9. Laboratory Control Spike forTO-14 Compound List. Commercial gas standard from Scott Marrin, Spectra, or Scott Specialty NIST traceable 1 ppmv gas standards for TO-14 target compounds.

7. STANDARDIZATION, CALIBRATION:

See Section 10 for initial and continuing calibration criteria.

8. OPERATING CONDITIONS:

Nutech Clamshell Heater to 280 C

Nutech Oven Temperature 120-150C

Nutech Heater to trap 195 C

GC/MS Conditions

Capillary Column: 60 meter, 0.25 DB-5 column
Carrier Gas: Helium, 20 psig column pressure; 150 ml/min (NUTECH)
Sample Flow: 100 ml/min

Summary of Method Parameters (Full Scan Regular List)

EM volts: based on tune
Mass scan: 35 to 260 amu
Scan/sec: 1.6
Threshold: 500

Temperature Program: -10°C, 3 min
Ramp Rate: 6°C/min
Final Temp: 203°C for 0.5 min
Run Time: 38 min

9. PROCEDURE:

9.2. Daily Startup

9.1.11. Refer to the primary SOP for this instrument

9.2 Daily Standardization

9.2.18. Refer to the primary SOP for this instrument

9.3. Laboratory Control Spikes

9.3.6. Refer to the primary SOP for this instrument

9.4. Zero Air Blank / Method Blank / System Blanking

9.4.9. Refer to the primary SOP for this instrument

9.5. Loading Ambient/Low Level Samples

9.5.11. Refer to the primary SOP for this instrument.

9.6 Loading Source Samples

9.6.5. Refer to the primary SOP for this instrument

10. QUALITY CONTROL CRITERIA:

Refer to the primary SOP for this instrument

11. CALCULATIONS, DATA PROCESSING:

11.2. Automated Data Processing

11.1.1. Refer to the primary SOP for this instrument.

12. METHOD PERFORMANCE, DETECTION LIMITS:

12.1. Refer to the primary SOP for this instrument

13. DATA ASSESSMENT:

13.1 Refer to the primary SOP for this instrument.

14. HANDLING UNACCEPTABLE DATA:

14.1 Refer to the primary SOP for this instrument.

15. REFERENCES:

15.1 Refer to the primary SOP for this instrument.

16. CORRECTIVE ACTIONS:

16.1 Refer to the primary SOP for this instrument.

17. TROUBLESHOOTING:

17.1 Refer to the primary SOP for this instrument

18. SAMPLE COLLECTION, SHIPPING, STORAGE:

18.1 Refer to the primary SOP for this instrument.

19. WASTE MANAGEMENT, POLLUTION PREVENTION:

19.1 Refer to the primary SOP for this instrument.

20. DEFINITIONS:

20.1 Refer to the primary SOP for this instrument

21. TABLES, DIAGRAMS, FORMS:

21.1 Refer to the primary SOP for this instrument.

ATTACHMENT 4

DETERMINATION OF USEPA METHOD TO-15 FULL SCAN MODE REPORTING LIMIT SUFFICIENCY

ATTACHMENT 4
DETERMINATION OF USEPA METHOD TO-15 FULL SCAN MODE REPORTING LIMIT SUFFICIENCY
(Page 1 of 2)

Compound	CAS Number	MDL ppbv	Reporting Limit ppbv	MDL µg/m^3	Reporting Limit µg/m^3	Reporting Limit OK?[1]	RL Needed µg/m^3	TO-15 SIM RL µg/m^3
1,1,1,2-Tetrachloroethane	630-20-6	0.1	0.51	0.72	3.62	NO	1.12	0.18
1,1,1-Trichloroethane	71-55-6	0.1	0.52	0.58	2.89	YES	--	--
1,1,2,2-Tetrachloroethane	79-34-5	0.1	0.52	0.73	3.65	NO	0.14	0.18
1,1,2-Trichloroethane	79-00-5	0.1	0.51	0.57	2.86	NO	0.52	0.14
1,1-Dichloroethane	75-34-3	0.1	0.52	0.43	2.15	YES	--	--
1,1-Dichloroethene	75-35-4	0.1	0.52	0.42	2.13	YES	--	--
1,1-Dichloropropene	563-58-6	0.1	0.49	0.46	2.3	YES	--	--
1,2,3-Trichloropropane	96-18-4	0.11	0.55	0.68	3.39	NO	0.015	0.16
1,2,4-Trichlorobenzene	120-82-1	0.1	0.52	0.79	3.94	YES	--	--
1,2,4-Trimethylbenzene	95-63-6	0.1	0.52	0.52	2.61	YES	--	--
1,2-Dibromo-3-chloropropane	96-12-8	0.22	1.1	2.2	11.0	NO	0.90	0.26
1,2-Dibromoethane	106-93-4	0.1	0.52	0.82	4.09	NO	0.015	0.20
1,2-Dichlorobenzene	95-50-1	0.1	0.52	0.64	3.2	YES	--	--
1,2-Dichloroethane	107-06-2	0.1	0.52	0.43	2.15	NO	0.32	0.11
1,2-Dichloropropane	78-87-5	0.1	0.52	0.49	2.46	NO	0.43	0.12
1,3,5-Trimethylbenzene	108-67-8	0.1	0.52	0.53	2.64	YES	--	--
1,3-Dichlorobenzene	541-73-1	0.1	0.52	0.64	3.2	YES	--	--
1,3-Dichloropropane	142-28-9	0.11	0.54	0.52	2.58	YES	--	--
1,4-Dichlorobenzene	106-46-7	0.1	0.52	0.64	3.2	NO	1.32	0.16
1,4-Dioxane	123-91-1	0.09	0.44	0.33	1.64	YES	--	--
2,2-Dichloropropane	594-20-7	0.11	0.53	0.5	2.53	YES	--	--
2-Butanone	78-93-3	0.09	0.43	0.26	1.31	YES	--	--
2-Hexanone	591-78-6	0.09	0.44	0.37	1.86	YES	--	--
Acetone	67-64-1	0.09	0.45	0.22	1.1	YES	--	--
Acetonitrile	75-05-8	0.22	1.12	0.48	2.39	YES	--	--
Benzene	71-43-2	0.1	0.52	0.34	1.7	NO	1.08	0.085
Benzyl chloride	100-44-7	0.09	0.45	0.48	2.41	NO	0.17	0.14
Bromochloromethane	74-97-5	0.1	0.51	0.55	2.76	YES	--	--
Bromodichloromethane	75-27-4	0.08	0.4	0.55	2.77	NO	0.47	0.18
Bromoform	75-25-2	0.09	0.47	0.99	4.96	YES	7.57	0.25
Bromomethane	74-83-9	0.1	0.51	0.41	2.04	YES	--	--
Carbon disulfide	75-15-0	0.09	0.45	0.29	1.45	YES	--	--
Carbon tetrachloride	56-23-5	0.1	0.52	0.67	3.38	NO	0.55	0.17
Chlorobenzene	108-90-7	0.1	0.52	0.5	2.48	YES	--	--
Chloroethane	75-00-3	0.1	0.51	0.28	1.39	YES	--	--
Chloroform	67-66-3	0.1	0.52	0.52	2.59	NO	0.36	0.13
Chloromethane	74-87-3	0.1	0.51	0.22	1.09	YES	--	--
cis-1,2-Dichloroethene	156-59-2	0.1	0.52	0.42	2.11	YES	--	--
cis-1,3-Dichloropropene	10061-01-5	0.1	0.52	0.48	2.41	YES	--	--
Dibromochloromethane	124-48-1	0.09	0.44	0.77	3.87	NO	0.35	0.23
Dibromomethane	74-95-3	0.11	0.55	0.97	4.84	YES	--	--
Dichlorodifluoromethane	75-71-8	0.1	0.51	0.52	2.61	YES	--	--
Dichloromethane	75-09-2	0.1	0.52	0.37	1.86	YES	--	--
Ethanol	64-17-5	0.22	1.12	0.44	2.18	YES	--	--
Ethylbenzene	100-41-4	0.1	0.52	0.46	2.33	YES	--	--
Freon 113	76-13-1	0.1	0.52	0.81	4.07	YES	--	--
Hexachlorobutadiene	87-68-3	0.1	0.52	1.14	5.68	NO	0.37	0.28
Isobutyl alcohol	78-83-1	0.23	1.13	0.84	4.21	YES	--	--
Isopropylbenzene	98-82-8	0.11	0.57	0.58	2.89	YES	--	--

ATTACHMENT 4
DETERMINATION OF USEPA METHOD TO-15 FULL SCAN MODE REPORTING LIMIT SUFFICIENCY

Compound	CAS Number	MDL ppbv	Reporting Limit ppbv	MDL µg/m^3	Reporting Limit µg/m^3	Reporting Limit OK?[1]	RL Needed µg/m^3	TO-15 SIM RL µg/m^3
Isopropyltoluene	99-87-6	0.11	0.55	0.62	3.12	YES	--	--
m & p-Xylene	108-38-3	0.21	1.03	0.92	4.61	YES	--	--
Methyl iodide	4227-95-6	0.19	0.94	1.13	5.67	YES	--	--
Methyl Isobutyl Ketone	108-10-1	0.09	0.46	0.38	1.95	YES	--	--
Methyl tert butyl ether	1634-04-4	0.08	0.39	0.29	1.45	YES	--	--
Naphthalene	91-20-3	0.22	1.09	1.19	5.9	NO	0.24	0.14
n-Butylbenzene	104-51-8	0.1	0.52	0.59	2.95	YES	--	--
n-Heptane	142-82-5	0.08	0.42	0.35	1.78	YES	--	--
n-Propylbenzene	103-65-1	0.11	0.54	0.55	2.74	YES	--	--
o-Xylene	95-47-6	0.1	0.52	0.46	2.31	YES	--	--
sec-Butylbenzene	135-98-8	0.11	0.52	0.59	2.95	YES	--	--
Styrene	100-42-5	0.1	0.52	0.45	2.26	YES	--	--
tert-Butylbenzene	98-06-6	0.11	0.52	0.59	2.85	YES	--	--
Tetrachloroethene	127-18-4	0.1	0.52	0.72	3.61	NO	1.39	0.18
Toluene	108-88-3	0.1	0.52	0.4	2.0	YES	--	--
trans-1,2-Dichloroethene	156-60-5	0.09	0.44	0.36	1.8	YES	--	--
trans-1,3-Dichloropropene	10061-02-6	0.1	0.52	0.48	2.41	YES	--	--
Trichloroethene	79-01-6	0.1	0.52	0.57	2.85	NO	0.073	0.14
Trichlorofluoromethane	75-69-4	0.1	0.51	0.59	2.95	YES	--	--
Vinyl acetate	108-05-4	0.09	0.43	0.31	1.56	YES	--	--
Vinyl chloride	75-01-4	0.1	0.51	0.27	1.35	NO	0.46	0.068

Note: The actual reported MDL may vary based on Canister dilution or matrix interferences.

[1]Based on a comparison of the calculated indoor air concentration, based on the reporting limit and flux chamber specifications, to the USEPA Region 9 air preliminary remediation goal (PRG).

ATTACHMENT 5
SUMMARY OF FIELD QUALITY CONTROL

TABLE OF FIELD QC- TVA 1000 HYDROCARBON ANALYZER

QC PARAMETER	SPECIFICATION	FREQUENCY
Electronic Zero	Electronic Package Operational	Pre-Use Instrument Check
Instrument Blank	Within +5 Times MDL	Pre and Post-Use Calibration
Span Calibration	+30% of Span Gas Value	Pre and Post-Use Calibration

TABLE OF FIELD QC- SAMPLE COLLECTION

QC PARAMETER	SPECIFICATION	FREQUENCY
Field Media Blank	None- used to qualify field data	One Per Trip up to 5%
Field System Blank	None- used to qualify field data	One per Trip; minimum of 5%
Field Sample Replicate	RPD of 50	One per Trip; minimum of 5%