

**Compendium of Methods
for the Determination of
Toxic Organic Compounds
in Ambient Air**

Second Edition

Compendium Method TO-15

**Determination Of Volatile Organic
Compounds (VOCs) In Air Collected In
Specially-Prepared Canisters And
Analyzed By Gas Chromatography/
Mass Spectrometry (GC/MS)**

**Center for Environmental Research Information
Office of Research and Development
U.S. Environmental Protection Agency
Cincinnati, OH 45268**

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6.2 Interferences in canister samples may result from improper use or from contamination of: (1) the canisters due to poor manufacturing practices, (2) the canister cleaning apparatus, and (3) the sampling or analytical system. Attention to the following details will help to minimize the possibility of contamination of canisters.

6.2.1 Canisters should be manufactured using high quality welding and cleaning techniques, and new canisters should be filled with humidified zero air and then analyzed, after "aging" for 24 hours, to determine cleanliness. The cleaning apparatus, sampling system, and analytical system should be assembled of clean, high quality components and each system should be shown to be free of contamination.

6.2.2 Canisters should be stored in a contaminant-free location and should be capped tightly during shipment to prevent leakage and minimize any compromise of the sample.

6.2.3 Impurities in the calibration dilution gas (if applicable) and carrier gas, organic compounds out-gassing from the system components ahead of the trap, and solvent vapors in the laboratory account for the majority of contamination problems. The analytical system must be demonstrated to be free from contamination under the conditions of the analysis by running humidified zero air blanks. The use of non-chromatographic grade stainless steel tubing, non-PTFE thread sealants, or flow controllers with Buna-N rubber components must be avoided.

6.2.4 Significant contamination of the analytical equipment can occur whenever samples containing high VOC concentrations are analyzed. This in turn can result in carryover contamination in subsequent analyses. Whenever a high concentration (>25 ppbv of a trace species) sample is encountered, it should be followed by an analysis of humid zero air to check for carry-over contamination.

6.2.5 In cases when solid sorbents are used to concentrate the sample prior to analysis, the sorbents should be tested to identify artifact formation (see Compendium Method TO-17 for more information on artifacts).

7. Apparatus and Reagents

[Note: Compendium Method To-14A list more specific requirements for sampling and analysis apparatus which may be of help in identifying options. The listings below are generic.]

7.1 Sampling Apparatus

[Note: Subatmospheric pressure and pressurized canister sampling systems are commercially available and have been used as part of U.S. Environmental Protection Agency's Toxic Air Monitoring Stations (TAMS), Urban Air Toxic Monitoring Program (UATMP), the non-methane organic compound (NMOC) sampling and analysis program, and the Photochemical Assessment Monitoring Stations (PAMS).]

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7.1.1 Subatmospheric Pressure (see Figure 1, without metal bellows type pump).

7.1.1.1 Sampling Inlet Line. Stainless steel tubing to connect the sampler to the sample inlet.

7.1.1.2 Sample Canister. Leak-free stainless steel pressure vessels of desired volume (e.g., 6 L), with valve and specially prepared interior surfaces (see Appendix B for a listing of known manufacturers/resellers of canisters).

7.1.1.3 Stainless Steel Vacuum/Pressure Gauges. Two types are required, one capable of measuring vacuum (-100 to 0 kPa or 0 to -30 in Hg) and pressure (0-206 kPa or 0-30 psig) in the sampling system and a second type (for checking the vacuum of canisters during cleaning) capable of measuring at 0.05 mm Hg (see Appendix B) within 20%. Gauges should be tested clean and leak tight.

7.1.1.4 Electronic Mass Flow Controller. Capable of maintaining a constant flow rate ($\pm 10\%$) over a sampling period of up to 24 hours and under conditions of changing temperature (20-40°C) and humidity.

7.1.1.5 Particulate Matter Filter. 2- μ m sintered stainless steel in-line filter.

using all reagents, standards, equipment, apparatus, glassware, and solvents that would be used for a sample analysis.

A laboratory method blank (LMB) is an unused, certified canister that has not left the laboratory. The blank canister is pressurized with humidified, ultra-pure zero air and carried through the same analytical procedure as a field sample. The injected aliquot of the blank must contain the same amount of internal standards that are added to each sample.

10.7.2 Frequency. The laboratory method blank must be analyzed after the calibration standard(s) and before any samples are analyzed.

Whenever a high concentration sample is encountered (i.e., outside the calibration range), a blank analysis should be performed immediately after the sample is completed to check for carryover effects.

10.7.3 Procedure. Fill a cleaned and evacuated canister with humidified zero air (RH >20 percent, at 25°C). Pressurize the contents to 2 atm.

The blank sample should be analyzed using the same procedure outlined under Section 10.8.

10.7.4 Calculations. The blanks are analyzed similar to a field sample and the equations in Section 10.5.4 apply.

10.7.5 Technical Acceptance Criteria. A blank canister should be analyzed daily.

The area response for each internal standard (IS) in the blank must be within ± 40 percent of the mean area response of the IS in the most recent valid calibration.

The retention time for each of the internal standards must be within ± 0.33 minutes between the blank and the most recent valid calibration.

The blank should not contain any target analyte at a concentration greater than its quantization level (three times the MDL as defined in Section 11.2) and should not contain additional compounds with elution characteristics and mass spectral features that would interfere with identification and measurement of a method analyte.

10.7.6 Corrective Action. If the blanks do not meet the technical acceptance criteria, the analyst should consider the analytical system to be out of control. It is the responsibility of the analyst to ensure that contaminants in solvents, reagents, glassware, and other sample storage and processing hardware that lead to discrete artifacts and/or elevated baselines in gas chromatograms be eliminated. If contamination is a problem, the source of the contamination must be investigated and appropriate corrective measures need to be taken and documented before further sample analysis proceeds.

If an analyte in the blank is found to be out of control (i.e., contaminated) and the analyte is also found in associated samples, those sample results should be "flagged" as possibly contaminated.

10.8 Sample Analysis

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10.8.1 Summary. An aliquot of the air sample from a canister (e.g., 500 mL) is preconcentrated and analyzed by GC/MS under conditions stated in Sections 10.1 and 10.2. If using the multisorbent/dry purge approach, adjust the dry purge volume to reduce water effects in the analytical system to manageable levels.

[Note: The analyst should be aware that pressurized samples of high humidity samples will contain condensed water. As a result, the humidity of the sample released from the canister during analysis will vary]

FINAL

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

October 2006

Prepared by:



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

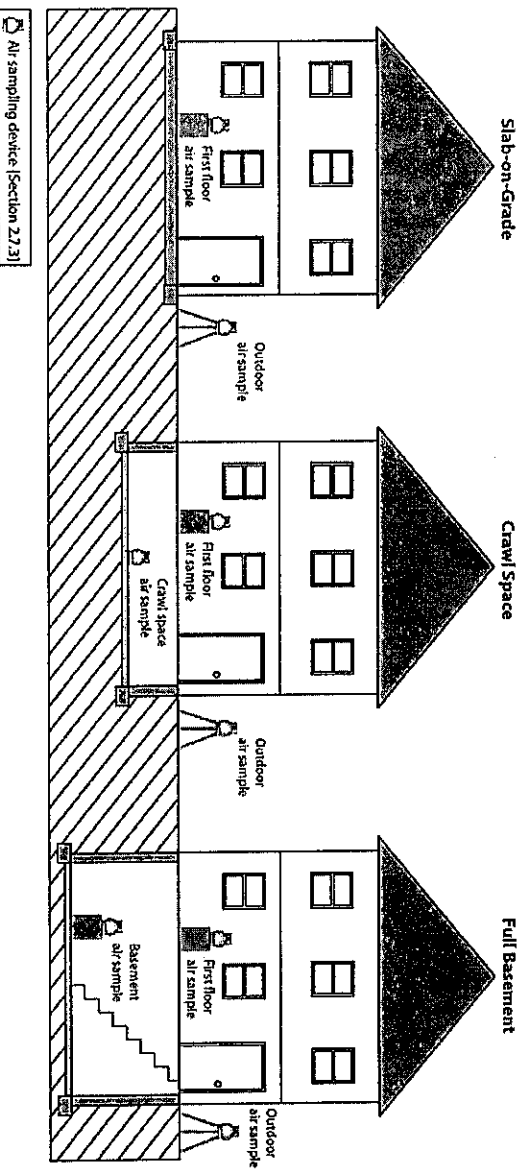


Figure 2.1

Schematic of indoor and outdoor air sampling locations

2.6.4 Outdoor air

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

* 2.7 Sampling protocols

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

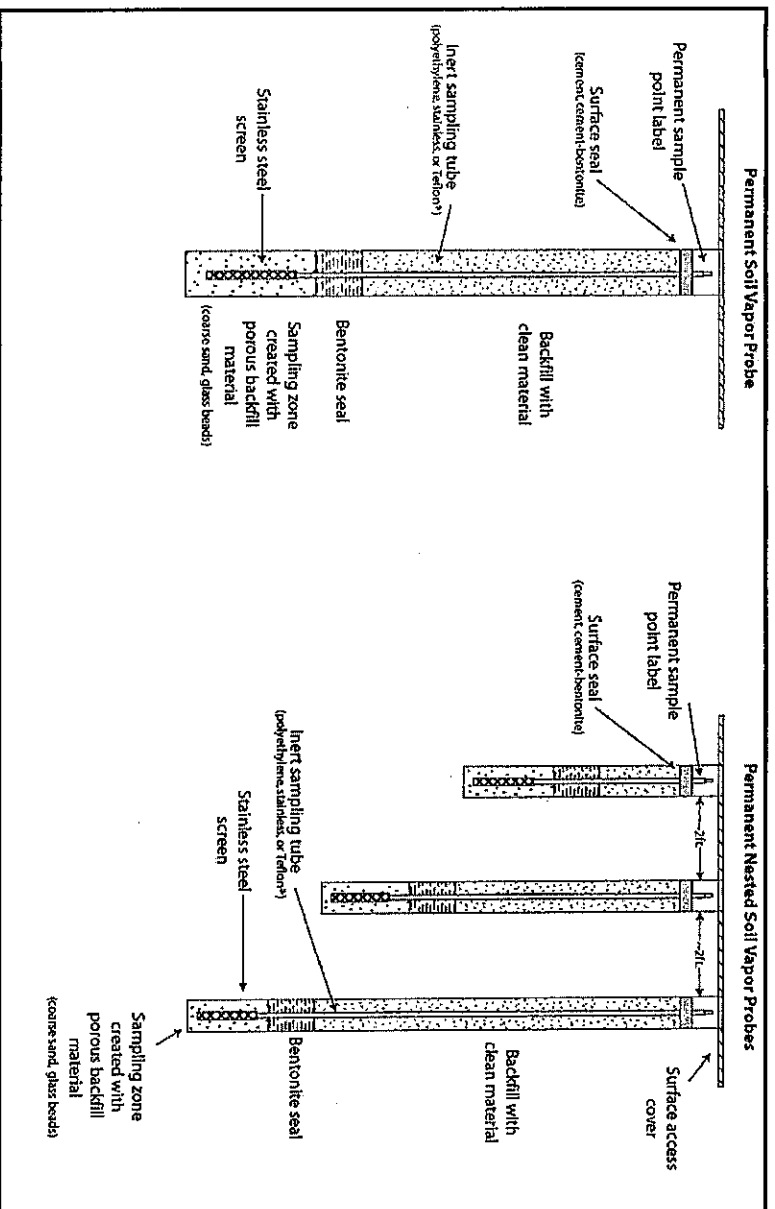


Figure 2.2

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

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

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

- a. at least 24 hours after the installation of permanent probes and shortly after the installation of temporary probes, one to three implant volumes (i.e., the volume of the sample probe and tube) should be purged prior to collecting the samples;
- b. flow rates for both purging and collecting should not exceed 0.2 liters per minute to minimize outdoor air infiltration during sampling;
- c. samples should be collected, using conventional sampling methods, in an appropriate container — one which
 - i. meets the objectives of the sampling (e.g., investigation of areas where low or high concentrations of volatile chemicals are expected; to minimize losses of volatile chemicals that are susceptible to photodegradation),
 - ii. is consistent with the sampling and analytical methods (e.g., low flow rate; Summa® canisters if analyzing by using EPA Method TO-15), and
 - iii. is certified clean by the laboratory;

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- d. sample size depends upon the volume of that will achieve minimum reporting limits [Section 2.9]; and
 - e. a tracer gas (e.g., helium, butane, sulfur hexafluoride, etc.) should be used when collecting soil vapor samples to verify that adequate sampling techniques are being implemented (i.e., to verify infiltration of outdoor air is not occurring) [Section 2.7.5].

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

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

- a. if sampling near a commercial or industrial building, uses of volatile chemicals during normal operations of the facility should be identified;
- b. outdoor plot sketches should be drawn that include the site, area streets, neighboring commercial or industrial facilities (with estimated distance to the site), outdoor air sampling locations (if applicable), and compass orientation (north);
- c. weather conditions (e.g., precipitation and outdoor temperature) should be noted for the past 24 to 48 hours; and
- d. any pertinent observations should be recorded, such as odors and readings from field instrumentation.

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

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

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

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

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

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

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

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

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

Results from EPA Funded Research Programs on the Importance of Purge Volume, Sample Volume, Sample Flow Rate and Temporal Variations on Soil Gas Concentrations

Blayne Hartman, H&P Mobile Geochemistry, Solana Beach, CA

Brian A. Schumacher and John H. Zimmerman, EPA-NERL, Las Vegas, NV

David S. Springer, R. James Elliot, Tetra Tech Inc., Santa Barbara, CA

Mark C. Riegby, Tetra Tech Inc., Lafayette, CA

ABSTRACT

Two research studies funded and overseen by EPA have been conducted since October 2006 on soil gas sampling methods and variations in shallow soil gas concentrations with the purpose of improving our understanding of soil gas methods and data for vapor intrusion applications. All studies consisted of a team of researchers. The EPA studies were conducted at a site with chlorinated solvent contamination at Vandenberg Air Force Base (CA). The first study consisted of controlled experiments on soil gas collection methods including purge volume, sample volume, and sample flow rate. Sample flow rate was found to not have a significant effect on soil gas concentrations for flows ranging from 100 cc/min to 5000 cc/min. There was a statistically significant positive correlation between the measured trichloroethylene (TCE) concentrations and purge volume ranging from 1 to 20 dead-space (system) volumes. The effect of purge volume on the measured volatile organic compound (VOC) concentrations was more pronounced than the effect of purge rate; however, this variability may not be significant in terms of site characterization. * Measured VOC concentrations were observed to increase with increasing sample volume from 25 to 1,000 ml, but then drop off in the 6,000 ml samples. This observation is significant as the 6,000 ml sample size is commonly used to achieve very low detection levels with EPA method TO-15. *

The second study investigated the temporal variation in shallow soil gas concentrations. In this study, soil gas concentrations were measured continuously around the clock for a period of 6 weeks at twelve sampling points in an uncovered field ranging in depth from 3' bgs to 17' bgs. The contamination source was TCE in groundwater. Over 11,000 analyses were collected by the automated continuous analyzer resulting in approximately 750 analyses per probe. Meteorological data were also collected. Observed TCE maximum and average concentration variations were less than 27% and 20%, respectively for all of the probes over the entire time period. Hence, meteorological variations had little effect on soil gas concentrations even as shallow as 3' bgs in a sandy soil with no surface covering.

INTRODUCTION

Soil gas data are widely used in site investigation and remediation projects to delineate volatile organic compound (VOC) contamination, as a screening tool to refine soil and groundwater sampling efforts, to track the progress of soil remediation, and to identify potential risks from vapor intrusion. Collection of representative samples, that is, samples that are representative of the environmental, chemical, and physical conditions present during the time of sample collection, is imperative for making responsible decisions. However, there currently exists doubt

over the effect of numerous environmental and sampling variables on measured soil gas concentrations.

To address this uncertainty, Environmental Sciences Division of the National Exposure Research Laboratory (NERL), a division of EPA's Office of Research and Development (ORD), funded a research program in the fall of 2006 to quantitatively assess the effect of certain sampling parameters on soil gas sample results, and a second program in the spring of 2007 to study temporal variations in soil gas concentrations due to changes in meteorological variables. The research program was designed and implemented by Tetra Tech, Inc. and H&P Mobile Geochemistry

STUDY site Description

The site selected for this research project was Vandenberg Air Force Base (VAFB), Installation Restoration Program (IRP) Site 15. VAFB is located on the central coast of California, approximately 120 miles west-northwest of Los Angeles and 225 miles southeast of San Francisco. IRP Site 15 is located approximately 1.5 miles from the Pacific Ocean and 1,300 feet north of San Antonio Creek on north VAFB. Tetra Tech has been investigating Site 15 under the VAFB IRP since 1993. A groundwater contamination plume exists at the site consisting primarily of trichloroethylene (TCE) and cis-1,2-dichloroethene (1,2 DCE). The maximum TCE and cis-1,2-DCE concentrations detected in this groundwater plume were 10,000 and 29 micrograms per liter ($\mu\text{g/L}$), respectively, in October 2005. Depth to groundwater was approximately 20 feet bgs at the time of the study.

Vadose zone soils in the study area consist of natural, undisturbed dune sand with sparsely vegetated cover (Figure 1). The sands have low moisture content and very low organic carbon. There were no buildings or surface coverings in the study area.

Figure 1. Photo of the study site showing sparsely vegetated sand dune environment. Mobile laboratory in center of photo. Conduit used to protect tubing from soil vapor probes to mobile lab visible in center. Weather station to right.

7.4.2

For the individual APH Calibration Standards, a pre-designated concentration is directly flow-injected into the concentrator/GC/MS by varying the volumes of the Working Standards. At a minimum, five different concentrations are required for a valid calibration curve. The concentrations must be evenly dispersed over the full working range of the detector with the lowest calibration point corresponding to the target RL. Tables 3a and 3b provide recommended concentrations and preparation methods for each Calibration Standard used in the initial calibration of hydrocarbon ranges and Target Analytes, respectively.

7.4.3

The range of volumes used for the APH Calibration Standards must be inclusive of the minimum and maximum sample volumes that will be used during routine sample analysis (e.g., as shown in Tables 3a and 3b, the minimum volume is 25 mL and the maximum volume is 250 mL). If sample volumes outside the range of calibration volumes are utilized, the laboratory must statistically demonstrate acceptable recovery of all target analytes over the full dynamic range of the calibration curve using the out-of-range injection volume. This statistical demonstration will be performed using the procedure described in Section 10.5, using the injection volume of interest with the higher concentration Working Standard. In any case, the minimum sample volume used should not be less than the manufacturer's recommendation for the concentrator (typically 20-25 mL).

7.5

Internal Standard and MS Tuning Standard

The recommended internal standards are Bromochloromethane, 1,4-Difluorobenzene, and Chlorobenzene-d5. The required MS tuning standard is BFB. Stock standards of these compounds should be prepared or purchased in a humidified canister at a concentration to accurately flow-inject a concentration of 10 ppbV or 10 µg/m³ into the trap during the collection time for all calibration, blank, and sample analyses, whether through a mass flow controller or a sample loop injector. The volume of internal standard mixture added for each analysis must be the same from run to run. The concentrations of internal standards can be assigned a nominal value of 10 ppbV or 10 µg/m³ for comparison and consistency with the laboratory's selected reporting units. This will vary among laboratories depending on which units are used during the calibration of the instrument.

8.0 SAMPLE COLLECTION AND HANDLING

8.1 Canister and Flow Controller Cleaning

All canisters must be leak tested and certified clean prior to being used for sampling. Associated canister sampling equipment (e.g., flow controllers, critical orifice assemblies) must also be deemed clean and appropriate for use prior to sampling. Cleaning techniques and acceptance criteria may vary between laboratories but, in general, procedures should include backflushing with humidified ultra zero air or UHP nitrogen. Flow controllers are calibrated with NIST-traceable flow meters. A detailed procedure for canister cleaning and maintenance is presented in Appendix 4.

8.2 Sample Collection

8.2.1 All samples must be accompanied by a chain-of-custody form, or equivalent, that documents the canister and flow controller serial numbers, date and time of sample collection, and all other pertinent sampling information.

8.2.2 Grab samples are collected by opening the sampling valve of a pre-evacuated canister (initial vacuum \geq 28 in. Hg) and allowing the canister to fill to ambient pressure. Equalization to atmospheric pressure under these conditions may be completed in a minute or less.

8.2.3 Time-integrated samples require the use of a properly calibrated flow controller. The flow controller's calibration must be performed and verified (by the laboratory) prior to sample collection. Upon receipt at the laboratory, a post-sampling flow controller calibration verification must be performed. The RPD between the initial and post sampling calibration readings must be calculated. As long as the RPD is \leq 20, the calibration and associated time interval are considered valid. If the RPD is $>$ 20, a notation must be provided in the data report form and case narrative disclosing the deficient RPD value. The flow controller RPD is one line of evidence in the proper

collection of samples for APH analysis. If the canister vacuum is acceptable after sampling and the flow controller RPD is outside of the acceptance criteria, data quality is not adversely affected.

Flow controllers will be calibrated such that a small amount of vacuum will remain in the canister at the end of sampling (approximately 5 in. Hg). The post-sampling canister vacuum will be measured by the laboratory using an annually calibrated, NIST-traceable vacuum/pressure gauge. The vacuum should be approximately 5 in. Hg to ensure a consistent flow rate throughout the measured time interval. However, due to temperature/pressure differences in the field, as well as site-specific conditions for various sampling applications (e.g., moisture levels, soil type, site access issues), the actual post-sampling canister pressure may be slightly different than 5 in. Hg.

8.2.4 Upon receipt at the laboratory, all samples must be assigned unique laboratory identification numbers.

8.2.5 The canister pressure of all grab and time-integrated samples must be measured and documented upon receipt at the laboratory. An annually calibrated NIST-traceable vacuum/pressure gauge is attached to the canister inlet, the sampling valve is briefly opened and the pressure is recorded. If the canister vacuum on receipt is > 15 in. Hg or if the canister vacuum measured on receipt at the laboratory differs from the final canister vacuum measured in the field by more than ± 5 in. Hg, the client should be contacted to determine if analysis should proceed. If client indicates that the analysis should proceed, the noted anomalies should be documented on the data report form or the case narrative.

8.2.6 Samples may be pressurized to a maximum of 5 psig with humidified ultra zero air or UHP nitrogen after receipt in the laboratory. Refer to Section 9.5.1.3 for the calculation of dilution factors for pressurized samples.

8.2.7 Documentation Requirements

8.2.7.1 Pre-Sampling Information: Provided by the Laboratory

- Canister Serial Number
- Individual or Batch Certification Results
- Canister Volume
- Pre-sampling Canister vacuum
- Flow controller serial number
- Date Canister Released by the laboratory

8.2.7.2 Sampling Information: Provided by the Sampler

- Site Location
- Sampling Date
- Sampling Location
- Sample Identification (ID)
- Canister Serial Number for each Sample ID
- Canister Volume (liters) for each Sample ID
- Sampling Duration
- Flow Controller Identification Number (if utilized) for each Sample ID
- Sampling Start and End Times
- Initial and Final Ambient Temperatures and Atmospheric Pressures
- Initial and Final Interior Temperatures
- Initial and Final Canister Vacuums (in. Hg)
- Date Shipped to Laboratory

USEPA CONTRACT LABORATORY PROGRAM

STATEMENT OF WORK

FOR

VOLATILE ORGANICS ANALYSIS

IN AIR

SAV01.X

Draft

June 2008

EXHIBIT D

ANALYTICAL METHOD FOR THE ANALYSIS OF VOLATILE ORGANIC COMPOUNDS (VOC)
IN AIR COLLECTED IN SPECIALLY-PREPARED CANISTERS AND ANALYZED
BY GAS CHROMATOGRAPHY/MASS SPECTROMETRY (GC/MS)

Appendix D -- Sections 7 & 8
Sample Collection, Preservation, Storage and Holding Times

7.2.7.2 The permeation system is usually held at a constant temperature to generate a constant concentration of trace gas. Commercial suppliers provide systems for generation and dilution of more than 250 compounds.

7.3 Storage of Standards

7.3.1 Working standards prepared in canisters may be stored for thirty days in an atmosphere free of potential contaminants.

7.3.2 It is required that a storage logbook be kept to document storage time.

8.0 SAMPLE COLLECTION, PRESERVATION, STORAGE AND HOLDING TIMES

8.1 Collection and Storage of Samples in Canisters

8.1.1 Samples are collected in leak-free stainless steel pressure vessels of desired volume (e.g., 6 L), with valve and specially prepared interior surfaces. All canisters must be certified as free from contaminants prior to sampling.

8.1.2 Each canister shall have a unique identification number and the laboratory must keep records of each canister's use for the life of the contract.

8.1.3 Canisters shall be stored at room temperature [22° (± 3°C)] in a contaminant free area. The temperature of the storage area must be recorded on a daily basis.

8.1.4 Samples must be analyzed within 30 days of collection.

8.2 Canister Cleaning Procedures

The canister cleaning procedures given in this section require that canister pressure be reduced to <0.05mm Hg before the cleaning process is complete. Depending on the vacuum system design (diameter of connecting tubing, valve restrictions, etc.) and the placement of the vacuum gauge, the achievement of this value may take several hours. In any case, the pressure gauge should be placed near the canisters to determine pressure. The objective of requiring a low pressure evacuation during canister cleaning is to reduce contaminants. If canisters can be routinely certified (< 0.2 ppbv for target compounds) while using a higher vacuum, then this criteria should always be followed. However, the ultimate vacuum achieved during cleaning should always be <0.2mm Hg. Canister cleaning as described in this section requires components with special features. The vacuum gauge must be capable of measuring 0.05mm Hg with less than a 20% error. The vacuum pump used for evacuating the canister must be noncontaminating while being capable of achieving the 0.05 mm Hg vacuum as monitored near the canisters. Thermoelectric vacuum gauges and turbomolecular drag pumps are typically used for these two components. An alternate to achieving the canister certification requirement of <0.2 ppbv for all target compounds is the criteria used in Compendium Method TO-12 that the total carbon count be <10ppbC. This check is less expensive and can be used if proven to be equivalent to the original requirement. This equivalency must be established by comparing the total nonmethane organic carbon (TNMOC) expressed in ppbC to the requirement that individual target compounds be <0.2 ppbv for a series of analytical runs.

6 Liter Can 7 day Recovery Test - 10% RH Filled to 10 psia

Compound	10 ppb Std	Can 3662 Silonite	Can 3661 Silonite	54106 Summa	15275 Summa	22094 Summa	54102 Summa	00405 TO Can	00490 TO Can	00548 TO Can	00408 TO Can
Fill/Run Order	1	8	2	5	3	7	10	9	4	6	11
Propene	10.00	9.58	9.73	8.87		8.89		8.80	9.04	9.01	9.05
Dichlorodifluoroethane	10.00	8.67	8.28	8.05		7.66		7.64			8.14
Chloromethane	10.00	8.50	8.53	7.91		7.79		7.82	7.92		7.89
Dichlorotetrafluoroethane	10.00	7.94	7.99								
Vinyl Chloride	10.00	8.38	8.39	7.69		7.70		7.65	7.79	7.54	7.83
1,3-Butadiene	10.00	8.53	8.46	7.84		7.93		7.80	8.00	7.82	8.08
Bromomethane	10.00	8.04	8.04	7.60							7.59
Chloroethane	10.00	8.43	8.53	7.94		7.79		7.66	7.83	7.88	7.99
Bromoethene	10.00	8.35	8.46	7.81		7.69		7.59	7.66	7.77	7.95
Trichlorofluoromethane	10.00	8.15	8.31	7.69		7.50			7.56	7.56	7.71
Acetone	10.00	9.28	11.59	10.02	13.07	8.84	9.68	10.40	11.70	10.08	10.79
Isopropyl Alcohol	10.00	8.75	8.86	8.13	10.92	8.26				9.25	
1,1-Dichloroethene	10.00	9.03	8.96	8.35		8.32		8.26	8.41	8.00	8.47
Trichlorotrifluoroethane	10.00	9.04	9.08	8.40		8.31		8.28	8.39	8.42	8.48
Allyl Chloride	10.00	9.31	9.40			8.41	3.07	8.13	8.48	8.76	8.08
Methylene Chloride	10.00	9.30	9.31	8.55		8.48		8.52	8.63	8.71	8.68
Carbon Disulfide	10.00	9.03	9.16	8.37		8.32		8.52	8.58	8.49	8.55
trans-1,2-Dichloroethene	10.00	9.29	9.38	8.60		8.55		8.51	8.63	8.78	8.71
Methyl tert-Butyl Ether	10.00	9.29	9.35	8.35		8.63		8.46	8.56	8.81	8.51
Vinyl Acetate	10.00	9.33	9.34	0.09		8.10	0.03	8.5	0.2	8.87	0.03
1,1-Dichloroethane	10.00	9.19	9.28	8.39		8.47		8.33	8.42	8.67	8.45
2-Butanone	10.00	9.62	9.77	8.34		8.64		8.31	8.37	8.90	8.21
n-Hexane	10.00	9.70	9.62	8.86		8.76		8.68	8.90	8.97	9.04
cis-1,2-Dichloroethene	10.00	9.38	9.41	8.72		8.66		8.57	8.73	8.90	8.80
Ethyl Acetate	10.00	9.51	9.73			8.83		7.92	7.50	9.09	
Chloroform	10.00	9.15	9.25	8.46		8.40		8.34	8.49	8.51	8.64
Tetrahydrofuran	10.00	9.57	9.82	8.63		8.94		8.52	8.71	9.16	8.50
1,1,1-Trichloroethane	10.00	9.36	9.45	8.62		8.61		8.47	8.67	8.77	8.69
1,2-Dichloroethane	10.00	9.29	9.40	8.61		8.49		8.49	8.64	8.73	8.74
Benzene	10.00	9.56	9.57	8.86		8.81		8.72	8.93	8.94	8.95
Carbon Tetrachloride	10.00	9.33	9.47	2.27				0.04	7.1	8.59	0.96
Cyclohexane	10.00	9.52	9.62	10.32		9.31		8.68	11.96	9.75	11.94
2,2,4-Trimethylpentane	10.00	13.42	10.97	10.17		11.14		9.45	11.61	11.61	12.45
n-Heptane	10.00	13.49	10.95	10.18		11.03		9.35	11.67	11.51	12.46
Trichloroethene	10.00	12.76	10.53	9.92		10.67		9.17	11.28	11.22	12.09
1,2-Dichloropropane	10.00	13.11	10.81	9.98		10.92		9.17	11.44	11.27	12.16
1,4-Dioxane	10.00	11.13	8.05			8.67		7.76	9.93	8.62	12.51
Bromodichloromethane	10.00	12.80	10.60	9.41		10.57		10.54	10.76	11.87	9.42
cis-1,3-Dichloropropene	10.00	13.01	10.75	9.36		10.78		8.08	11.20	11.27	12.16
4-Methyl-2-pentanone	10.00	13.35	10.79			11.23		8.41	8.08	12.57	
trans-1,3-Dichloropropene	10.00	12.92	10.72	7.81		10.61		10.76	11.00	12.18	10.31
Toluene	10.00	13.34	10.86	10.05	7.50	11.08		9.28	11.57	11.52	12.36
1,1,2-Trichloroethane	10.00	13.30	10.82	10.00		11.14		9.06	11.47	11.52	11.24
2-Hexanone	10.00	12.82	10.33			10.61				12.56	

6 Liter Can 7 day Recovery Test - 10% RH Filled to 10 psia

Compound	10 ppb Std	Can 3662 Silonite	Can 3661 Silonite	54106 Summa	15275 Summa	22094 Summa	54102 Summa	00405 TO Can	00490 TO Can	00548 TO Can	00408 TO Can
Fill/Run Order	1	8	2	5	3	7	10	9	4	6	11
Dibromochloromethane	10.00	13.18	10.90	9.19		10.74		10.05	10.76	12.15	7.78
Tetrachloroethene	10.00	13.35	10.85	10.18		11.16	9.17	11.60	11.51	12.42	11.14
1,2-Dibromoethane	10.00	12.84	10.53	9.63		10.73	8.56	11.20	11.07	12.00	10.86
Chlorobenzene	10.00	13.03	12.72	12.19	8.60	14.07	9.31	13.07	15.87	13.58	13.80
Ethylbenzene	10.00	13.27	12.72	12.14	8.77	14.29	9.69	13.21	15.88	14.11	13.66
m-Xylene	10.00	12.94	12.13	11.53	8.52	14.00	10.00	12.78	15.27	14.27	13.05
p-Xylene	10.00	12.21	11.99	11.12	8.17	13.50	8.98	12.43	14.75	13.15	12.65
Styrene	10.00	13.00	12.61	10.26	8.12	13.43	7.65	10.71	13.43	13.00	10.92
o-Xylene	10.00	13.14	12.59	11.84	8.70	14.25	9.85	13.01	15.62	14.00	13.49
Bromoform	10.00	12.88	12.71	10.49	8.41	13.24	2.67	10.27	14.00	13.14	6.99
1,1,2,2-Tetrachloroethane	10.00	12.78	12.47	10.99	8.39	13.47	8.20	12.50	15.12	12.97	13.05
4-Ethyltoluene	10.00	13.07	12.72	11.24	8.70	14.11	8.61	12.73	15.36	13.52	12.71
1,3,5-Trimethylbenzene	10.00	12.94	12.71	10.59	8.42	13.95	8.34	12.18	14.51	13.41	12.15
1,2,4-Trimethylbenzene	10.00	12.84	12.65	10.18	8.44	13.77	8.07	12.10	14.45	13.31	11.96
1,3-Dichlorobenzene	10.00	12.67	12.44	11.15	8.09	13.57	8.19	12.79	15.50	13.20	13.36
Benzyl Chloride	10.00	12.97	13.03	10.09		11.84	10.10	6.49		12.77	2.62
1,4-Dichlorobenzene	10.00	12.51	12.19	10.76	7.85	13.46	7.86	12.77	15.34	163.21	13.32
1,2-Dichlorobenzene	10.00	12.56	12.28	10.62	8.06	13.30	7.99	12.51	15.10	13.09	13.18
1,2,4-Trichlorobenzene	10.00	9.65	8.86			10.50		10.29	11.89	11.09	10.13
Hexachlorobutadiene	10.00	12.13	10.82	8.02		12.74		11.65	13.83	12.43	11.87