MONTEREY APARTMENTS
STATE OF WASHINGTON
REMEDIAL INVESTIGATION/FEASIBILITY STUDY
PILOT SOIL-GAS STUDY

SAMPLING ANALYTICAL PLAN
FINAL

Contract No.: C0089007

Work Assignment No. 1

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Prepared By: ECOLOGY AND ENVIRONMENT, INC.

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Prepared For: WASHINGTON STATE DEPARTMENT OF ECOLOGY
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Approved By:

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Ecology Quality Assurance Officer	Date	Lynn Guilford E & E Quality Assurance Officer	7/27/90 Date

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DOCUMENT DISTRIBUTION LIST
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1. INTRODUCTION

As a contractor to the Washington State Department of Ecology (Ecology), Ecology and Environment, Inc. (E & E) has the responsibility to implement minimum procedures to assure that the precision, accuracy, representativeness, comparability, and completeness of its data are known and documented. To ensure this responsibility is met uniformly, E & E must prepare a written analytical quality assurance plan for each sampling project it is tasked to perform.

This Sampling Analytical Plan was prepared for the Monterey Apartments reconnaissance pilot study under Work Assignment No. 1 (Contract No. C0089007).

The plan covers only activities for field and analytical aspects associated with the soil-gas pilot study. As subsequent sampling plan and quality assurance plan task phases emerge, formal State Remedial Investigation/Feasibility Study (State RI/FS) Quality Assurance Project Plan (QAPP) deliverables will be written to delineate Quality Assurance/ Quality Control (QA/QC) requirements required under the State RI/FS system.

2. PROJECT DESCRIPTION

2.1 SITE DESCRIPTION AND APPROACH

The Monterey Apartments site (622 First Avenue West) is situated in a mixed residential/light commercial area in the Queen Anne District of the City of Seattle. The apartment building basement living units have been impacted by free organic vapors originating from an underground storage tank gasoline spill. The fuel product appears to have been released from nearby service station tank(s) and migrated along a local, shallow perched water-table aquifer.

2.2 SAMPLE COLLECTION AND ANALYSIS

The State RI/FS Phase I pilot soil-gas study will involve the limited direct collection of soil-gas (3 to 8 samples) for gas chromatograph/mass spectrometer (GS/MS) analysis of volatile organic compound (VOCs) analytes. The samples will be delivered for analysis to the Ecology Manchester Laboratory that will follow, or use modified, United States Environmental Protection Agency (EPA) analytical methodologies (see Section 6). Specific QA requirements to be applied as part of the project analytical work are discussed in detail in the remaining sections of this plan.

2.3 DATA USE

Data generated as a part of the Monterey Apartments pilot soil-gas study will be used to determine the feasibility of applying soil-gas surveying techniques at the site. Specific issues to be addressed will include:

- Determining if the fill and native soil conditions of the immediate study area are conducive to adequate gas migration for successful detection of gas vapors;

- Establishing, and testing, the design and assembly of a soil-gas sampling device to collect samples which can be efficiently delivered and analyzed by the Ecology Manchester Laboratory;
- Establishing specific sampling protocols during the installation and acquisition of soil-gas samples in the field; and
- Applying the protocols, data, and information generated by the pilot study to the development of the Phase I Sampling Plan.

2.4 DATA QUALITY OBJECTIVE PROCESS

Data quality objectives (DQOs) were determined in consultation with Ecology staff and are based on the data-use requirements described in Section 2.3. The DQO development process was based on protocols set forth in Data Quality Objectives for Remedial Response Activities (EPA/540/G-87/003; Oswer Directive 9335.0-7; March 1987). Resulting in qualitative and quantitative statements that specify the quality of data required to support project decisions. These project specific DQOs are presented in terms of precision, accuracy, representativeness, completeness, and comparability in Section 4 of this plan.

The data quality objectives for soil-gas probe installation and sample collection during the pilot study are to initiate estimation of the extent of gasoline migration at the site, determine soil-gas VOC compositions, and begin to examine for detectable degradation as migration has occurred from the source(s). The data obtained will support the selection of a full-scale soil-gas investigation as part of the State RI.

2.5 SCHEDULE AND MILESTONES

Activities	Dates
Sampling Analytical Plan (Draft Final)	July 10, 1990
Field Sampling Work	July 16 to July 20

Activities	Dates
Laboratory Analysis	July 17 to July 23
Delivery of Laboratory Package	By August 14
E & E Data Validation	August 15 to August 29

3. PROJECT ORGANIZATION AND RESPONSIBILITY

Experienced senior staff have been assigned to the project QA/QC functions. E & E quality assurance contacts for the Monterey project include:

- Mr. Peter Jowise
  Program Manager
- Mr. John L. Roland
  Project Manager
- Ms. Lynn Guilford
  Quality Assurance Officer

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The various QA functions are explained below.

Program Manager

The program manager has overall responsibility for ensuring that work on the Monterey Apartments site meets client objectives and E & E quality standards. In addition, he will be responsible for technical quality control and project oversight, and will provide the project manager with access to corporate management.

Project Manager

The Project manager will be responsible for implementing the project and will have the authority to commit the resources necessary to meet project objectives and requirements. The Project manager's primary function is to ensure that technical, financial, and scheduling objectives are achieved successfully. The Project manager will report directly to Ecology, and will provide the major point of contact and control for matters concerning the project.

Technical Staff

The technical staff (team members) for this project will be drawn from E & E's pool of corporate resources. The technical team staff will be utilized to gather and analyze data, and to prepare various task reports and support materials. All of the technical team members are experienced professionals who possess the degree of specialization and technical competence required to effectively and efficiently perform the required work.

QA Officer

The QA officer is responsible for maintaining QA for the project. Specific functions and duties include:

- Providing an external, and thereby independent, QA function;
- Coordinating with Ecology QA Officers and the E & E program and Project manager, to ensure that QA objectives appropriate to the project are set;
- Coordinating with project management and personnel to ensure that QC procedures appropriate to demonstrating data validity sufficient to meet QA objectives are developed and in place;
- Requiring and/or reviewing actions taken in the event of QC failures;
- Reporting non-conformance with QC criteria or QA objectives, including an assessment of the impact on data quality or project objectives, to the site manager; and
- Performing audits, QA reports and other duties described in the Sampling Analytical Plan.

Data Validation Specialist

Duties and responsibilities for the Monterey Apartments site data validation specialist include the following:

- Assessing and summarizing the accuracy, precision, and reliability of data for determination of its usability; and
- Documenting for the historical record any factors affecting its usability.

4. QUALITY ASSURANCE OBJECTIVES FOR MEASUREMENT DATA

The overall data quality objective for the data uses presented in Section 2.3 of this plan is to produce data of known and documented quality with analytical results derived from Ecology approved analytical methods. The E & E Project Manager will coordinate all analytical requirements through the Ecology Manchester Laboratory quality assurance and sample control personnel.

4.1 PROJECT GOALS

Analytical services for the Monterey Soil-Gas Pilot Study work will conform to the modified EPA TO-14 GC analytical procedure approved and agreed upon by Manchester (see Section 6).

The characteristics of major importance for the assessment of generated data are: precision, accuracy, representativeness, completeness, and comparability (PARCC parameters). Project QA objectives/ targets for the PARCC parameters are presented in the following section.

4.2 QA OBJECTIVES/TARGETS

Data derived from soil-gas investigations serve as screening information on plume migration. The quantified data reported by laboratory analyses will be applied as a qualitative investigative indicator for plume delineation. The data do not represent direct quantitative correlations with either soil or groundwater volatile organic contaminant concentrations.

4.2.1 Precision and Accuracy

Target values for laboratory quantitation limits, method accuracy (percent spike recovery), and method precision (RPD of duplicates/ replicates) are specified in Section 6, and the Manchester analytical standard operating procedure (SOP) found in Appendix B. Ranges for quality assurance objectives presented for the modified TO-14 analyses represent the overall method limits. Note that high concentrations and matrix interferences can preclude achievement of quantitation limits or other QC criteria. In such instances, the progress reports to the Ecology Project Officer will indicate the occurrence and cause of any deviation from the tabulated QC criteria.

4.2.2 Representativeness

Representativeness expresses the degree to which data accurately and precisely represent a characteristic of a population, parameter variations at a sampling point, a process condition, or an environmental condition.

For the Monterey Apartments pilot study, representativeness of the data to be produced will be maximized through careful consideration of site conditions and location of sampling sites to adequately account for site variations. Sample locations will be chosen so as to maximize the probability of vadose zone contaminant presence. Although not representative of the wide-range of site conditions, the pilot study is focused on a measure of technique viability.

4.2.3 Comparability

Data produced for the study will not be directly comparable to data produced for other site investigations because similar sampling techniques and the specified analytical procedures have not been previously paired together in a manner specifically similar to the techniques to be applied. This is not expected to impact pilot study results due to the qualitative, exploratory nature of the intended data use.

The analytes identified by the modified TO-14 method (see Appendix B) will include EPA Target Compound List (TCL) VOC parameters, as well as other volatile gasoline components reported as Tentatively Identified Compounds (TICs). The list will include the detection of benzene, ethylbenzene, toluene, and xylene (BETXs) components. Quantitation limits will be equal to or less than 5 micrograms per cubic meter ($\mu g/M^3$). The sample holding time for this project has been established at 14 days.

4.2.4 Completeness

An overall target value of 80 percent completeness has been set for the Monterey soil-gas pilot study. The value was selected due to the pilot program nature of the study, and uncertainties surrounding soil conditions, sample collection, and laboratory approach.

5. SAMPLING PROCEDURES

Soil gas sampling to be conducted during the pilot study will be of limited extent and duration (see Section 2.5). Three to eight exploratory probes placed into soil in areas of known and suspect fuel contamination are scheduled. The probes will be constructed of threaded 1-inch O.D. hollow alloy steel probe rods. The 3-foot rod sections will be equipped with expendable steel drive points and threaded top drive cap assembly. The rods will be driven into the surface soils manually using sledge hammers. The depth of insertion will be dependent upon the composition of soils to be penetrated. The preferred depth of penetration will be 4 feet below ground surface (bgs), with a minimum depth of 2 feet bgs. If penetration can not be obtained, the location will be abandoned and an alternate location will be chosen.

Once in place, the probe will be fitted with a sampling train assembly utilizing Teflon tubing between the probe and sample canister. The probe annular space and soil pore space immediately surrounding the probe tip will be purged by pulling a minimum of 3 equivalent probe volumes of soil gas using an MSA or Gillian portable air sampling pump. The pumping rate for purging will be set at between 200 and 1,000 milliliters per minute. Once purged, the soil gas will be sampled by diverting flow, using stainless steel or Teflon/glass shut-off valves, to a separate line connected to 6-liter, Manchester certified blank, fully evacuated Suma canisters. The canister valves will be opened completely, and the samples will be directly displaced into the canisters. The filled canisters will then be closed, labeled, recorded and transported according to the procedures described in Section 9.

During the sampling effort, an HNu organic vapor analyzer will be used at each station to estimate the potential vapor concentration range (i.e., high [>500 ppm], medium [10 to 500 ppm], or low [<10 ppm]). In addition, an ambient air sample (blank) also will be collected during the effort to identify potential interference contamination conditions and to assure that atmospheric influences are not inadvertently incorporated into the soil-gas samples.

At least one inferred background soil-gas location will be tested to identify natural soil conditions. This will identify residual contaminants that may interfere with, or bias interpretation of, study area station results. Seattle area barometric pressure and temperature conditions will be obtained daily for supporting the laboratory data reduction calculations (see SOP, Appendix B).

6. ANALYTICAL PROCEDURES

Analytes of interest for this project (see Appendix B) are those compounds currently comprising the VOCs of the TCL as stipulated in the EPA Contract Laboratory Program (CLP) Statement of Work (SOW) for organic (2/88) analyses. The laboratory performing the analysis also will identify and report other TICs of gasoline derivation (20 TICs identified per sample) not included on the TCL. The analytical methodology will follow EPA Method TO-14; the method will be modified as directed by the Manchester Laboratory. Any significant alteration of the procedures will be reported on the **SAMPLE ALTERATION CHECKLIST** found in Appendix A.

Analyses provided by the Manchester Laboratory will conform to requirements stipulated in the request for analytical services prepared by the E & E chemistry group. In addition, the analysis will include a minimum of one laboratory duplicate to evaluate data agreement. The analyst will be requested to select an anticipated low concentration sample to be used as the duplicate. The laboratory will also analyze a minimum of one laboratory prepared blank. The E & E analytical request has been prepared in compliance with conditions set forth during the QA scoping meeting held at Manchester on June 13, 1990.

7. DATA REDUCTION, VALIDATION, AND REPORTING

7.1 GENERAL

Prior to submission of the draft final report, all data will be evaluated for precision, accuracy, and completeness. Specific procedures for data validation to be used on this project are presented in the following EPA document: CLP Laboratory Data Validation Functional Guidelines for Evaluating Organic Analyses (February 1, 1988). Laboratory deliverable requirements to be fulfilled by the performing laboratory are presented in Appendix B of this document.

7.2 DATA REDUCTION

Data reduction includes all processes that change the numerical value of the raw data. All laboratory data reduction will be performed by the Manchester Laboratory, or appropriate state contract laboratory, and presented as sample results; no further data reduction will be performed. Laboratories will be required to submit results as well as back-up data and QA/QC results as specified in the Laboratory Deliverable Requirements outlined in Appendix B.

7.3 DATA VALIDATION

Data validation procedures for the laboratory analyses will not be equivalent to CLP data validation. Validation of all analytical data will be performed by chemists at E & E. Validity of all data will be determined based on the precision, accuracy, and completeness assessments discussed in Section 4.

7.4 DATA REPORTING

Prior to the submission of the report to Ecology, all data will be evaluated for precision, accuracy, and completeness. Upon completion of the review, the chemist will develop a QA review report for the analytical package. The raw data package and respective QA review report generated from the project will be appropriately identified within the final soil-gas pilot study report.

8. CALIBRATION PROCEDURES AND FREQUENCY

All instruments and equipment used during sampling and analysis, specifically the HNu organic vapor analyzer and water level indicator, will be operated, calibrated, and maintained according to the manufacturer's guidelines and recommendations, as well as criteria set forth in the applicable analytical methodology references. Operation, calibration, and maintenance will be performed by personnel properly trained in these procedures. Documentation of all routine and special maintenance and calibration information will be maintained in an appropriate logbook or reference file, and will be available on request. Brief descriptions of calibration procedures for major field and laboratory instruments follow.

8.1 LABORATORY INSTRUMENTATION

Laboratory capabilities will be demonstrated for instrument and reagent/standards performance as well as accuracy and precision of analytical methodology as stated in Exhibit E of the CLP Statements of Work for Organic (2/88) analyses. Brief descriptions of calibration procedures for major instrument types follow.

Gas Chromatography/Mass Spectrometry. All GC/MS instruments will be calibrated at five different concentration levels for analytes of interest, using the procedures outlined in the EPA CLP procedures and standards traceable to EPA reference materials. Compounds selected as System Performance Check Compounds (SPCCs) must show a minimum mean response factor (specified) for the initial calibration to be considered valid. Compounds selected as Calibration Check Compounds (CCCs) must show a Relative Standard Deviation (RSD) for response factors less than a specified amount for the calibration to be considered valid. This calibration can be utilized as long as it can be demonstrated to remain valid.

On a 2-hour basis, the instrument is hardware-tuned using bromofluorobenzene (BFB) for volatile analytes. Mass spectral peaks must conform both in mass numbers and relative intensity to EPA's requirements before analysis can proceed.

Following a successful tuning, the initial five-point calibration is verified by a single mid-range standard. SPCCs and CCCs are used to check response factors for adequacy and consistency against required limits before analytes can proceed.

A successful continuing calibration is followed by a method blank analysis. All target analytes (with the exception of common laboratory solvents) must be below target detection limit before analysis can proceed.

9. SAMPLE CUSTODY

This section describes standard operating procedures for sample identification and chain-of-custody to be utilized. The purpose of these procedures is to ensure that the quality of samples is maintained during their collection, transportation, and storage through analysis. All sample control and chain-of-custody procedures will follow CLP's User's Guide (9240.0-1, December 1988) and the National Enforcement Information Center (NEIC) Enforcement Consideration for Evaluation of Uncontrolled Hazardous Waste Disposal Sites (April 1980).

Sample identification documents must be carefully prepared so that sample identification and chain-of-custody can be maintained and sample disposition controlled. Sample identification documents include:

- Field notebooks,
- Sample labels,
- Custody seals, and
- Chain-of-custody records.

9.1 CHAIN-OF-CUSTODY

The primary objective of the chain-of-custody procedures is to provide an accurate written or computerized record that can be used to trace the possession and handling of a sample from collection to completion of all required analyses. A sample is in custody if it is:

- In someone's physical possession;
- In someone's view;
- Locked up; or
- Kept in a secured area that is restricted to authorized personnel.

9.1.1 Field Custody Procedures

- As few persons as possible should handle samples.
- Sample containers must be obtained precleaned from the laboratory performing the analyses. Coolers or boxes containing cleaned sample containers should be sealed with a custody tape seal during transport to the field or while in storage prior to use.
- The sample collector is personally responsible for the care and custody of samples collected until they are transferred to another person or dispatched properly under chain-of-custody rules.
- The sample collector will record sample data in the field notebook.
- The site team leader will determine whether proper custody procedures were followed during the fieldwork and decide if additional samples are required.

9.1.2 Sample Shipping Log Sheet

For each sampling event at the Monterey Apartments site a shipping log will be prepared. The sample shipping log sheets will be used to verify receipt of a complete data package from the laboratory and provide data reviewers necessary information identifying field blanks, duplicates, etc. They are part of the evidentiary record and must be completed by the field personnel.

9.1.3 Sample Tags

Sample tags attached to or affixed around the sample container will be used to properly identify all samples collected in the field. The sample tags are to be placed on the containers so as not to obscure any QA/QC identifier numbers on the containers or other pertinent information. Sample information must be printed in a legible manner using waterproof ink.

Field identification must be sufficient to enable cross-reference with the logbook. For chain-of-custody purposes, all QC samples are subject to exactly the same custodial procedures and documentation as "real" samples.

9.1.4 Chain-of-Custody Record

The chain-of-custody record will be fully completed at least in duplicate by the field technician who has been designated by the site manager as responsible for sample shipment to the appropriate laboratory for analysis. In addition, for samples known to require rapid turnaround in the laboratory, the person completing the chain-of-custody record will note these constraints in the "Remarks" section of the custody record.

9.1.5 Transfer of Custody and Shipment

- The coolers or packages in which the samples are packed must be accompanied by a chain-of-custody record and other required report forms. When transferring samples, the individuals relinquishing and receiving them must sign, date, and note the time on the chain-of-custody record. This record documents sample custody transfer.
- Shipping containers must be secured with custody seals for delivery to the laboratory. The method of shipment, name of courier, and other pertinent information are entered in the "Remarks" section of the chain-of-custody record.

9.2 DOCUMENTATION

9.2.1 Sample Identification

Each sample will be labeled and sealed immediately after collection. The sample tag will be filled out using waterproof ink and will be firmly affixed to the sample containers. The sample tag will give the following information:

- Name of sampler,
- Date and time of collection,
- Sample number,
- Analysis required,
- Preservation.

9.2.2 Daily Logs

Daily logs and data forms are necessary to provide sufficient data and observations to enable participants to reconstruct events that occurred during the project and to refresh the memory of the field personnel if called upon to give testimony during legal proceedings. All daily logs will be kept in a bound waterproof notebook containing numbered pages. All entries will be made in waterproof ink, dated, and signed. No pages will be removed for any reason. The daily logs will include:

- Names of team members on-site.
- Levels of personnel protection:
  - Level of protection originally used;
  - Changes in protection, if required; and
  - Reasons for changes.
- Time spent collecting samples.
- Documentation on samples taken, including:
  - Sampling location and depth station numbers;
  - Sampling date and time, sampling personnel;
  - Type of sample (grab, composite, etc.); and
  - Sample matrix.
- On-site measurement data.
- Field observations and remarks.
- Weather conditions, wind direction, etc.
- Unusual circumstances or difficulties.
- Initials of person recording the information.

9.2.3 Corrections to Documentation

Notebook. As with any data logbooks, no pages will be removed for any reason. If corrections are necessary, these must be made by drawing a single line through the original entry (so that the original entry can still be read) and writing the corrected entry alongside. The correction must be initialed and dated. Most corrected errors will require a footnote explaining the correction.

Sampling Forms. As previously stated, all sample identification tags, chain-of-custody records, and other forms must be written in waterproof ink. None of these documents are to be destroyed or thrown away, even if they are illegible or contain inaccuracies that require a replacement document.

If an error is made on a document assigned to one individual, that individual may make corrections simply by crossing a line through the error and entering the corrected information. The incorrect information should not be obliterated. Any subsequent error discovered on a document should be corrected by the person who made the entry. All corrections must be initialed and dated.

9.2.4 Photographs

Photographs will be taken as directed by the team leader. Documentation of a photograph is crucial to its validity as a representation of an existing situation. The following information will be noted in the task log concerning photographs:

- Date, time, location photograph was taken;
- Photographer (signature);
- Weather conditions;
- Description of photograph taken;
- Reasons why photograph was taken;
- Sequential number of the photograph and the film roll number; and
- Camera lens system used.

9.3 SAMPLE HANDLING, PACKAGING, AND SHIPPING

The transportation and handling of samples must be accomplished in a manner that not only protects the integrity of the sample, but also prevents any detrimental effects due to the possible hazardous nature of samples. Regulations for packaging, marking, labeling, and shipping hazardous materials are promulgated by the United States Department of Transportation (DOT) in the Code of Federal Regulations, 49 CFR 171 through 177.

9.3.1 Sample Packaging

Samples must be packaged carefully to avoid breakage or contamination and must be shipped to the laboratory at proper temperatures. The following sample package requirements will be followed:

- The Suma canisters will be labeled using detachable tags as per Manchester Laboratory's instructions.
- The canisters will be stored in coolers to protect their integrity during handling, transport, or shipment.
- Any remaining space in the cooler should be filled with inert packing material. Under no circumstances should material such as sawdust, sand, etc., be used.
- Two copies of the custody record must be placed in a plastic bag and accompany the samples.

9.3.2 Shipping Containers

Delivery of the Suma canisters is anticipated to be direct transfer by E & E to the Manchester Laboratory. If sent by courier, shipping containers are to be custody-sealed for shipment or transport as appropriate. Field personnel will make arrangements for transportation of samples to the laboratory. When custody is relinquished to a shipper, field personnel will telephone the laboratory sample custodian, to inform him/her of the expected time of arrival of the sample shipment and to advise him/her of any time constraints on sample analysis.

10. PERFORMANCE AND SYSTEMS AUDITS

No E & E performance or systems audits are planned for the pilot study.

11. PREVENTIVE MAINTENANCE

All field instruments and equipment used for analysis will be serviced and maintained only by qualified personnel. All instruments are maintained by senior staff and/or electronic technicians. All repairs, adjustments, and calibrations will be documented in an appropriate logbook or data sheet that will be kept on file. The instrument maintenance logbooks will clearly document the date, the description of the problems, the corrective action taken, the result, and who performed the work.

All equipment used by E & E in the field is subject to standard preventive maintenance schedules established by corporate equipment protocols. When in use, equipment is inspected at least twice daily, once before start-up in the morning and again at the end of the work shift prior to overnight storage or return to the charging rack. Regular maintenance such as cleaning lenses, replacement of in-line filters, and removal of accumulated dust is to be conducted according to manufacturer's recommendations and in-field need, whichever is appropriate. All preventive maintenance performed will be entered in the individual equipment's logbook.

In addition to preventive maintenance procedures, daily calibration checks will be performed at least once a day in the morning prior to use and duly recorded in the respective logbooks. Additional calibration checks will be performed as required. All logbooks will become part of either the permanent site file or the permanent equipment file.

12. CORRECTIVE ACTIONS

Corrective actions for the Monterey Apartments Soil-Gas Pilot Study will be recorded and appended to this document using the **CORRECTIVE ACTION CHECKLIST** found in Appendix A.

Appendix A

CHECKLISTS

- Sample Alteration Checklist
- Corrective Action Checklist

SAMPLE ALTERATION CHECKLIST

Project Name and Number:

Material to be Sampled:

Measurement Parameters:

Standard Procedure for Field Collection and Laboratory Analysis:
(cite references)

Reason for Change in Field Procedure or Analytical Variation:

Special Equipment, Material, or Personnel Required:

Initiator's Name: ______________________ Date: __________
Project Approval: ______________________ Date: __________
Laboratory Approval: ______________________ Date: __________
QA Officer/Reviewer: ______________________ Date: __________
Sample Control Center: ______________________ Date: __________

CORRECTIVE ACTION CHECKLIST

Project Name and Number:

Sample Dates Involved:

Measurement Parameters:

Acceptable Data Range:

Problem Area Requiring Corrective Action:

Measures Required to Correct Problem:

Means of Detecting Problems and Verifying Correction:

Initiator's Name: ______________________ Date: __________

Project Approval: ______________________ Date: __________

Laboratory Approval: ______________________ Date: __________

QA Officer/Reviewer: ______________________ Date: __________

Sample Control Center: ______________________ Date: __________

Appendix B

LABORATORY DELIVERABLE REQUIREMENTS AND MANCHESTER MODIFIED METHOD TO-14 ANALYTICAL SOP

LABORATORY DELIVERABLE REQUIREMENTS

Laboratory Deliverable Requirements:

- A statement of the analytical method used, and documentation of any deviation from the method, sample preparation, detection and/or quantitation limits, GC columns used, GC conditions;
- A statement of sample holding times;
- Analytical sequence, including times and dates;
- Internal standard areas for all analyses;
- Tuning data and summary sheets;
- Initial and continuing calibration summary sheets, including relative response factors, percent relative standard deviations, and percent difference values;
- Surrogate recovery calculations for each analysis;
- Data summary sheets for all samples analyzed including up to 20 hydrocarbon and aromatic TICs;
- Data summary sheets for all blank and duplicate analyses including up to 20 hydrocarbon and aromatic TICs;
- Copies of instrument had copy pertaining to samples, blanks, duplicated, standards and calibrations;
- A statement of volumes, dilutions, etc. used in analysis.

The laboratory will keep all raw data generated during analysis on file for at least 1 year, allowing E & E to purchase the data at a later date if necessary.

STANDARD OPERATING PROCEDURE FOR THE ANALYSIS OF AMBIENT AIR VOLATILE ORGANIC COMPOUNDS (VOCs) IN CANISTERS BY GC/MS ANALYSIS

Revision 3.0

DRAFT

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STANDARD OPERATING PROCEDURE FOR THE ANALYSIS OF AMBIENT AIR VOLATILE ORGANIC COMPOUNDS (VOCs) IN CANISTERS BY GC/MS ANALYSIS

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STANDARD OPERATING PROCEDURE FOR THE ANALYSIS OF AMBIENT AIR VOLATILE ORGANIC COMPOUNDS (VOCs) IN CANISTERS BY GC/MS ANALYSIS.

. Scope

1.1 This document describes a procedure for analysis of volatile organic compounds (VOCs) in ambient air. The method is based on collection of whole air samples in passivated stainless steel canisters at less than ambient pressures. The VOCs are subsequently separated by gas chromatography and measured by mass spectrometry in the SCAN mode.

1.2 This method is applicable to specific VOCs listed in <u>Method TO-14, The Determination of Volatile Organic Compounds (VOCs) in Ambient Air Using Summa Passivated Canister Sampling and Gas Chromatographic Analysis,</u> that have been tested and determined to be stable when stored in pressurized and subatmospheric pressure canisters. Numerous compounds, many of which are chlorinated VOCs, have been successfully tested for storage stability in pressurized canisters. This method also includes other chlorinated and/or aromatic VOCs that are listed in Method 524.2, Volatile_Organic_Compound_Analysis_in_Water by_Purge_and_Trap_Capillary_Column_GC/MS. However, minimal documentation is currently available demonstrating stability of VOCs in subatmospheric pressure canisters.

1.3 The organic compounds to be analyzed are listed in Table 1. They are determined at the parts per billion (mole/mole) level.

. Summary of Method

2.1 An ambient air sample is collected in a pre-cleaned stainless steel canister and is submitted to the lab for analysis. It is connected to the pressurization apparatus and the initial pressure (in torr) is measured. The canister is then pressurized with humidified prepurified N2 to a pressure of about 1000 torr.

2.2 The canister is next connected to the canister analysis system. The canister is sampled at a fixed rate as determined by a mass flow controller for a desired period of time; the sample passes through a trap designed to capture organic compounds in the purge and trap instrument.
After collection, the sample is desorbed onto the GC column where it is cryofocused with a dewar flask of liquid nitrogen. When the dewar is removed, the GC program and MS acquisition are simultaneously started to analyze the sample.

2.3 The sample is analyzed in the SCAN mode to use the MS as a universal detector to search for other target and tentative compounds. The target compounds are positively identified by their retention times and their mass spectral ions. The target VOCs are quantified by standardization from a standard canister prepared by a static dilution bottle.

. Apparatus

3.1 Canister Sampling System

3.1.1 Mass flow controller (1); maintain constant flow of gas from a canister (Tylan model FC-260, 0-100SCCM, Tylan Corp., Torrance, CA).

3.1.2 Mass flow controller readout box (Tylan model RO-32).

3.1.3 Six-port stainless steel valve, medium temperature, 1/16 in. tubing, with 6 in. stand-off assembly (Valco Instruments Co., Houston, TX).

3.1.4 Controlled heating assembly for the 6-port valve to warm the valve and prevent condensation of organic compounds (medium temperature, 110v, Valco Instruments Co.).

3.1.5 Transfer line heater to warm the stainless steel (ss) 1/16 in tubing to prevent VOC condensation (Tekmar Co., Cincinnati, OH). [NOTE: In lieu of using ss tubing as the sample line, success has be reported using Nickel tubing with no heating assmebly. Nickel tubing can be obtained from Alltech Associates Inc./ Applied Science Labs, Deerfield, IL.]

3.1.6 Vacuum pump; general purpose, used to draw the sample through the trap (Thomas Industries Inc., Sheboygan, WI, model 107CA18, or equivalent).

3.2 GC/MS System

3.2.1 Purge and trap instrument; containing the specialized trap (see 3.2.2), the transfer line interfaces directly to the GC column through a special GC injector fitting (Tekmar Co., Cincinnati, OH, model LSC-3, or equivalent).

3.2.2 Carbopack B/Carbosieve S-III trap; installed in the purge and trap to capture the VOCs (Supelco Co., Bellefont, PA, catalogue no. 2-0321).

3.2.3 Finnigan 5100 GC/MS; capable of either scanning continuously the mass range 35-300 amu in about 1 second, and equiped with a NOVA 4 computer with INCOS software, version 6.5, and a 96 mB CDC disk drive. The GC oven and all fine tuning functions are controlled through the keyboard (Finnigan MAT, San Jose, CA).

3.3 Canister Cleaning System; turn-key, bench top manifold system (Scientifi Instrumentation Specialists, Moscow, ID, model GS-ERS-A2/4).

3.4 Pressurization/Standardization Apparatus

3.4.1 Pressure transducer; capable of reading 0-1000 torr (MKS Instruments, Andover, MA, model 122AA-01000AB).

3.4.2 Pressure transducer readout box (MKS Instruments, model PDR-D-1, o equivalent).

3.4.3 Cajon Ultra-torr union, 1/2 in. OD tubing (SS-8-UT-6); Whitey ball valves(2), 1/4 in. (SS-42S4); stainless steel (ss) union tees (400-3 and 100-3); ss union (200-6); ss reducing union (810-6-4); ss reducing unions (2# 200-6-1, and 1# 400-6-2); all of the above are used to assemble the entire apparatus (Swagelok Co., Bellevue WA).

3.4.4 Stainless steel (or nickel) tubing ranging in OD from 1/16 in. to 1/2 in., varying lengths (Alltech Associates /Applied Science).

3.4.5 Gas sampling syringe, 0-2.0 ml (Supelco Co., catalogue number 2-2265, or equivalent).

3.4.6 1/16 in. TFE tubing.

3.4.7 Glas-col heating mantles, top and bottom, 2 liter size.

3.4.8 Powerstat (Superior Electric, Bristol, CT, or equivalent).

3.4.9 Thermolyne magnetic stirrer.

3.4.10 2 liter round bottom flask with 24/40 joint; 24/40 TFE stopper; mininert valve, 1/4-28 male thread.[NOTE: you may substitute a static dilution bottle, catalogue number 14-2080-000 and its accessories, Tekmar Co.).

4. Reagents and Materials

4.1 Gas cylinders of helium and nitrogen, ultrahigh purity grade, or zero grade air, best source.

4.2 Static dilution bottle standards, liquid standards with purity of 98+% best source:

Chloroform
1,2-Dichloroethane
Benzene
Toluene
Dichloromethane
1,1-Dichloroethane
cis-1,2-Dichloroethene
trans-1,2-Dichloroethene
1,1-Dichloroethene
1,2-Dichloropropane
1,1,2-Trichloroethane
1,2-Dibromomethane
Tetrachloroethene
cis-1,3-Dichloropropane
trans-1,3-Dichloropropane
Ethylbenzene
o-Xylene
m-Xylene
p-Xylene
Freon 11
Freon 113
Styrene
1,1,2,2-Tetrachloroethane
1,3,5-Trimethylbenzene
1,2,4-Trimethylbenzene
1,2-Dichlorobenzene
1,3-Dichlorobenzene
Benzyl Chloride
1,2,4-Trichlorobenzene
Bromodichloroethane
Dibromochloromethane
Bromoform
Dibromomethane
1,1,1,2-Tetrachloroethane
1,3-Dichloropropane
Bromobenzene
Butylbenzene
1,2,3-Trichloropropane
Isopropyltoluene
Propylbenzene
Isopropylbenzene
Freon 114

4.3 Purged standards, EPA Repository, Las Vegas, or best source.
4.4 Cryogen; liquid nitrogen (b.p. -195.8oC), best source.
4.5 Deionized water, Milli-Q system or equivalent.
4.6 4-Bromofluorobenzene, for tuning the GC/MS, best source.
4.7 Methanol, Burdick and Jackson Purge and Trap Grade, or equivalent.

5. Standard Canister Preparation

5.1 Static Dilution Bottle Preparation

5.1.1 The basic theory and operation for the preparation of a static dilution bottle (SDB) has been described by Berkley, et.al. (1). This method advocates the use of a multiple mix standard for ease and efficiency of operation. An advantage of the SDB is that a variety of VOCs can be target compounds; all that is needed is a source of the neat compound with a purity of 98+%. If the study of VOCs other than those available in USEPA audit cylinders is desired, positive identification and reasonable quantitation of those compounds is obtainable. The main disadvantage is that the components in the SDB standard must be in liquid form and be able to be measured and added by a microsyringe. For this reason, volatile gases (chloromethane, bromomethane, vinyl chloride, chloroethane, and Freon 12), or other compounds that are solid at room temperature, cannot be used. They can be part of the target compound list and positively identified by retention time and spectra, but their concentration can only be estimated.

5.1.2 The compounds listed in 4.2 are added to a 20ml VOA bottle fitted with a screw-top lid with a mininert valve. The density of each compound is noted when determining what volume is added to the standard mixture; enough of the compound is added so that approximately 500+ 10 mg of a compound is added to the mix. The sum of al individual component volumes is used to determine the concentratio of each in the mix.

5.1.3 Place a 2 l round bottom flask, whose exact volume has been determined, in the heating mantles; several glass beads and a magnetic stirring bar are added to the flask. A 24/40 teflon stopper that has been fitted with a mininert valve is snugly place in the flask. The temperature of the SDB is controlled by a powerstat; set the powerstat to a predetermined setting that will keep the SDB at a desired temperature. Add 90 ul of the mixed standard, and allow it to equilibrate for several hours at 100 C.

5.1.4 After equilibration, connect a cleaned, evacuated canister to the system illustrated in Figure 1. Before tightening the connection o the canister valve, flush the system with N2 or zero grade air for a few minutes. Loosen the septum nut on the injection tee, and the tighten the canister connection. Stop the N2 flow from the tank an tighten the septum nut. Close the system valve, open the transduce valve, and then open the canister valve. Open the system valve, an add humidified N2 to the canister at a slow and steady rate. When the canister pressure is about 300 torr, take the 2.0ml gas syring that has been warmed to about 100oC from the oven, and insert the needle through the mininert valve into the SDB. Fill and expel the syringe 4 or 5 times in the SDB, and then take a 1.14ml aliquot of the SDB standard gas. Close the transducer valve on the system and slowly inject (over 10 seconds) the gas into the system through the septum in the tee connection. With the needle still in the septum, slowly fill and expel the gas syringe to flush traces of compounds out of the syringe and into the system. Remove the syringe and open the transducer valve.

5.1.5 Continue adding N2 to the canister until the transducer reads 1005 torr; close the system valve and let the pressure between the canister and transducer equilibrate. Note the final pressure and close the canister valve. Shut off the N2 tank regulator and remov the canister from the system.

5.2 Purged Standard Preparation

5.2.1 Many of the limitations of the static dilution bottle can be eliminated by using the Purged Standard Technique for preparing standard canisters. The limitation of no gaseous or solid compounds no longer exists, and all of the compounds listed in EPA Method 524.2 with the addition of benzyl chloride, freon 113, and freon 114 can be positively identified and quantitated. Mixes or neat solutions of the compounds in methanol of known concentrations can be prepared or obtained from commercial or government sources.

5.2.2 When first filling an evacuated canister with nitrogen or zero air the teflon tubing of the incoming gas stream will be below the surface of the water. Add 0.5 ml or less of deionized water to the humidifier to prevent aspiration of water drops into the gas lines or canister. Connect a cleaned, evacuated canister to the system illustrated in Figure 1; since a gaseous mix will not be added to the nitrogen or air stream entering the canister, the union tee with injection septum can be removed from the apparatus. Before tightening the connection on the canister valve, flush the system with nitrogen or zero grade air for a few minutes. Close the syste valve, and then tighten the canister connection.

5.2.3 Open the system valve, the transducer valve, and the canister valve. Add humidified nitrogen to the canister at a steady, but not too rapid rate. When the pressure is about 600 torr, close the system valve and transducer valve and stop the nitrogen flow. Add 5 ml of water to the humidifier tube that contains the target compounds and internal and surrogate standards in concentrations that will give desired concentrations in the canister. Re-connect the tube, open the system valve, and turn the nitrogen flow back on. Purge for 10 minutes with the transducer valve closed; then open the transducer valve and continue to add nitrogen until the transducer reads 1005 torr.

5.2.4 Close the system valve and let the pressure between the canister and transducer equilibrate. Note the final pressure and close the canister valve. Shut off the nitrogen tank regulator if no other canisters are to be prepared, and remove the canister from the system.

6. Analytical System (Figure 2).

6.1 System Description

6.1.1 The GC/MS system used for analyzing canisters is basically the sam as for analyzing soil or water samples, but with some extra apparatus and steps upstream of the purge and trap instrument. The main difference is the addition of a heated 6-port valve with connections to the sample, purge and vent ports of the purge and trap instrument and to a pump and a mass flow controller in line with the canister. The connections and sample lines are illustrate in Figure 2. These connections permit flow through the trap to be either He gas (regulated by the purge and trap instrument), or canister sample gas. If desired, with this configuration the trap can be dry purged after the canister is sampled. The other difference is the use of a Carbopack B/Carbosieve S-III trap. This trap reportedly is highly retentive for chloromethane (and presumedly for the rest of the VOCs) while allowing water to breakthrough at much lower purge volumes (2). The use of this trap makes the Nafion dryer unnecessary and simplifies the apparatus. I also allows polar compounds to be analyzed.

6.1.2 In the SCAN mode, the GC/MS uses a MID descriptor to scan from masses 35 to 300 with a total scan time of 1.110 seconds. Six scan per eluting chromatographic peak are provided by this rate. The quantitation mass for a specific compound is searched for over a desired narrow region of the chromatogram, and it is quantitated i the CHRO program and identified by a library search.

6.1.3 In the SIM mode, the GC/MS uses a SEQUence descriptor that is a collection of MID descriptors that contains selected masses for specific compounds. The SEQU descriptor turns on and off these MID descriptors at desired times during the chromatographic analysis when the compounds are expected to be eluted. The SIM mode is best used when a limited number of compounds are to be searched for and quantitated. Usually 2 spectral ions for each compound is enough to make a positive identification.

6.2 GC/MS System Performance

6.2.1 Daily GC/MS Tuning.

6.2.1.1 At the beginning of each day or prior to a standardization the GC/MS system must be tuned to verify that acceptable spectra can be achieved.

6.2.1.2 After initial tuning of the GC/MS with FC43, 4-Bromofluorobenzene (BFB) is introduced by purging 50ng mixed with 5ml of H2O in a purge vessel. The key ions and ion abundance criteria that must be met are listed in Tabl 3.

6.2.1.3 The performance criteria must be achieved before any samples, blanks or standards are analyzed. If any key ion abundance observed for the daily BFB mass tuning check doe not meet the specifications, the instrument must be retune or the sample and/or standardization gases reanalyzed unti the above condition is met.

6.2.2 GC/MS Standardization

6.2.2.1 Initial Standardization. Perform a multipoint humid static standardization using 3 canisters (plus zero humid air) that are each filled at a different concentration between 0.04-0.20 ug/l (1-20 ppb) from different amounts of gas from the SDB, or the standards in the purged humidifer tube. The expected retention and mass spectra abundance ar used to verify proper operation of the GC/MS system. A response factor (RF) is determined for each compound (usin either the external calibration method for the static dilution bottle or the internal calibration method using the purged humidifier tube) at each level and written to a response list with the proper subcommand in the QUAN program of INCOS. After all 3 of them are collected, the average RF, its standard deviation (   ) and the percent relative standard deviation is computed.

6.2.2.2 Routine Standardization. Use the mid-range standard canister to perform the daily standardization. After quantitating the standard and updating its library, append the RF to the response list with the appropriate subcomman in QUAN. Compare the new RF with the initial standardization; if the daily RF is not within + 30 % of the initial RF average for at least 6 of the CCC compounds, re-run the standard. After the single point standardization, run a canister with humidified zero air to ensure the analytical system has less than 0.2 ppb of the VOCs.

6.3 Analytical Procedures

6.3.1 Canister Receipt

6.3.1.1 Observe the overall condition of each sample canister. Eac canister should be received with an attached sample ID tag

6.3.1.2 Record each canister in the dedicated lab logbook. Also note on the ID tag the date received and initials of recipient.

6.3.1.3 Attach the canister to the pressurization apparatus. Befor tightening the connection to the canister valve, flush the system lines with prepurified N2 for a few minutes. Loosen the septum nut on the injection tee (if present) and tighten the canister valve connection. Turn off the N2 flo while tightening the septum nut. With the system valve closed, open the canister valve and the transducer valve and note the pressure of the canister in the logbook. Turn on the nitrogen flow at the tank regulator and open the system valve; adjust the nitrogen flow so the dimple in th water (or the bubbling of the gas through the water in the teflon tubing is immersed) from the gas flow is easily noticeable. Pressurize the canister to 1005 torr and then close the system valve. After the reading equilibrates, record this reading in the sample logbook. The dilution factor (DF) is also recorded:

$$DF = Ya/Xa$$

where

Xa = canister pressure (absolute) before dilution.

Ya = canister pressure (absolute) after dilution.

6.3.1.4 When internal and surrogate standards are to be added to the canister, the humidifier tube should be filled with 5 ml of the standards in the amounts necessary to yield th desired canister concentration after the initial canister pressure is noted but before it is pressurized.

6.3.2 GC/MS-SCAN Analysis

6.3.2.1 The analytical system should be properly assembled, humid zero air certified (to assure there is no system contamination), operated and standardized for accurate VOC determination.

6.3.2.2 The mass flow controller should be checked and adjusted to provide a correct flow rate to the purge and trap.

6.3.2.3 Connect the canister to the mass flow controller (MFC) inlet side; set the MFC flow rate to 40 cm3/min.

6.3.2.4 Start the pump and MFC, and open the canister valve. Start the purge and trap flow and flush the lines for about 1 minute. After flushing, turn the 6-port valve to direct th canister flow onto the trap and start a stopwatch. Collect about 500 cm3 of canister gas.

6.3.2.5 After the appropriate sampling time, turn the 6-port valve back to its original position and note the mass flow rate and sampling time in the sample log book. (If needed, the trap can be dry purged at this time for up to 10 minutes.)

6.3.2.6 Place a dewar flask filled with liquid N2 (LN2) into the G oven. Place a loop from the front end of the GC column end into the dewar. Switch the purge and trap to desorb and desorb the trap for 4 minutes at 220 C.

6.3.2.7 After 4 minutes, initiate the data collection and GC temperature program, switch the purge and trap instrument to trap bake, remove the dewar from the oven, and shut the oven door. Bake the trap for 7 minutes at 260 C.

6.3.2.8 The GC temperature program starts at 10 C and is held ther for 2 minutes. It is then programmed from 10 C to 102 C at a rate of 8 C/minute, and then programmed to 180 C at 60 C/minute. It holds the final temperature for 5.3 minutes. The total temperature program time is 20 minutes.

6.3.2.9 The MS scans from 35 to 300 amu in a total scan time of 1.110 seconds. This corresponds to about 6 scans per eluting chromatographic peak.

6.3.2.10 Primary identification is based upon retention time and relative abundance of eluting ions as compared to the spectral library stored on the disk of the GC/MS computer.

6.3.2.11 After acquisition, the VOCs are automatically searched and if present, the amounts are determined by the computer by using the area of the target compound base peak, and the area of the appropriate internal standard, if the internal standard method of quantitation is used.

6.3.2.12 The computer program calculates the amount (in ng) of each VOC present and lists them in the quantitation report. The results are most easily reported by the computer in nanograms or micrograms per liter. To convert them to part per billion (ppb):

$$ppb = \frac{\frac{NG}{GMW}}{\frac{L}{24.4}} \quad x \quad DF$$

where:
NG = ng of compound found, on column.
GMW = gram molecular weight of the compound, in gm.
L = liters sampled from the canister.
DF = dilution factor, from pressurizing the canister.
24.4 = gram molecular volume, in liters at 25 C.

This final calculation is done manually on the final repor sheet.

6.3.3 GC/MS-SIM Analysis

6.3.3.1 Steps 6.3.2.1 through 6.3.2.8 are also followed for the SIM analysis.

6.3.3.2 The mass spectrometer will scan for specific ions at specified elution times of the chromatographic run using MID descriptors listing at least two ions of the compound being sought. A SEQU descriptor lists the appropriate MID descriptors and turns them on and off at the proper times.

6.3.3.3 Primary identification is based upon retention time and presence of the two or more spectral ions of the target compound in the correct ratio and their simultaneous elution.

6.3.3.4 After acquisition, the VOCs are automatically searched anc if present, the amounts are determined by the computer by using the area of the target compound base peak and the area of the appropriate internal standard, if the internal standard method of quantitation is used.

6.3.3.5 The computer program calculates the amount (in ng) of each

VOC present and lists them in the quantitation report. The target compouds can be easily reported in ng or ug per liter, or they can be reported in ppb by the formula in section 6.3.1.12.

7. Canister Cleaning and Certification

7.1 All canisters must be clean and free of any contaminants before sample collection.
7.2 Leak test the canisters by pressurizing them to about 30 psig with zero air or prepurified N2. If leak tight, the pressure should not vary more than + 2 psig over a 24 hour period.
7.3 The canisters are cleaned using the model GS-ERS-A2/4 manifold system available through Scientific Instrumentation Specialists (Figure 3). Open the pump and canister valves, evacuate the canisters and let the pump valve remain open to the system for at least a minute after the manifold gauge reads maximum system vacuum. Close the pump valve and bleed humidified, prepurified N2 or zero air into the manifold through the vent valve. Close the vent valve when the system is at ambient pressure. Repeat the process 4 more times.
7.4 After the canister comes to ambient pressure, analyze its contents by GC/MS-SCAN to verify that they contain less than 0.2 ppb of any VOC.
7.5 If the canisters are clean, evacuate them a final time and prepare them for use in the field.
7.6 If they are not clean, repeat steps 7.3 and 7.4.
7.7 If they are still not clean, evacuate the canisters and leave them attached to the vacuum pump for 24 hours before testing for cleanliness again.

8. Performance Criteria and Quality Assurance

8.1 Relative Accuracy. Accuracy can be determined by analyzing a canister that has been filled with components from an NBS-SRM or EPA-CRM audit cylinder. The percent relative accuracy is

$$\text{Percent Relative Accuracy} = \frac{X - Y}{X} \ (100\%)$$

where

X = concentration of VOC in audit cylinder
Y = concentration of VOC determined by this method

The relative accuracy should fall between 75 and 125 percent.

8.2 Quality Assurance

8.2.1 The analytical system is verified to be clean by analyzing clean canisters filled with humid prepurified N2[∂r0zero air and the target VOC content is less than 0.2 ppb.
8.2.2 They system meets the daily BFB tuning requirements.
8.2.3 The initial multipoint average RF percent relative standard deviation should be 30% or less, and the daily RF of the VOCs should be within 30% of the initial average RF.
8.2.4 Each day a canister filled with a known concentration of Group V compounds from an EPA Perfromance Audit Cylinder is analyzed to verify the accuracy of the daily standard.
8.2.5 If any of the target VOCs are beyond the highest initial standard

level, then the canister is analyzed again with an appropriately lower sample volume taken from the canister.

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TABLE 1. VOLATILE ORGANIC COMPOUND DATA SHEET

COMPOUND (SYNONYM)	FORMULA	MOLECULAR WEIGHT	BOILING POINT (°C)	MELTING POINT (°C)	CAS NUMBER
Freon 12 (Dichlorodifluoromethane)	Cl_2CF_2	120.91	-29.8	-158.0	75-71-8
Methyl chloride (Chloromethane)	CH_3Cl	50.49	-24.2	-97.1	74-87-3
Freon 114 (1,2-Dichloro-1,1,2,2-tetrafluoroethane)	$ClCF_2CClF_2$	170.93	4.1	-94.0	76-14-2
Vinyl chloride (Chloroethylene)	$CH_2{=}CHCl$	62.50	-13.4	-1538.0	75-01-4
Methyl bromide (Bromomethane)	CH_3Br	94.94	3.6	-93.6	74-83-9
Ethyl chloride (Chloroethane)	CH_3CH_2Cl	64.52	12.3	-136.4	75-00-3
Freon 11 (Trichlorofluoromethane)	CCl_3F	137.38	23.7	-111.0	75-69-4
Vinylidene chloride (1,1-Dichloroethene)	$C_2H_2Cl_2$	96.95	31.7	-122.5	75-35-4
Dichloromethane (Methylene chloride)	CH_2Cl_2	84.94	39.8	-95.1	75-09-2
Freon 113 (1,1,2-Trichloro-1,2,2-trifluoroethane)	CF_2ClCCl_2F	187.38	47.7	-36.4	76-13-1
1,1-Dichloroethane (Ethylidene chloride)	CH_3CHCl_2	98.96	57.3	-97.0	74-34-3
cis-1,2-Dichloroethylene	$CHCl{=}CHCl$	96.94	60.3	-80.5	156-59-2
Chloroform (Trichloromethane)	$CHCl_3$	119.38	61.7	-63.5	67-66-3
1,2-Dichloroethane (Ethylene dichloride)	$ClCH_2CH_2Cl$	98.96	83.5	-35.3	107-06-2
Methyl chloroform (1,1,1-Trichloroethane)	CH_3CCl_3	133.41	74.1	-30.4	71-55-6
Benzene (Cyclohexatriene)	C_6H_6	78.12	80.1	5.5	71-43-2
Carbon tetrachloride (Tetrachloromethane)	CCl_4	153.82	76.5	-23.0	56-23-5
1,2-Dichloropropane (Propylene dichloride)	$CH_3CHClCH_2Cl$	112.99	96.4	-100.4	78-87-5
Trichloroethylene (Trichloroethene)	$ClCH{=}CCl_2$	131.29	87	-73.0	79-01-6
cis-1,3-Dichloropropene (cis-1,3-dichloropropylene)	$CH_3CCl{=}CHCl$	110.97	76		10061-01-5

TABLE 1. VOLATILE ORGANIC COMPOUND DATA SHEET (cont.)

COMPOUND (SYNONYM)	FORMULA	MOLECULAR WEIGHT	BOILING POINT(°C)	MELTING POINT (°C)	CAS NUMBER
trans-1,3-Dichloropropene (cis-1,3-Dichloropropylene)	$ClCH_2CH{=}CHCl$	110.97	112.0		10061-02-6
1,1,2-Trichloroethane (Vinyl trichloride)	$CH_2ClCHCl_2$	133.41	113.8	-36.5	79-00-5
Toluene (Methyl benzene)	$C_6H_5CH_3$	92.15	110.6	-95.0	108-88-3
1,2-Dibromoethane (Ethylene dibromide)	$BrCH_2CH_2Br$	187.88	131.3	9.8	106-93-4
Tetrachloroethylene (Perchloroethylene)	$Cl_2C{=}CCl_2$	165.83	121.1	-19.0	127-18-4
Chlorobenzene (Phenyl chloride)	C_6H_5Cl	112.56	132.0	-45.6	108-90-7
Ethylbenzene	$C_6H_5C_2H_5$	106.17	136.2	-95.0	100-41-4
m-Xylene (1,3-Dimethylbenzene)	1,3-$(CH_3)_2C_6H_4$	106.17	139.1	-47.9	108-38-3
p-Xylene (1,4-Dimethylxylene)	1,4-$(CH_3)_2C_6H_4$	106.17	138.3	13.3	106-42-3
Styrene (Vinyl benzene)	$C_6H_5CH{=}CH_2$	104.16	145.2	-30.6	100-42-5
1,1,2,2-Tetrachloroethane	$CHCl_2CHCl_2$	167.85	146.2	-36.0	79-34-5
o-Xylene (1,2-Dimethylbenzene)	1,2-$(CH_3)_2C_6H_4$	106.17	144.4	-25.2	95-47-6
1,3,5-Trimethylbenzene (Mesitylene)	1,3,5-$(CH_3)_3C_6H_6$	120.20	164.7	-44.7	108-67-8
1,2,4-Trimethylbenzene (Pseudocumene)	1,2,4-$(CH_3)_3C_6H_6$	120.20	169.3	-43.8	95-63-6
m-Dichlorobenzene (1,3-Dichlorobenzene)	1,3-$Cl_2C_6H_4$	147.01	173.0	-24.7	541-73-1
Benzyl chloride (α-Chlorotoluene)	$C_6H_5CH_2Cl$	126.59	179.3	-39.0	100-44-7
o-Dichlorobenzene (1,2-Dichlorobenzene)	1,2-$Cl_2C_6H_4$	147.01	180.5	-17.0	95-50-1
p-Dichlorobenzene (1,4-Dichlorobenzene)	1,4-$Cl_2C_6H_4$	147.01	174.0	53.1	106-46-7
1,2,4-Trichlorobenzene	1,2,4-$Cl_3C_6H_3$	181.45	213.5	17.0	120-82-1
Hexachlorobutadiene (1,1,2,3,4,4-Hexachloro-1,3-butadiene)		260.76			87-68-3

TABLE 1. VOLATILE ORGANIC COMPOUND DATA SHEET

COMPOUND (SYNONYM)	FORMULA	MOLECULAR WEIGHT	BOILING POINT (°C)	MELTING POINT(°C)	CAS NUMBER
trans-1,2-Dichloroethene	$C_2H_2Cl_2$	96.94	48°	-50°	156-60-5
Dibromomethane	CH_2Br_2	173.94	96-98°	-52°	74-95-3
Bromodichloromethane	$BrCl_2CH$	163.83	87°	-55°	75-27-4
1,3-Dichloropropane	$C_3H_6Cl_2$	112.99	120-122°	-99°	142-28-9
Dibromochloromethane	Br_2ClCH	208.29	119-120°	-22°	124-48-1
1,1,1,2-Tetrachloroethane	$C_2H_2Cl_4$	167.85	138°	(< 0°)	630-20-6
Bromoform	$CHBr_3$	252.75	150-151°	8.3°	75-25-2
Isopropylbenzene (cumene)	$C_6H_5C_3H_7$	120.20	152-154°	-96°	98-82-8
Bromobenzene	C_6H_5Br	157.02	156°	-31°	108-86-1
1,2,3-Trichloropropane	$C_3H_5Cl_3$	147.43	156°	-14°	96-18-4
Propylbenzene	$C_6H_5C_3H_7$	120.20	159°	-99°	103-65-1
p-Isopropyltoluene (p-cumene)	$CH_3C_6H_4C_3H_7$	134.22	176-178°	-68°	99-87-6
Butylbenzene	$C_6H_5C_4H_9$	134.22	183°	-88°	104-51-8

TABLE 2. ION/ABUNDANCE AND EXPECTED RETENTION TIME FOR SELECTED VOCs ANALYZED BY GC-MS-SIM

Compound	Ion/Abundance (amu/% base peak)	Expected Retentio Time (min)
Freon 12 (Dichlorodifluoromethane)	85/100	1:01
	87/ 31	
Methyl chloride (Chloromethane)	50/100	1:14
	52/ 34	
Freon 114 (1,2-Dichloro-1,1,2,2-tetrafluoroethane)	85/100	1:11
	135/ 56	
	87/ 33	
Vinyl chloride (Chloroethene)	62/100	1:24
	27/125	
	64/ 32	
Methyl bromide (Bromomethane)	94/100	1:51
	96/ 85	
Ethyl chloride (Chloroethane)	64/100	1:39
	29/140	
	27/140	
Freon 11 (Trichlorofluoromethane)	101/100	2:27
	103/ 67	
Vinylidene chloride (1,1-Dichloroethylene)	61/100	3:13
	96/ 55	
	63/ 31	
Dichloromethane (Methylene chloride)	49/100	4:02
	84/ 65	
	86/ 45	
Freon 113 (1,1,2-Trichloro-1,2,2-trifluoroethane)	151/100	3:18
	101/140	
	103/ 90	
1,1-Dichloroethane (Ethylidene dichloride)	63/100	5:04
	27/ 64	
	65/ 33	
cis-1,2-Dichloroethylene	61/100	5:56
	96/ 60	
	98/ 44	
Chloroform (Trichloromethane)	83/100	6:28
	85/ 65	
	47/ 35	
1,2-Dichloroethane (Ethylene dichloride)	62/100	7:14
	27/ 70	
	64/ 31	
Methyl chloroform (1,1,1-Trichloroethane)	97/100	6:36
	99/ 64	
	61/ 61	
Benzene (Cyclohexatriene)	78/100	7:10
	77/ 25	
	50/ 35	
Carbon tetrachloride (Tetrachloromethane)	117/100	6:51
	119/ 97	

(continued

TABLE 2. ION/ABUNDANCE AND EXPECTED RETENTION TIME FOR SELECTED VOCs ANALYZED BY GC-MS-SIM (cont.)

Compound	Ion/Abundance (amu/% base peak)	Estimated Retentio Time (min)
1,2-Dichloropropane (Propylene dichloride)	63/100 41/ 90 62/ 70	8:29
Trichloroethylene (Trichloroethene)	130/100 132/ 92 95/ 87	8:10
cis-1,3-Dichloropropene	75/100 39/ 70 77/ 30	9:41
trans-1,3-Dichloropropene (1,3 dichloro-1-propene)	75/100 39/ 70 77/ 30	10:36
1,1,2-Trichloroethane (Vinyl trichloride)	97/100 83/ 90 61/ 82	10:53
Toluene (Methyl benzene)	91/100 92/ 57	10:08
1,2-Dibromoethane (Ethylene dibromide)	107/100 109/ 96 27/115	11:36
Tetrachloroethylene (Perchloroethylene)	166/100 164/ 74 131/ 60	11:00
Chlorobenzene (Benzene chloride)	112/100 77/ 62 114/ 32	12:27
Ethylbenzene	91/100 106/ 28	12:41
m,p-Xylene(1,3/1,4-dimethylbenzene)	91/100 106/ 40	12:56
Styrene (Vinyl benzene)	104/100 78/ 60 103/ 49	13:38
1,1,2,2-Tetrachloroethane (Tetrachloroethane)	83/100 85/ 64	14:34
o-Xylene (1,2-Dimethylbenzene)	91/100 106/ 40	13:37
4-Ethyltoluene	105/100 120/ 29	
1,3,5-Trimethylbenzene (Mesitylene)	105/100 120/ 42	14:51
1,2,4-Trimethylbenzene (Pseudocumene)	105/100 120/ 42	15:16
m-Dichlorobenzene (1,3-Dichlorobenzene)	146/100 148/ 65 111/ 40	15:31

(continued)

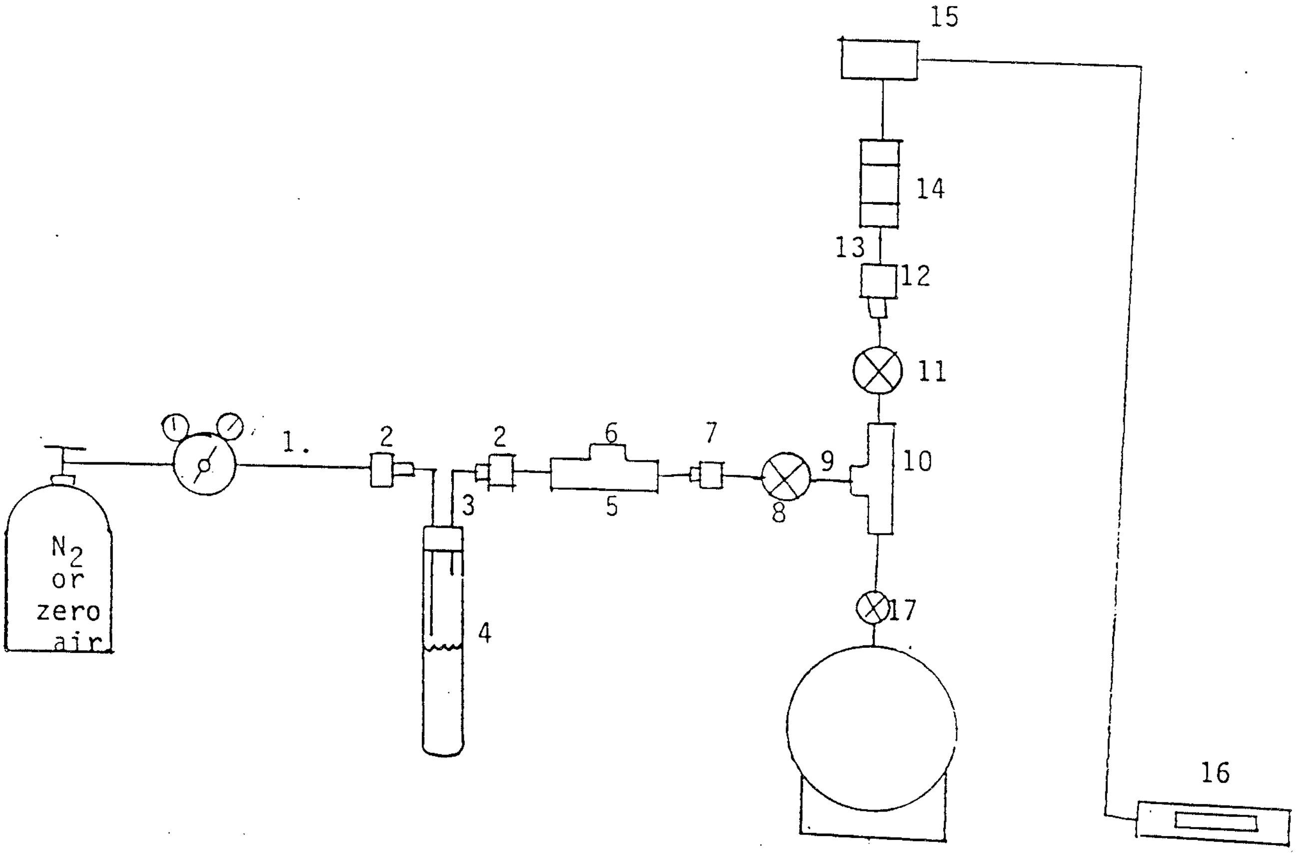
TABLE 2. ION/ABUNDANCE AND EXPECTED RETENTION TIME FOR SELECTED VOCs ANALYZED BY GC-MS-SIM (cont.)

Compound	Ion/Abundance (amu/% base peak)	Expected Retention Time (min)
Benzyl chloride (α-Chlorotoluene)	91/100 126/ 26	15:47
p-Dichlorobenzene (1,4-Dichlorobenzene)	146/100 148/ 65 111/ 40	15:38
o-Dichlorobenzene (1,2-Dichlorobenzene)	146/100 148/ 65 111/ 40	16:00
1,2,4-Trichlorobenzene	180/100 182/ 98 184/ 30	17:39
Hexachlorobutadiene (1,1,2,3,4,4 Hexachloro-1,3-butadiene)	225/100 227/ 66 223/ 60	17:51
trans-1,2-Dichloroethene	61/100 96/84	4:26
Dibromomethane	174/100 93/95	8:41
Bromodichloromethane	83/100 85/66	8:59
1,3-Dichloropropane	76/100 41/78	11:07
Dibromochloromethane	129/100 127/77	11:29
1,1,1,2-Tetrachloroethane	131/100 133/96	12:39
Bromoform	173/100 171/50	13:52
Isopropylbenzene (cumene)	105/100 120/25	14:08
Bromobenzene	77/100 156/77	14:27
1,2,3-Trichloropropane	75/100 110/37	14:35
Propylbenzene	91/100 120/21	14:38
p-Isopropyltoluene	119/100 91/42	15:36
Butylbenzene	91/100 92/55 134/24	16:01

TABLE 3. 4-BROMOFLUOROBENZENE KEY IONS AND ION ABUNDANCE CRITERIA

Mass	Ion Abundance Criteria
50	15 to 40% of mass 95
75	30 to 60% of mass 95
95	Base Peak, 100% Relative Abundance
96	5 to 9% of mass 95
173	<2% of mass 174
174	>50% of mass 95
175	5 to 9% of mass 174
176	>95% but< 101% of mass 174
177	5 to 9% of mass 176

FIGURE 1. CANISTER PRESSURIZATION/STANDARDIZATION SYSTEM.



1. 1/8 in. tubing, ss
2. 1/8 to 1/16 in. reducing union
3. 1/16 in. TFE tubing
4. humidifier
5. 1/8 in. union tee
6. injection septum
7. 1/4 to 1/8 in. reducing union
8. system valve (SS-42S4)
9. 1/4 in. tubing, ss
10. 1/4 in. union tee
11. transducer valve (SS-42S4)
12. 1/2 to 1/4 in. reducing union
13. 1/2 in. tubing, ss
14. Cajon Ultratorr union, 1/2 in.
15. pressure transducer
16. pressure readout box
17. canister valve

FIGURE 2. ANALYTICAL SYSTEM

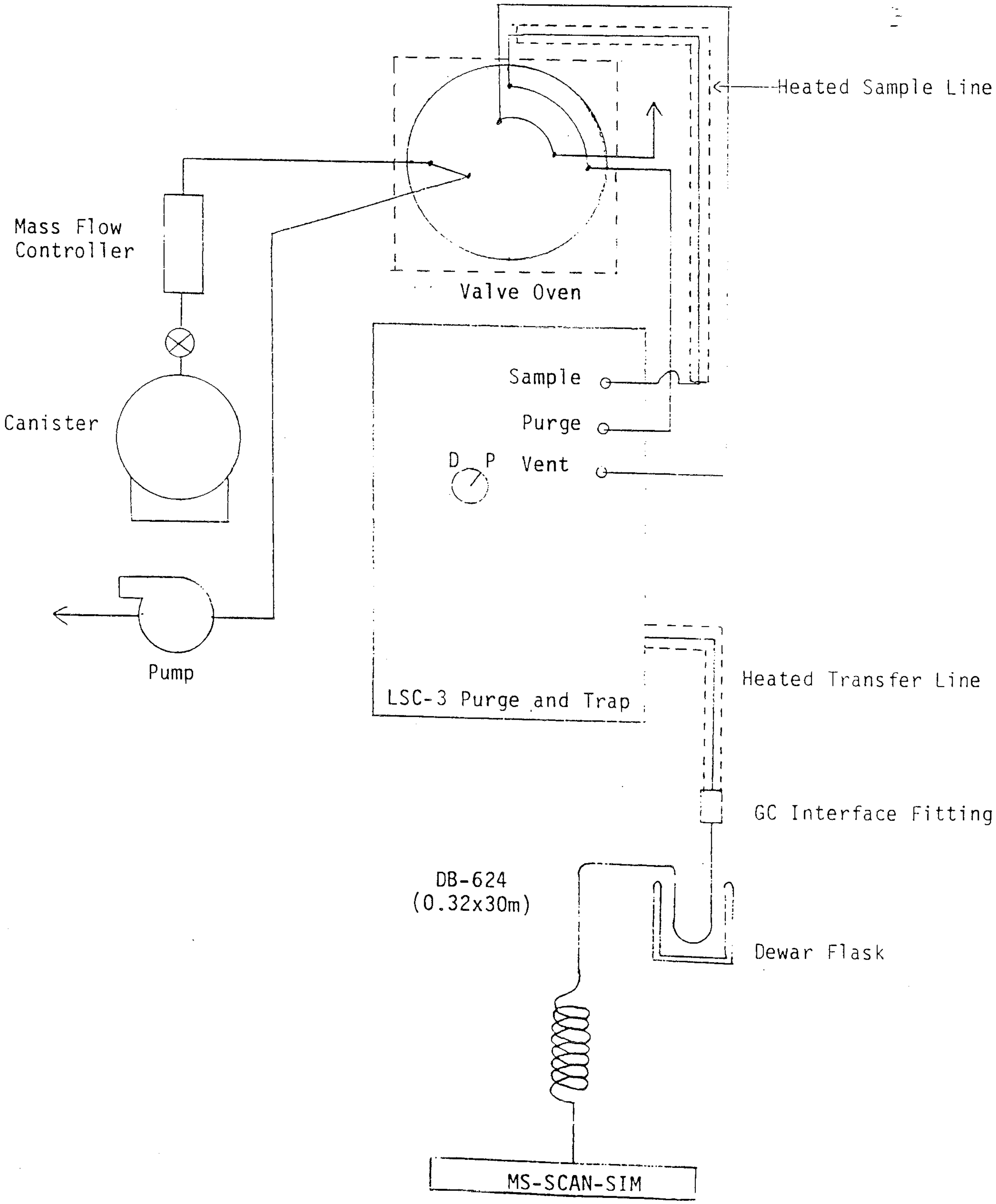
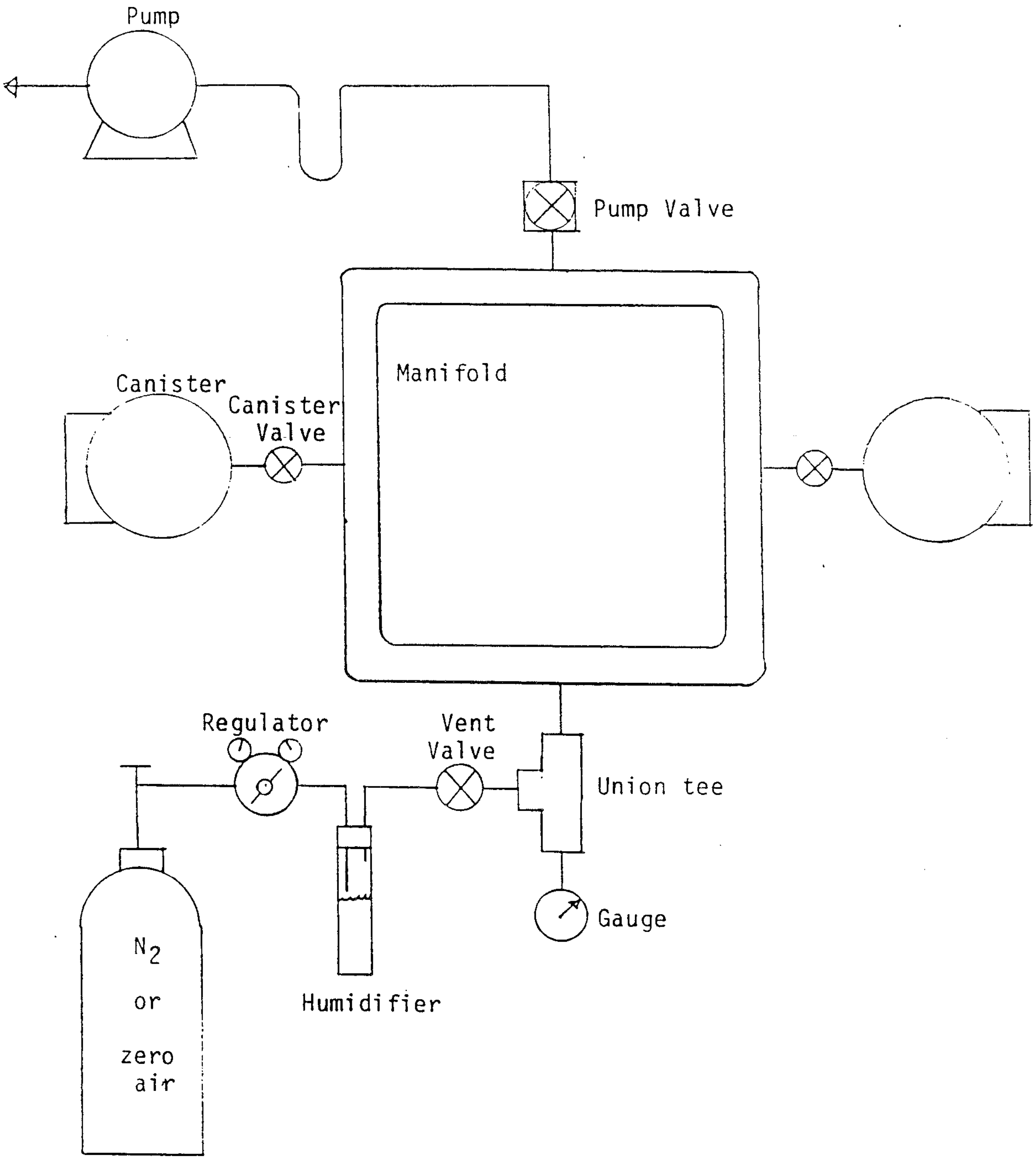


FIGURE 3. CANISTER CLEANING SYSTEM



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